

**Study of the Effect of Mechanical Cycling on the Constitutive Behavior of
Lead-Free Solders**

by

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Abstract

Solder joints in electronic assemblies are often subjected to cyclic (positive/negative) mechanical strains and stresses. Such exposures can occur in variable temperature application environments or during accelerated life thermal cycling tests used for qualification. Lead free solder materials are widely used in the electronic packaging industry due to environmental concerns. However, experimental testing and microstructural characterization have revealed that Sn-Ag-Cu (SAC) lead free solders exhibit evolving properties that change significantly with isothermal aging and cyclic loading. This dissertation addresses those changes in the microstructure and properties of lead free solders by conducting four different projects.

In the first project, the effects of isothermal mechanical cycling on damage accumulation in lead free SAC305, SAC_Q and Innolot bulk solder alloys have been explored. Uniaxial samples of SAC305, SAC_Q and Innolot were prepared and subjected to various durations of prior mechanical cycling. The cycled samples were then subsequently subjected to stress-strain or creep testing. The cycling induced changes in several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) have been characterized and quantified with the amount of prior cycling. Moreover, these evolutions observed in the three lead free alloys were correlated with the observed changes in their microstructures that occurred during the cycling.

In the second project, the effects of elevated temperature isothermal mechanical cycling on damage accumulation in lead free SAC305 and SAC_Q bulk solder were explored. Uniaxial samples of SAC305 and SAC_Q were prepared and subjected to various durations of prior mechanical cycling at 100 °C. The cycled samples were then

subsequently subjected to stress-strain or creep testing. The cycling induced reduction of several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) have been characterized and quantified with the amount of prior cycling. The results obtained were then compared with the results obtained with room temperature cycling to better understand the effect of elevated temperature mechanical cycling. Moreover, these evolutions observed in the two lead free alloys were correlated with the observed changes in their microstructures that occurred during the cycling and compared with the changes in the microstructure obtained during room temperature cycling.

In the third part of this dissertation, the effect of strain range on the damage evolution of mechanically cycled SAC305 solder material was investigated. The samples were cycled at various strain ranges and the corresponding hysteresis loop areas were evaluated. The data was then used to generate plots of initial loop area vs strain range which allowed the estimation of initial plastic work for any given strain range. Samples were then cycled at room temperature and elevated temperature such that the initial plastic work remained the same. This was done for four different initial plastic work. Such a study would allow us to deduce the changes brought about by cycling with any particular initial plastic work when cycled at either 25 °C or 100 °C. This evolution in the properties that occur during mechanical cycling would then allow us to estimate and quantify the damage accumulated due to mechanical cycling at any particular strain range. These properties could then be incorporated into a constitutive model (such as Anand Model) that could then provide a method which would allow the modelling of the damage accumulation within SAC305 lead free alloys.

In the next part of this dissertation, the aging induced evolutions of the cyclic stress-strain behavior of SAC305 solder material was investigated. The specimens were aged at 125 °C for different durations (0, 1, 2, 5, 10, and 30 days) and the evolution of the hysteresis loop area, peak load, and plastic strain range were characterized. Later, the effects of prior aging on damage accumulation occurring in SAC305 was investigated while subjected to mechanical cycling (fatigue testing). Regions of interest near the center of the sample were marked using a nanoindentation system. Samples were then subjected to aging at 125 °C. After aging, the samples were then subjected to mechanical cycling. After various durations of cycling (e.g. 0, 10, 25, 50, 75, 100, 200, 300 cycles), the fixed were examined using scanning electron microscopy (SEM) Using the recorded images, the microstructural evolutions in the fixed regions were observed, and the effects of the initial aging on the results were determined.

In the following project, the mechanical cyclic induced evolutions in mechanical properties of SAC 305 and SAC_Q individual solder balls were investigated. The test assemblies in this study were (3X3) BGA packages composed of total nine 0.75 mm diameter lead free solder balls which were formulated by reflowing solder spheres soldered onto 0.55 mm diameter Cu pads on FR4 coupons. The solder balls were cycled using an Instron Micromechanical tester along with a newly designed fixture which facilitates the tester to cycle a solder ball individually. Nanoindentation tests were performed on the specimens to study the evolution in mechanical properties (e.g. elastic modulus, hardness and creep strain rate) of the solder balls as a function of duration of cycling. A finite element model was also used to develop to estimate the plastic work accumulated in these solder balls when subjected to shear cycling.

In the last chapter of this dissertation, 3X3 BGA sandwiched SAC305 solder joints soldered on to Cu pads on FR4 coupons were mechanically cycled in a shear direction. This was done using the MT-210 microtester machine coupled with newly designed fixtures to firmly grip the specimens. The load-displacement curves obtained were then studied. These sandwiched specimens were also polished using a novel technique that allowed the extraction of these samples after polishing. After polishing, some of the samples were aged at 125 °C for 10 days. The aged and non-aged samples were then subjected to shear mechanical cyclic loads at either room temperature or elevated temperature. The changes in microstructure of the solder joints with mechanical cycling were then recorded using a cross-polarized optical microscope. Any recrystallization effects brought about by mechanical shear cycling (both at 25 °C or 100 °C) or by aging were also investigated. Finally, a finite element model was developed to estimate the plastic work accumulated during the mechanical cycling and validated with experimental studies.

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CHAPTER 1

INTRODUCTION

1.1 Overview of Solders in Microelectronics

Solders have been used as the interconnecting material in electronic assemblies to provide both electrical and mechanical connections between components. As a joining material, in electronic assemblies, solder provides mechanical support, electrical and thermal interconnection between the semiconductor device (Si die) and the bonding pad. It also helps to dissipate the heat generated from the Si-chip [1].

In the past, the conventional eutectic or near eutectic Sn-Pb solder has been extensively used as primary interconnect material in electronic packaging industries. The eutectic (63Sn-37Pb) tin-lead alloy was very attractive due to its relatively low melting temperature (183 °C), which allows the soldering process compatible with most of the substrate materials and devices. Lead (Pb) provides many technical advantages to tin-lead solders that includes 1) prevent allotropic transformation of β -tin to α -tin at low temperature, 2) enhance wetting by lowering the surface tension of the solder and 3) increase corrosion resistance of the solder by formation of a oxide film which is insoluble and resistant to chemical attack etc.[1, 2].

However, lead is poisonous to human being as well as animals. According to a recent study, lead (Pb) in the child's blood could cause serious problem in neurological and physical development. Moreover, being a toxic element, it has some detrimental effect on the environment [3-8]. As a result, despite of all the advantages of tin-lead solders, governments of many countries are imposing regulations against the use of lead in the

electronic devices [1]. As an effort of eliminating lead in electronics, the EU adopted two directives, 1) the Waste of Electrical and Electronic Equipment (WEEE) and 2) the Directive of the Restriction of the Use of Certain Hazardous Substances (RoHS) in June 2000 [9]. As a result of the enforcement of the directives, all electrical or electronic equipment and devices produced in or imported to E.U. member states must comply with these lead-free standards except those items that are exempted from the bans. In addition to legislation enforcement there are emerging detection technologies to enforce compliance. So due to the general push towards the eco-efficiency and green electronics, manufacturers are motivated for the adoption of lead free electronics. Therefore, the conversion to lead free solders in the global electronic market appears imminent [10].

In the United States, as soon as lead-free solder legislation was proposed, the lead-free solder project headed by the NCMS initiated research and development of lead-free solder in a program lasting 4 years [11]. The results of the project have been made available in a database and offer information on such matters as modifying equipment and processes for selecting alternative materials. The project initially selected for study 79 types of alloys considered at the time to be potential candidates for use in lead-free solder. Basic attributes considered included toxicity, resource availability, economic feasibility, and wetting characteristics. The selection process narrowed the field down to the final seven alloys, and these received secondary evaluation for reliability and ease of mounting manufacturing. Evaluation of the individual alloys did not result in the final selection of a single candidate, but three alloys, Sn–58Bi, Sn–3.5Ag–4.8Bi, and Sn–3.5Ag, were recommended as candidates. Screening comments indicated that the Sn–58Bi eutectic alloy was not suitable for use as standard solder due to the scarcity of Bi resources. However, since this material

can be used for mounting at less than 200 °C, and has chalked up a 20-year plus record of use in mainframe computers, this solder was deemed suitable for special applications. These results were used to construct a database on lead-free solder that includes the information in these tables along with other items such as (1) recommended applications for lead-free solder, (2) alloy composition guidelines reflecting price and availability, (3) database of the 7 selected alloys and comparison with Sn–Pb eutectic alloy, (4) data on the characteristics of the other 70 eliminated alloys, (5) optimal process conditions using various test PWBs, (6) strength evaluation and metallurgical reaction analysis for the selected alloys and various surface mounting process reactions, (7) estimated life (using NCMS Project proprietary life estimation software) and thermal fatigue evaluation for 4 of the selected alloys, and (8) assessment of nontoxicity and alloy composition.

1.2 Lead Free Solders

While selecting the suitable lead free alternative, it is important to consider that the properties of the lead free solder must comparable or superior to Tin-lead (Sn-Pb) solders. The performance requirement of solder alloys, for microelectronics application, are very strict. In general, the lead free solder alloy must have desired level of electrical and mechanical properties, melting temperature, wetting capability on most common PCB substrates etc. while at the same time must not enhance assembly cost. Compatible candidates of the Sn-Pb solders must have the following behaviors [5]:

- Melting temperature similar to eutectic Sn-Pb for a similar reflow profile
- Sufficient wettability for good metallization process
- Good electrical properties for transmitting electrical signals
- Strong mechanical properties for good fatigue resistance and reliability

- Inexpensive and easier manufacturability

Among various alloy systems that are considered as lead-free solder candidates, Sn-Ag-Cu alloys have been recognized as the most promising because of their relatively low melting temperature (compared with the Sn-Ag binary eutectic lead free solder), superior mechanical properties, and good compatibility with other components [12-14]. Sn-Ag-Cu alloys are widely used as lead-free solutions for ball-grid-array (BGA) interconnection in the microelectronic packaging industry as solder balls and pastes. Although no “drop in” replacement has been identified that is suitable for all applications, Sn-Ag, Sn-Ag-Cu (SAC), and other alloys involving elements such as Sn, Ag, Cu, Bi, In, and Zn have been identified as promising replacements for standard 63Sn-37Pb eutectic solder. Industries have proposed several SAC alloys which include 96.5Sn-3.0Ag-0.5Cu (SAC 305) in Japan, 95.5Sn-3.8Ag-0.7Cu (SAC 387) in the EU, and 95.5Sn-3.9Ag-0.6Cu (SAC 396) in the USA. The International Printed Circuit Association has suggested that 96.5Sn-3.0Ag-0.5Cu (SAC305) and Sn-3.9Ag- 0.6Cu (two near-eutectic alloys) will be the most widely used alloys in the future [15]. This estimation is attributed to their good mechanical properties, acceptable wetting properties, and suitable melting points [12, 16, 17].

1.3 Candidates for Alternative Lead Free Solders

About 70 different alloys were proposed as an alternative to the Sn-Pb solder. Most of these are Sn based solder where Sn is the main constituent along with one, two or even three other minor elements. These minor elements are added mainly to 1) decrease the melting temperature and 2) improve wetting and reliability of the solder [1]. The properties

of Sn and the effects of different alloying elements on the Sn based solder alloys are described below:

1.3.1 Tin

The melting temperature of elemental Sn is 231 °C. One of the main reasons of choosing Sn as the principle component in the lead free solder for electronic applications is its ability to spread and wet a number of various different substrates. In the solid state, tin can have two different phases or crystal structures 1) white or β -Sn with tetragonal crystal structure and 2) gray or α -Sn with diamond cubic crystal structure. At the room temperature the thermodynamically stable phase is β -Sn. Upon cooling, when the temperature goes below 13 °C (allotropic transformation temperature), α -Sn becomes the thermodynamically stable phase. The allotropic transformation of β -Sn to α -Sn, results a significant volume change (around 27%) causing blistering of the tin surface, cracking or disintegration. This phenomenon is mainly a surface event and often referred as ‘tin pest’. The low ductility of α -Sn is another reason of causing blistering and cracking of the tin after the transformation [1, 6, 8]. Although the equilibrium temperature for β -Sn to α -Sn is 13 °C, the transformation occurs only after a significant undercooling and an extensive incubation period as long as several years. For example, no ‘tin pest’ was found on a SAC387 bulk sample after storing at -40 °C for 5 years [6]. Presence of heterogeneous nuclei, also known as seeding, can considerably accelerate the kinetics of the transformation. The possibility of $\beta \rightarrow \alpha$ transformation in an actual solder joint is even limited due to the constraints (component and substrate) on the both side of the joint [6, 8].

Addition of other elements, as an impurity, can affect the allotropic transformation. For example, the presence of Pb, Bi, Sb, Cu, Ge and Si inhibit $\beta \rightarrow \alpha$ transformation while the presence of As, Zn, Al and Mg promote the transformation [6, 8].

Sn has a body centered tetragonal crystal structure (β -Sn), at room temperature, which is anisotropic. Hence, tin shows an anisotropic thermal (e.g. CTE) and mechanical (e.g. elastic modulus) properties as shown in Figure 1.1. As a result, during thermal cycling experiment, cracking occurs along the grain boundaries [1].

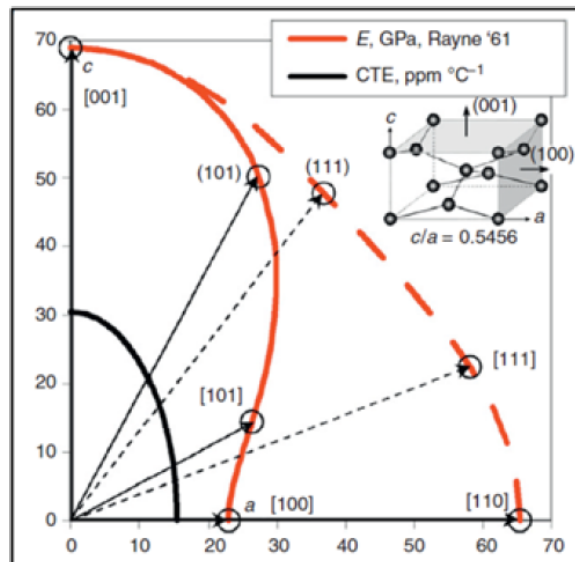


Figure 1.1 Elastic Modulus and Coefficient of Thermal Expansion (CTE) of Tin as a Function of Crystal Orientation [1]

1.3.2 Chromium (Cr)

Cr helps to improve shear ductility after long term aging. Besides, it also suppress Kirkendall void formation in the solder joints [3].

1.3.3 Nickel (Ni)

Among all the micro-alloy additives, Ni is one of the most common element. Addition of Ni improves fluidity of SAC solders. Ni also improves the high strain rate

properties of solder joints. For example, the drop strength of SAC alloys can be significantly improved by Ni addition. Although Ni does not have any significant influence on the creep properties of the solder, it inhibits Cu diffusion and thus reduce the thickness of Cu_3Sn intermetallic compound (IMC). The Ni addition greater than 0.01 wt% could suppress the growth of Cu_3Sn IMC even after long term (2000 hours) aging. Since Cu_3Sn IMC is very brittle, the growth of Cu_3Sn layer is very critical for the brittle failure of the solder joints. Addition of Ni on SAC solder causes to form more stable $(\text{Cu}, \text{Ni})_6\text{Sn}_5$ IMC which act as a barrier layer and suppress the growth of Cu_3Sn layer. Therefore, by reducing the thickness of brittle Cu_3Sn layer, Ni helps to improve shear strength of the solder joints. Ni also helps to improve strength by refining solder microstructure [7].

1.3.4 Zinc (Zn)

While Ni reduces only the formation Cu_3Sn , Zn doping can retard both Cu_3Sn and Cu_6Sn_5 IMC's in SAC solders. Zn also helps to improve the interface quality after multiple reflow and high temperature aging. Addition of 1.5% Zn in SAC207 solder could refine Ag_3Sn and Cu_6Sn_5 IMC's and hence increase strength by dispersion strengthening [3].

1.3.5 Cobalt (Co)

Co helps to reduce the growth of Cu_3Sn layer during high temperature aging. Addition of small amount of Co (<0.1 wt%) can improve the properties of SAC solders. Co increases the number of nucleation sites and hence significantly refine the grains of SAC305 solder. Thus it helps to improve shear strength of the solder joints [3].

1.3.6 Bismuth (Bi)

If added in small amount, Bi can improve the wetting ability and reduce melting temperature of lead free solder alloys. It also increases strength of the bulk solder and inhibit the large Ag_3Sn formation in the bulk solder. It is recommended to avoid Pb contamination in the solder before using Bi as an additive. Because Bi can react with Pb to form a brittle IMC at the grain boundary and reduce strength significantly. If present in excess amount, Bi can cause solidification crack due to the increase in the gap between solidus and liquidus temperature [3].

1.3.7 Antimony (Sb)

Sb improves mechanical properties of lead free solders but it is toxic in nature. A small percentage (0.5 wt%) of Sb can improve drop test reliability of SAC solder joints [8]. Besides, Sb also helps to enhance strength of the solder by solid solution strengthening [7].

1.3.8 Germanium (Ge)

Ge doped solders shows low Cu dissolution from the Cu pad and hence suitable for the wave soldering process. Ge significantly improve wetting properties of the lead free solders and refine the solder microstructure. As a result, Ge addition improves the strength and ductility of the lead free solder [4].

1.3.9 Sn-Ag-Cu System

A variety of SAC alloys with different chemical compositions have been the proposed by various user groups and industry experts. Among the various lead free solder candidates, Sn-Ag-Cu alloys have gained a significant interest in the academia as well as in the industry

because of their superior mechanical properties, relatively low melting temperature, and good solderability or wetting properties [13, 14] . A graphical representation of the market share of different lead free solders is presented in Figure 1.2. It is clear from the figure that Sn-Ag-Cu alloys are dominating in the lead free solder market.

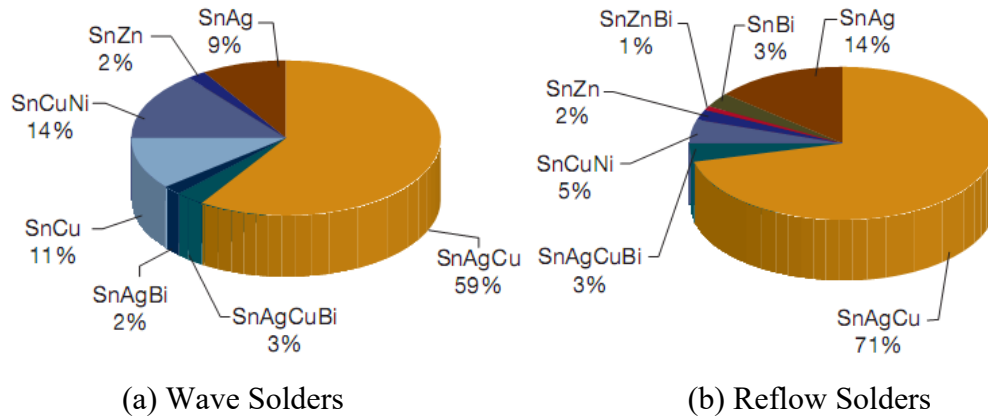


Figure 1.2 Market Share of Lead Free Solder
(https://www.ameslab.gov/files/LeadFreeSolder_Foundation.pdf)

Figure 1.3 shows a typical 3-D ternary phase diagram. The contours on the top surfaces of the figure represent the isothermal lines. Each of the 3 sectors represents the binary phase diagram of two of the three elements. The center of the diagram, where the isothermal lines reach the common, lowest point, is the eutectic point of the ternary system.

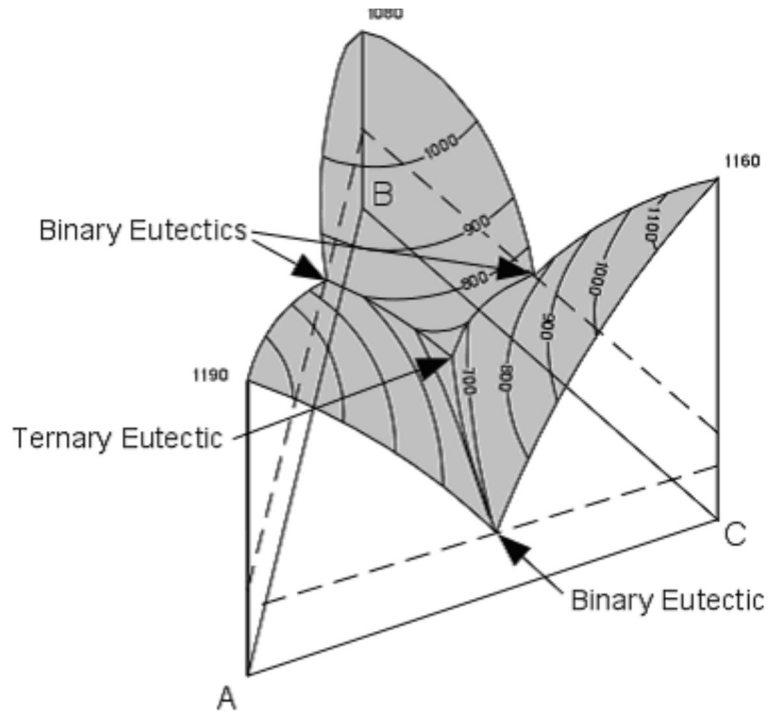


Figure 1.3 Typical 3-D Ternary Phase Diagram

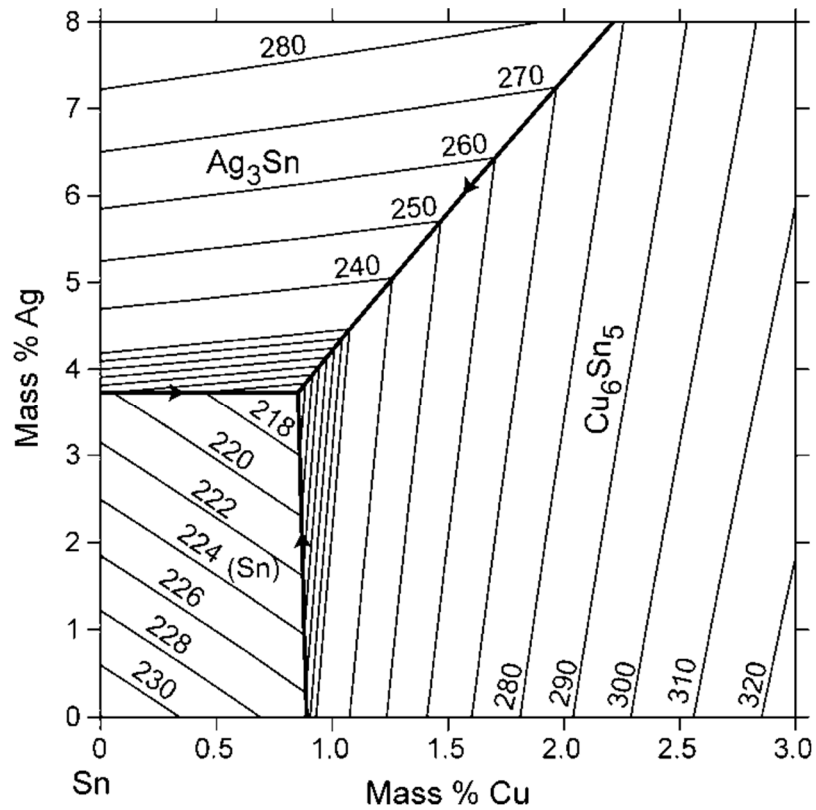


Figure 1.4 Sn-Ag-Cu Ternary Phase Diagram

Figure 1.4 is the top view (2-D) of the ternary phase diagram of Sn-Ag-Cu. The area indicated in the red box is the near eutectic region. Most of the SAC alloy compositions currently on the market are within this region. The eutectic and near eutectic melting temperature has been determined to be 217 °C, although the precise eutectic point is not known [18]. In SAC alloys, the formation of intermetallic compounds between the primary elements Sn and Ag, and Cu affect all the properties of the alloys. There are three possible intermetallic compounds that may be formed: Ag_3Sn forms due to the reaction between Sn and Ag and Cu_6Sn_5 forms due to the Sn and Cu reaction. The compound Cu_3Sn will not form at the eutectic point unless the Cu content is high enough for the formation of Cu_3Sn at higher temperatures, so in bulk specimens Cu_3Sn is not presented. There is no reaction between Ag and Cu to form any kind of intermetallic compounds. Figure 1.5 shows a typical microstructure of SAC lead free solders. The near eutectic SAC solidifies into three phases, i.e., β -Sn (primary phase), Ag_3Sn and Cu_6Sn_5 . It consists of β -Sn dendrites surrounded by interdendritic eutectic regions incorporating a fine dispersion of Ag_3Sn and Cu_6Sn_5 intermetallic particles in β -Sn. The volume fraction of Ag_3Sn IMCs is much higher than that of Cu_6Sn_5 IMCs in SAC solder microstructures. The particles of intermetallic compounds possess much higher strength than the bulk material. Fine intermetallic particles in the Sn matrix can therefore strengthen the alloys. The intermetallic compounds can also improve the fatigue life of the solders, as SAC alloys are reported to be 3-4 times better fatigue properties than the Sn-Pb eutectic solders. Moreover, the dendrite structure can effectively pin and block movement of dislocations and therefore improve solder mechanical behavior.

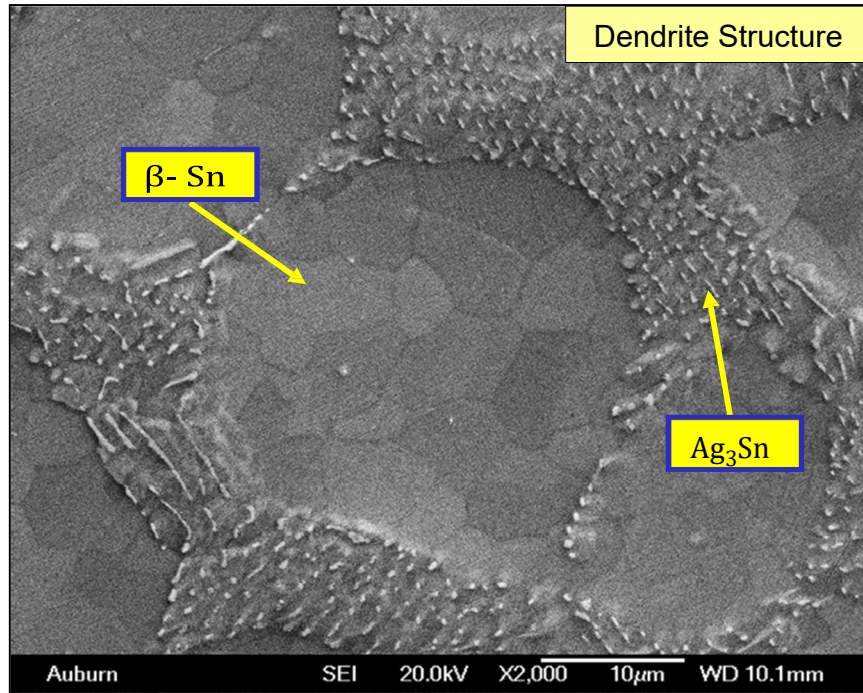


Figure 1.5 Typical Microstructure of SAC Lead Free Solders

During the initial stages of the development of lead free solder, eutectic Sn-Ag-Cu alloy (SAC387: 95.5Sn-3.8Ag-0.7Cu) was most popular. But, due to the cost of the patent by Iowa State University and the higher Ag%, SAC305 (96.5Sn-3.0Ag-0.5Cu) was seen to be advantageous over SAC387. Moreover the performance of SAC305 is similar to that of the eutectic Sn-Ag-Cu alloy. As a result, the electronic industry has chosen SAC305 to use in most of the lead-free applications [3].

1.3.10 Sn-Ag-Cu + X System

Despite the benefits mentioned above, SAC family solders sometimes are still questionable as complete substitutes for eutectic Sn-Pb because of costs, some patent issues, aesthetic consideration, and relatively high melting temperature (217 °C vs. 183 °C). Moreover, due to the increased price of Ag, the cost of SAC305 solder bar has

increased about 3 times in the past 10 years [3]. Besides, the mechanical properties of the lead free alloy is not stable during the long time exposure at an elevated temperature (aging effect). It has been reported previously, that isothermal aging causes a significant reduction in the reliability of the lead free solder joints [19]. As a result, people are now looking for a low cost alternative with better reliability than SAC305. According to the results of many recent studies, performance characteristics of SAC solder alloys are able to be optimized by doping, that is, by adding a small amount of other alloying elements into the SAC solder alloys.

The proposed doping element candidates include Bi, Ni, Co, Ge, Zn, La, Mg, Mn, Ce, Ti, Fe, In, B, etc. For example, adding 0.05% (wt.) Ni can successfully stabilize the microstructure, inhibit the excessive consumption of metal base and thus increase the reliability of the solder joints [20-22]. In addition, doping rare earth (RE) elements can significantly enhance wettability, refine microstructure and improve ductility of SAC alloys [23-25]. Even though dopants can greatly alter the mechanical, electrical and physical behavior of SAC solders, the effect on melting temperature, however, is found to be negligible. This is another advantage for doped solder alloys because manufacturers can still use the same processing conditions as conventional SAC alloys. However, it takes much more time and cost to figure out the optimal composition levels for the dopants.

1.4 Mechanical Properties of Lead Free Solders

For the SAC lead free solders, the properties of Sn dominate their behavior as Sn has a very high weight percentage. Sn has a Body-Centered Tetragonal (BCT) crystal structure with lattice parameters $a = b = 5.83 \text{ \AA}$ and $c = 3.18 \text{ \AA}$. Therefore, it is highly anisotropic in mechanical properties such as elastic modulus and hardness. In other words,

the mechanical properties of Sn are highly dependent on the grain orientation. As shown in Figure 1.6, the elastic modulus of Sn is 67.6 GPa in the (001) direction, while it is only 23.6 GPa in the (100) direction [26]. Also, Sn is highly thermally anisotropic: the coefficients of thermal expansion (CTE) in the principal directions at 30 °C are $\alpha[100] = \alpha[010] = 16.5 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ and $\alpha[001] = 32.4 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ [27].

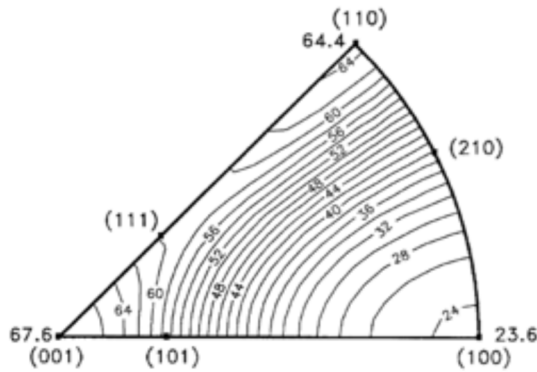


Figure 1.6 Variation of Elastic Modulus of β -Sn with Grain Orientation [22]

In an electronic device, a number of different types of engineering materials exists in a close proximity. For example, a printed circuit board (PCB) is typically a glass fiber reinforced polymer (composite material), a die is a semiconductor material which is often encapsulated in a plastic or a ceramic, and the tracking and the solder joints are metallic materials. Solder joints are used to create an electrical circuit by mounting chips and components on the PCB. Hence an ideal solder joint should have a good conductivity to transmit electrical signals and at the same time, adequate strength to provide mechanical support and connection. Hence, mechanical properties of solder joints are critically important to ensure reliability of the electronic products. Among all the mechanical properties, tensile, fatigue, and creep performance of the solder are critically important. Thus an accurate measurement of mechanical properties and development of constitutive

equations for solder materials are required in mechanical design, process optimization and reliability assessment.

1.4.1 Tensile Properties (Stress-Strain Behavior)

Under the action of an increasing stress, metals usually exhibit elasticity, plasticity, and a maximum in stress is followed by necking and fracture. The slope of the linear elastic portion of the stress vs. strain plot is the modulus, and the stress at termination of elastic behavior is the yield stress. Tensile properties indicate how the material will react to forces being applied in tension. Although in real life, actual solder joints are rarely under uniaxial tensile load, these properties are required for design purpose. Tensile properties help to determine the response of a material under uniaxial tension loading. Tensile tests provide us information about several material properties, such as ultimate tensile strength (UTS), yield strength (YS), effective elastic modulus (E), elongation, etc.

The tensile behavior of a material is commonly described by a stress-strain curve. A tensile test experiment typically provide a load-elongation curve which is then converted into a stress-strain curve using following equations:

$$\sigma_e = \frac{F}{A_o} \quad (1.1)$$

$$\epsilon_e = \frac{\Delta L}{L_o} = \frac{L_f - L_o}{L_o} \quad (1.2)$$

Where, F is the applied load, A_o is the initial cross-sectional area, L_o is the initial length, L_f is the final length (after deformation), σ_e is the engineering stress, and ϵ_e is the engineering strain. Since the change in physical shape is negligible, for most of the cases, ‘engineering’ stress-strain curves are used instead of ‘true’ stress-strain curves. A typical stress-strain curve for a ductile material is presented in Figure 1.7. As shown in the figure,

stress and strain shows a linear relationship at the beginning of the test. In this linear portion of the curve, stress and strain exhibits a particular relationship known as Hook's Law which is described as:

$$\sigma = E\varepsilon \quad (1.3)$$

Where E is the effective elastic modulus which is defined by the slope of the initial portion of the stress-strain curve. No plastic deformation occurs in the linear elastic part of the stress-strain curve. Hence theoretically, within the linear portion, if the stress is released, the material should come back to its original shape (zero deformation). However, for a viscoplastic material like solder, time dependent plastic deformation or creep occurs even with a small load (below yield stress). As a result, the value of effective elastic modulus, obtained from stress-strain curve, is typically smaller than the value of modulus measured from a dynamic measurement technique like ultrasonic or acoustic method. The rapid wave propagation, in a dynamic technique, mostly eliminates any inelastic deformation [28-30].

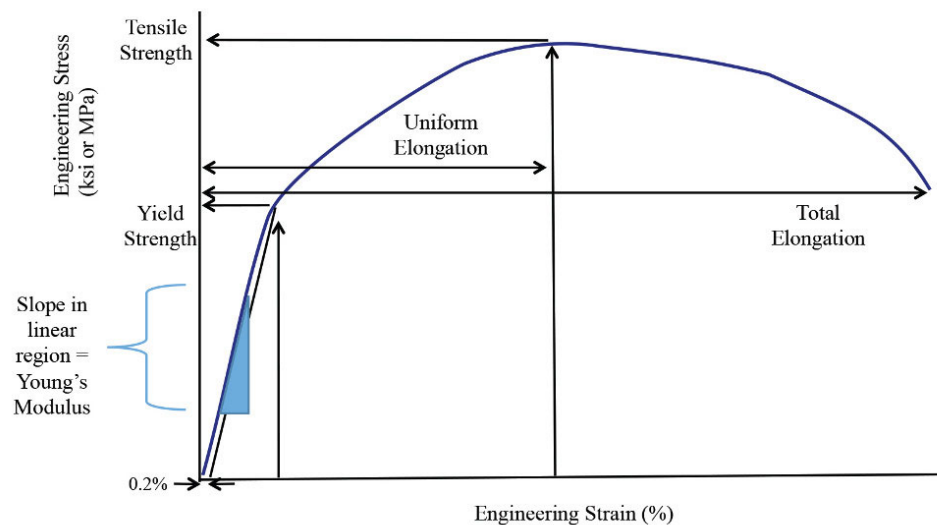


Figure 1.7 Typical Tensile Test Response of a Ductile Material

[<https://www.thefabricator.com/article/metalsmaterials/getting-to-know-more-about-the-metal-you-are-forming>]

Eventually, after a certain period of time the stress-strain curve become nonlinear. This nonlinearity is caused by the stress induced inelastic deformation or plastic flow of the material. In this stage, the atoms of the test specimen undergoes a rearrangement among themselves and moves to a new equilibrium position. In a crystalline material, like solder, this rearrangement is caused by dislocation motion. The point where the curve begins to show a nonlinear behavior or inelastic plastic flow is called yield point and the stress associated with the point is known as yield strength (YS). Since it is difficult to identify the exact stress that caused the plastic flow, YS is typically defined as the stress required to cause 0.2% plastic deformation. Yield strength of a material can be determined from the stress-strain curve by drawing a line parallel to the initial linear portion but 0.2% offset from the origin. The stress associated with the intersection of the parallel line and the stress-strain curve is the yield strength of the material. The ultimate tensile strength (UTS) or tensile strength is defined as the maximum stress obtained from a uniaxial tension test. After UTS, the decrease in stress (as shown in Figure 1.7) is due to the localized deformation also known as necking in the test sample. If the test continues beyond the UTS, the test sample will eventually break or fail. The stress associated with it is called failure strength and strain at the failure point is known as total elongation (Figure 1.7).

1.4.2 Creep

Creep deformation refers to the time dependent plastic flow or deformation of a material that occurs when the material is exposed to a constant load, typically below yield strength, for a long period of time. Creep deformation becomes significant when the

material operates at a high homologous temperature (T_h), which is defined by the ratio of operating temperature (T) and the melting temperature (T_m) of the material.

$$T_h = \frac{T}{T_m} \quad (1.4)$$

Creep deformation becomes the dominant failure mode in a metallic material if T_h is greater than $0.5T_m$. [31]. The melting temperature of lead free SAC solder is around 217 °C (490 K) causing T_h for the alloys, for room temperature (298 K) operating conditions, is $0.61T_m$. As a result, lead free SAC solder alloys display creep deformation even in room temperature operating condition. Due to the mismatches of the coefficient of thermal expansion (CTE) of silicon chip and other assembly materials used in an electronic package, solder joints are remain under mechanical stress. These mechanical stresses can cause time dependent creep deformation of solder materials. In microelectronic packaging, creep deformation is regarded as one of the major failure mechanisms of solder joints [32].

Creep test is typically conducted by applying a constant uniaxial load on the test specimen at a particular temperature. During the test, deformation of the test specimen is recorded as a function of test time and the result of the creep test is presented as a ‘creep strain’ vs. ‘time’ plot. The extent of creep deformation significantly depends on the applied stress level and the test temperature. Figure 1.8 represents a typical creep curve which consists of three distinct regions, after the initial jump, namely, primary, secondary, and tertiary regions.

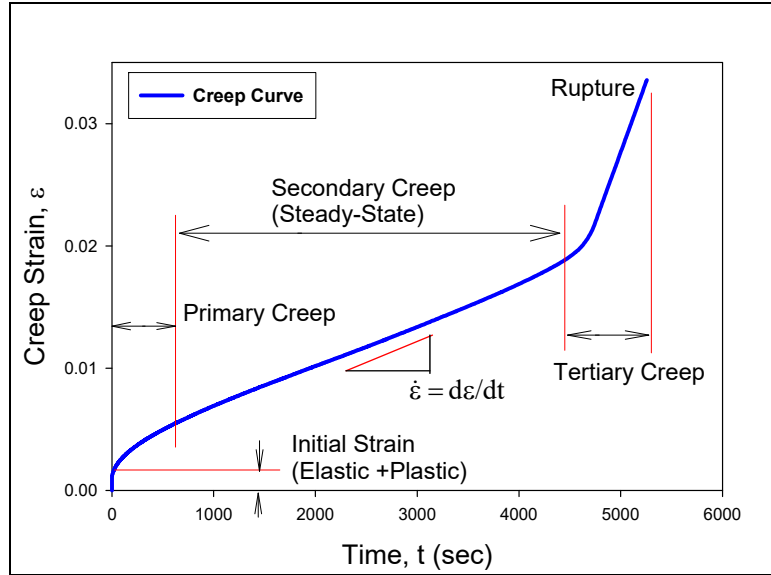


Figure 1.8 Typical Creep Curve

Every creep test begins with an initial strain which corresponds to the instantaneous response (mostly elastic) of the material due to the applied force/load. In the primary creep stage, the material starts to deform with at a high strain rate (high slope at the beginning of the primary region) and then the strain rate decrease gradually with increasing time. This is due to the work hardening of the material which resists deformation. Eventually, with increasing test time, the creep strain rate reaches to a steady state stage which is known as steady state creep or secondary creep region. The constant creep rate, in the secondary stage, is due to the dynamic balance between strain hardening and recrystallization [33]. The strain rate in the secondary stage is very important since very often researchers use this parameter in the finite element simulations to estimate reliability of the solder joints under different test conditions. After secondary creep, the material enter into the tertiary creep region followed by an immediate rupture. Tertiary region begins when the strain rate start increasing abruptly from the constant value.

1.4.3 Fatigue

Thermally cycling induced solder joint fatigue is a common failure mode in electronic packaging. When subjected to temperature changes, stresses in electronic assemblies are typically developed due to the mismatches in CTE of the soldered components and the Printed Circuit Board (PCB). Cyclic temperature changes, either due to external environment or power switching, can therefore lead to substantial alternating stresses and strains within the solder joints. During cyclic loading, micro cracks form within the solder material followed by macro cracks which leads to damage and ultimately to fatigue failure (see Figure 1.9). The facts that the original bulk design strengths are not exceeded and the only warning sign of an impending fracture is an often indiscernible internal crack, make fatigue damage especially dangerous for electronic packages.

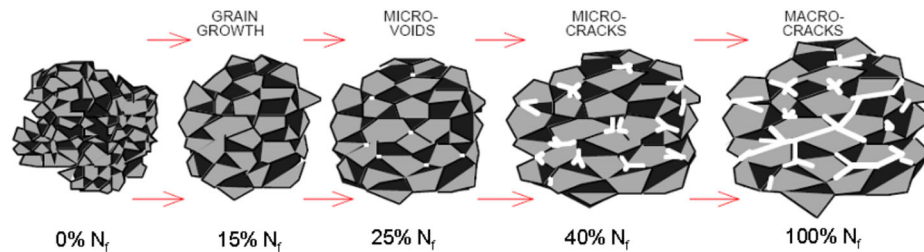


Figure 1.9 Depiction of the Effects of the Accumulating Fatigue Damage [34]

Fatigue test results are obtained by cycling smooth or notched specimens until failure, and are presented in a form of S-N diagram (where S is the stress amplitude, and N the number of cycles to failure) as shown in Figure 1.10. Since the 1950s, researchers have developed several models to estimate the number of cycles to failure of solder materials including the Engelmaier-Wild equation [34], Palmgren-Miner linear damage law, Coffin-Manson relation [35], etc.

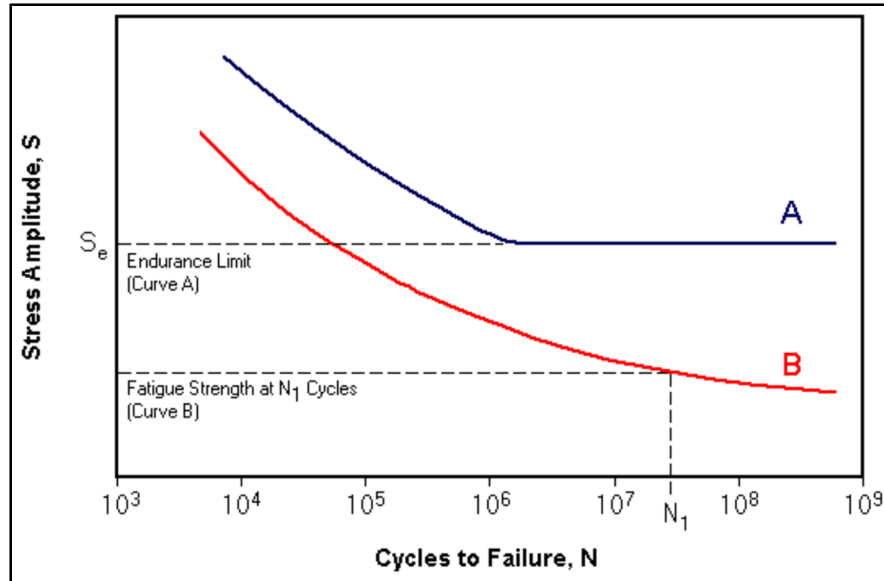


Figure 1.10 Typical S-N Curve for Ductile Materials
(<http://www.mdme.info/MEMmods/MEM30007A/properties/Properties.html>)

During fatigue damage process, slip occurs first, followed by cracks that can be seen only at high magnification. These cracks continue to grow under cycle loading and eventually become visible to the unaided eye. The cracks tend to combine such that just a few major cracks grow. These cracks (or crack) reach a critical size, and sudden fracture occurs. The higher the stress magnitude, the sooner all processes occur. Cracks may also stop without further growth as a result of compressive residual stress fields or as a crack grows out of a high-stress region such as a notch. Thus fatigue generally consists of crack nucleation, growth, and final fracture. At high stress levels, a large portion of the total fatigue life is associated with microcrack and macrocrack growth. At low stress levels, a great deal of the total fatigue life is spent in the nucleation of the crack and microcrack growth.

1.4.4 Cyclic Stress-Strain Behavior

The stress-strain behavior obtained from a monotonic tension or compression test can be quite different from that obtained under cyclic loading. This was first observed during the late nineteenth century by Bauschinger [36]. His experiments indicated that the yield strength in tension or compression was reduced after applying a load of the opposite sign that caused inelastic deformation. Therefore, one single reversal of inelastic strain can change the stress-strain behavior. Three types of cyclic stress-strain behavior can be obtained for most of metal alloys at different initial conditions [37] as shown in Figure 1.11: (a) cyclic hardening for fully annealed condition, (b) both cyclic hardening and softening for partially annealed condition, and (c) cyclic softening for cold-worked condition. The mechanism of hardening and softening can be described in terms of dislocation substructure, motion, and slip. If dislocations are constrained to move or glide and slip is minimized, the material is said to cyclically harden. Reconfiguration of the dislocation structure can also tend to promote greater dislocation mobility. Therefore, dislocations are able to circumnavigate around microstructural barriers that generally tend to restrict deformation, such as precipitates and grain boundaries. Thus, such a material is said to cyclically soften.

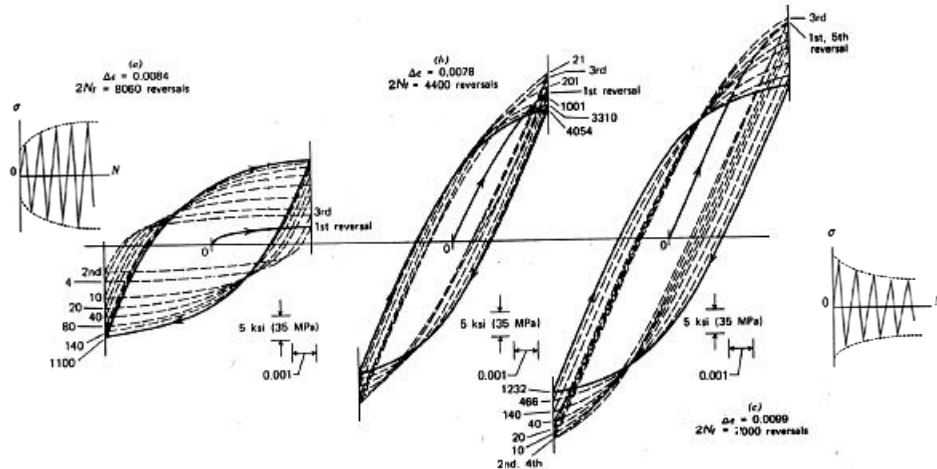


Figure 1.11 Cyclic Stress-Strain behavior of Alloys (a) Cyclic hardening (b) Cyclic Hardening and Softening (c) Cyclic softening

1.4.5 Slip, Slip Lines and Slip Bands

When a crystalline solid whose surface has been polished is plastically deformed, the surface of the solid tends to get covered with a one or multiple collections of parallel lines. These lines occur due to the sliding of crystal blocks over one another in certain directions along specified slip planes when subjected to a stress applied externally. This movement of two nearby crystal regions relative to each other is known as slip. Slip is thus a shear deformation that occurs in a material and is defined as a relationship between dislocations and inelastic deformation. Slip occurs in metals within grains due to the movement of dislocations in certain crystallographic planes associated with that metal. Slip causes the atoms to move a multiple of atomic distances along the slip planes, this motion causes a step to be produced. This step appears as a line called slip line. There are numerous amount of planes parallel to this slip plane in a crystal and the slip or the dislocation of atoms will occur in each of these parallel planes, thus producing slip lines along all these planes. The collection of these slip lines together is known as slip band.

For materials that are initially soft, the density of dislocations is initially less and any application of cyclic load that introduces an inelastic deformation in the material causes the dislocation density to grow in magnitude. This ultimately hinders the motion of the dislocation, thus cyclically hardening the material. For materials that are initially hard, application of a cyclic strain loading rearranges the dislocation structure in a manner that eases the mobility of the dislocations, thus cyclically softening the material.

1.5 Objectives of This Research

This research mainly concentrates on the effects of aging on the microstructure and the cyclic stress-strain or fatigue behavior of doped Sn-Ag-Cu (SAC) lead free solders. Also, some work will be done to better understand the damage accumulation that occurs in lead free solder balls and joints during fatigue testing. The general objectives of this research are:

- (1) Explore the mechanical cycling induced evolution of cyclic stress-strain behavior, stress-strain behavior (Elastic Modulus, Yield Stress, and Ultimate Strength), creep behavior, and microstructure of SAC305, SAC_Q and Innolot lead free solder;
- (2) Determine the effect of elevated temperature on the damage accumulation when mechanically cycling lead free solders by performing cycling tests, tensile tests, creep tests, and microstructural studies of SAC305 and SAC_Q samples cycled at 100 °C;
- (3) Investigate the effect of strain range on the cyclic stress-strain behavior of SAC and doped SAC solder alloys;

- (4) Cycle SAC305 samples (25 °C and 100 °C) at strain ranges such that the initial hysteresis loop area is same (done for 4 magnitudes) and compare the cyclic and tensile property evolution when cycling at these particular strain ranges;
- (5) Examine the aging induced changes in cyclic properties and microstructural changes occurring in small fixed regions of reflowed (RF) SAC305 lead free solder and correlate the changes with experimental results;
- (6) Conduct isothermal shear cycling on polished SAC305 and SAC_Q solder balls followed by nanoindentation tests on it to determine the reduction brought about by shear cycling in Elastic Modulus, Hardness and Creep Resistance in individual solder balls;
- (7) Investigate the effect of shear mechanical cycling (25 °C and 100 °C) on the properties and microstructure sandwiched SAC305 aged (10 days @ 125 °C) and non-aged solder joints .
- (8) Develop finite element models to estimate the plastic work accumulated when lead free solder balls and sandwiches SAC solder joints are subjected to isothermal shear mechanical cycling and be able to estimate fatigue life.

1.6 Organization of the Dissertation

This dissertation is presented in the following chapters:

Chapter 1: Introduction to lead free solders alloys and mechanical properties of solder materials.

Chapter 2: Literature review on isothermal aging effects, mechanical properties of lead free solder, cyclic stress-strain behavior, effects of aging on solder

mechanical properties, microstructural evolution during aging of SAC alloys, life estimation and reliability models of electronic packages.

Chapter 3: Description of experimental procedure, sample preparation, experimental equipment, empirical models and data processing.

Chapter 4: Evolution of mechanical properties in lead free and doped lead free solders during cyclic loading.

Chapter 5: Effect of elevated temperature cycling on the properties of lead free solder alloys.

Chapter 6: Effect of strain range on the cyclic and constitutive properties of lead free solder alloys.

Chapter 7: Aging effects on the cyclic properties of SAC305 lead free solder.

Chapter 8: Evolution of the mechanical properties of individual lead free solder balls subjected to cyclic loading.

Chapter 9: Effects of mechanical cycling in shear on lead free solder joints.

Chapter 10: Summary and conclusions of the dissertation.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Thermally cycling induced solder joint fatigue is a common failure mode in electronic packaging. Lead free solder joints in electronic assemblies are typically subjected to cyclic loadings, either in actual application or in accelerated life testing used for qualification. When exposed to extreme temperature thermal cycling, these solder joints experience a cyclic shear strain caused because of the differences in coefficients of thermal expansion of the various components of the package. Cyclic temperature changes can therefore lead to substantial alternating stresses and strains within the solder joints. Energy dissipation occurs during such cyclic loading due to yielding and occurrence of viscoplastic deformations, and the strain energy density dissipated per cycle can be calculated from the area of stress-strain hysteresis loops. The cycling eventually leads to micro cracks being formed within the solder material, followed by macro cracks which leads to damage progression and ultimately to fatigue failure. Similar effects also occur in lead free solder uniaxial samples subjected to tension and compression cyclic loading when they are exposed to isothermal repetitive mechanical cycling between two fixed strain extremes. The material damage that occurs during cyclic loading becomes immediately evident through the “load drop” and “widening” that occurs in the cyclic stress-strain curves as cycling progresses. Eventually, this damage leads to micro crack formation, crack growth, and failure. Aging of solder materials reduces their mechanical and creep properties, and will exacerbate the rate of damage accumulation during cycling loading.

As the electronic industries transition to lead free soldering by the motivation of environmental concerns, legislative mandates, and market differentiation, great efforts have been undertaken to develop desirable lead free solders and establish a corresponding database of material properties. Many researchers have attempted to measure the key mechanical properties of lead free solders. However, large discrepancies have been found in the published data from various groups. There are several reasons for these discrepancies. Firstly, the differences in specimen preparation methods among the researchers cause different microstructures in the specimens that directly affect the experimental results significantly. Secondly, the testing methods and the test conditions may also be different which will again affect the results. Thirdly, mechanical properties obtained from the bulk solders might be different from the measured properties of solder joints. Finally, the lack of standardization in the data acquisition and processing of mechanical properties makes it difficult to obtain good laboratory-to-laboratory comparisons. Apart from the above mentioned reasons for the discrepancies in solder material properties, another critical factor is aging effects. Aging is mostly neglected in the majority of prior studies, which will further exacerbate these problems. Thermal aging causes changes in the microstructure and hence the mechanical response of lead free solders. Changes in mechanical response include reductions in the initial effective elastic modulus, yield stress, and ultimate strength, as well as increased creep rates. Solder joints with highly degraded microstructure and material properties are so vulnerable that the service life of the package is often severely shortened.

2.2 Effects of Isothermal Aging on Solder Mechanical Properties

Several studies have been performed on reduction of solder material properties when the alloys are exposed to isothermal aging. For example, Ma, et al. [38] studied the evolution of Young's modulus, yield stress, and ultimate tensile strength of SAC305 and SAC405 solder alloys under various aging conditions. A linear-exponential model was developed to describe the material property evolution. They have shown that the material properties decreased dramatically in the first 20 days for both room temperature aging as well as elevated temperature aging. After 20 days of aging, the properties change slowly and linearly and it continues for longer aging time. Zhang, et al. [39] also studied the aging effects on tensile properties of SACN05 (N = 1%, 2%, 3% and 4% silver) series solders for different amount of aging at temperatures 25-125 °C. They have demonstrated that the mechanical properties reduced more dramatically when the aging temperature was increased. The data also shows that the reduction becomes linear with longer aging time. Cai, et al. [40] have also shown that the aging effects are significant for lead free solders (SAC105, SAC205, SAC305 and SAC405) for room temperature aging as well as elevated temperature aging. They have also shown that the aging effects can be reduced by using certain dopants to (e.g. Bi, In, Ni, La, Mg, Mn, Ce, Co, Ti, Zn, etc.) SAC solder alloys to enhance the reliability of lead free solders. Che, et al. [41] have measured stress-strain curves for various SAC alloys at different strain rates, while Zhu, et al. [42] have measured the mechanical properties of SAC305 at different strain rates and temperatures. The mechanical properties of a lead free solder are strongly influenced by its microstructure, which is controlled by its thermal history including solidification rate and thermal aging after solidification. Due to aging phenomena, the microstructure, mechanical response, and

failure behavior of lead free solder joints in electronic assemblies are constantly evolving when exposed to isothermal aging and/or thermal cycling environments. Such aging effects are greatly exacerbated at higher temperatures typical of thermal cycling qualification tests. However, significant changes occur even with aging at room temperature [42].

Aging effects on the constitutive and failure behaviors of lead free solders have been studied extensively by several prior researchers [19, 38-51]. In early investigations, the mechanical properties and creep behavior of SAC alloys were shown to be severely reduced by prior exposure to room temperature (25 °C) and elevated temperature (50, 75, 100, and 125 °C) aging [19, 38-42, 48, 50, 51]. The measured stress-strain data demonstrated large decrease in stiffness, yield stress, ultimate strength, and strain to failure (up to 50%) during the first 6 months after reflow solidification. After approximately 10-20 days of aging, the lead free solder joint material properties were observed to reduce at a slow but constant rate. In addition, even more dramatic evolution was observed in the creep response of aged solders, where up to 10,000X increases in the secondary creep rates were observed for aging up to 6 months. The aged solder materials were also found to enter the tertiary creep range (imminent failure) at much lower strain levels than virgin solders (non-aged, tested immediately after reflow solidification). Mustafa, et al. [46] have demonstrated that the hysteresis loop area in cyclic (tension/compression) loading of various SAC solder alloys changes significantly with aging. For strain controlled tests, the hysteresis loop area decreases and for the stress controlled tests, the loop area increases with aging time. Using the developed approaches, the influence of aging on the fatigue life of lead free alloys was also examined [47]. Zhang and coworkers [43, 48, 49] have shown that large reductions in the thermal cycling reliability of BGA test assemblies occurs when

they are subjected to aging prior to accelerated life testing. Motalab, et al. [44, 45] have included aging effects in the Anand constitutive model and energy density based failure criterion for SAC solders, and then used these theories with finite element analyses to estimate the thermal cycling life of aged BGA assemblies. Good correlations were achieved with the measured lifetimes from references [48, 49]. Other groups have also noted the strong effects of aging, and extensive listing of references can be found in [19, 38-51].

2.3 Constitutive Modelling for Solder Models (Anand Model)

In the electronic packaging industry, it is important to be able to make accurate estimations of board level solder joint reliability during thermal cycling exposures. The solder material constitutive law plays an important role in the development of thermo-mechanical models for microelectronic assemblies. Under thermo-mechanical loading, the solder material undergoes elastic and in-elastic deformations. Elastic deformations are recoverable, while inelastic deformations consist of time-independent plastic deformations and time-dependent creep deformations, which are not recoverable. Solder constitutive behavior can be represented by a combination of elastic, plastic (isotropic or kinetic hardening), and viscoplastic/creep models. The linear elastic region in a uniaxial stress-strain curve can be modeled by Hooke's law where stress and strain are related by an elastic modulus (E). The plastic strain hardening region can be modeled by a time-independent non-linear stress-strain relationship based on either isotropic or kinematic strain hardening. Isotropic hardening assumes that the origin of the von Mises yield surface remains stationary in the stress space and the size of its yield surface expands resulting from strain hardening. In kinematic hardening, the von Mises yield surface does not change in size,

but the origin of the yield surface is allowed to translate in the stress space to model strain hardening effects of increasing plastic flow stress. For solder materials, the tensile stress and strain curves are dependent on the test temperature and strain rate. The elastic modulus (E), yield stress (YS) and the tensile strength (UTS) properties vary with temperature and strain rate.

The Anand viscoelastic constitutive model is often used to represent the material behavior of the solder in finite element simulations. This model is defined using nine material parameters, and the reliability estimation results are often highly sensitive to the Anand parameters. The viscoplastic constitutive equations proposed by Anand [52] have become popular for rate-dependent deformation of metals at high temperatures. They were initially developed for structural metals, but have been adopted for microelectronic solders (Sn-Pb and lead free) for homologous temperatures in excess of $0.5T_m$. The so-called Anand model has been widely applied for the solder stress-strain relations in finite element simulations of electronic packages, where solders undergo small elastic deformations and large viscoplastic deformations. For example, Che, et al. [53] have considered multiple constitutive theories, and then demonstrated that the Anand equations were well matched with estimations of lead free solder fatigue life. In addition, Pei and coworkers [54] have calculated the nine parameters of the Anand model by conducting tensile testing of Sn3.5Ag and Sn3.8Ag0.7Cu lead free solders at several temperatures and strain rates.

Mysore, et al. [55] have found the Anand material parameters for SAC305 (Sn3.0Ag0.5Cu) lead free solder alloy by performing double lap shear tests. They suggested that the Anand parameters for solder joint samples are significantly different than those measured by testing of bulk solder specimens. In addition, Motalab, et al. [56]

have evaluated the Anand parameters for SAC305 solder using two approaches, including stress-strain testing and creep testing. Bai and coworkers [57] suggested a modified approach for SAC305 (Sn3.0Ag0.5Cu) lead free solder where strain rate and temperature dependent values were included for Anand model parameter h_0 . The Anand model constants for SAC105 (Sn1.0Ag0.5Cu) lead free solder have been reported by Amagai, et al. [58] and Kim, et al. [59]. Finally, Basit et al. reported the Anand model constants for SACN05 (N=1, 2, 3, 4) lead free solder, different solidification profile and aging effects [60].

2.4 Reduction of Aging Effect by Dopant

Addition of 4th element in the SAC solder is known as doped SAC alloy. Dopants (microalloy additions) play an important role to control microstructure and mechanical properties of the alloy. Dopants have been found to strongly influence the properties and behaviors of lead free solders. For example, addition of Bismuth (Bi) as a dopant has been demonstrated to have several beneficial effects. Bi helps to reduce solidification temperature, increases strength by means of precipitation hardening, and also helps to reduce IMC (Intermetallic Compound) layer thickness in lead free solder materials [61]. The Effect of Bi on the mechanical properties of a SAC (Sn3.5Ag0.9Cu) alloy was investigated by Matahir and coworkers [62]. They reported that the shear strength increased with increasing Bi addition up to 2 wt%. Beyond that point, the shear strength decreased with increasing Bi%. Improved shear strength might attribute to the role of Bi on the morphology of microstructure and distribution of dominant IMC (Ag_3Sn). Decrease of strength at higher Bi content was due to the evolution of Bi rich phase and fragmentation

of the IMC. Pandher, et al. [63] also reported that addition of 2% Bi in SAC alloys improves wetting and alloy spreading.

Zhao, et al. [64] found that addition of 0.02% Ni to SAC105 increased the formation of NiCuSn IMC and reduced the localized grain size at solder/NiAu pad interface. In addition, the effects using various doped elements (i.e. Co, Fe, In, Ni, Zn and Cu) in SAC305 BGA solder joints on Cu pads were studied by Sousa, et al. [65]. They concluded that addition of low levels of Zn had a significant beneficial effect on the interfacial IMC. Lee and coworkers [66] found that micro-alloying SAC alloys with Ni and Bi improved thermal fatigue life and drop impact resistance. Yeung, et al. [67] studied a novel lead-free solder SACQ. Based on drop test, thermal cycling, and finite element simulation, they conclude that the doped alloy has improved board level reliability when compared to SAC105.

Sun, et al. [68] reviewed the effects of different alloying elements (Mn, Fe, Bi, Ni, In, Zn, Ga, Sb, Mg,) Rear Earth (RE) Elements (Ce, La, Y, Er, Pr, Nd, Yb,) and nanoparticles (Al_2O_3 , Al, TiO_2 , ZnO, ZrO_2 , CNT, Graphene, CeO_2 , TiB_2 , Ni-Coated CNT, Mo, SiC, SrTiO_3 , Co) on melting temperature, wettability, mechanical properties, microstructure, interfacial reaction and Sn whiskers.

For SAC305 solder, average width of eutectic region was found as $6.8 \pm 2.8 \mu\text{m}$ and grain size of β -Sn was $24.8 \pm 5.9 \mu\text{m}$. Indium (In), helps to refine IMC and Sn-rich phase as well as makes the microstructure more uniform. Titanium (Ti) can significantly reduce Sn grain size and width of eutectic region by heterogeneous nucleation of IMC's. Iron (Fe) forms large FeSn_2 IMC which has a weak interface with β -Sn matrix. Magnesium (Mg) helps to coarsen eutectic region. Addition of Al in SAC105 refines β -Sn dendrites and

enlarge eutectic regions. Besides, it also prevent Ag_3Sn and Cu_6Sn_5 and forms two new IMC Ag_3Al and Al_2Cu . Zn also helps to refine $\beta\text{-Sn}$ dendrites significantly. Ni was found to reduce the size of Sn-rich phase and refine the microstructure. Since Antimony (Sb) has higher affinity towards Sn, presence of Sb reduces the driving force to form Cu-Sn IMC's resulting a narrow IMC layer in the solder joint. Sb also helps to refine IMC grain size.

RE elements can significantly refine the microstructure of SAC solders. Er can reduces the particle size of Ag_3Sn and Cu_6Sn_5 whereas Pr and Nd refine $\beta\text{-Sn}$ dendrites and IMC particle size by forming uniformly dispersed fine RESn_3 . These fine particles act as heterogeneous nucleation sites during solidification. However, excessive amount of RE elements will cause to form bulk RESn_3 phase which has a negative effect on mechanical properties. La, Ce and Y also have a similar effect on solder microstructure.

Al and Ni nanoparticle was found to reduce IMC particle size, spacing and IMC layer thickness by forming very fine and uniformly dispersed Sn-Ni-Cu and Sn-Sg-Al IMCs. Addition of small amount of Fe nanoparticles refine the microstructure and forms FeSn_2 phase. Al_2O_3 nanoparticles increase the size of eutectic region and reduce Ag_3Sn particle size. TiO_2 and SiC nanoparticle reduces the size and spacing between Ag_3Sn particles. SrTiO_3 nanoparticle reduces the size of Ag_3Sn and Cu_6Sn_5 particles by promoting the rate of nucleation during solidification. ZnO suppresses Ag_3Sn and Cu_6Sn_5 IMC formation and reduces $\beta\text{-Sn}$ grain size by 22%.

The Effect of dopants on the aging induced changes in microstructure was also studies by a number of scientists. Sadiq, et al. [69] worked with different Lanthanum (La) doped SAC305 alloys and recorded the changes in microstructure and mechanical properties during isothermal aging at 150 °C for 6 different aging conditions (i.e. 0 , 10,

25, 50, 100 and 200 hours). They reported that La drastically reduces the IMC particle size and also significantly inhibit the growth of IMC particles during isothermal aging. Based on polarized light image they found that in as cast condition, grain size of SAC305 was ~8 mm and was significantly reduced (~1 mm) after La addition. From the graph presented in that paper, it is also clear that aging doesn't have any significant influence in average grain size.

In another study, Lee, et al. [70] studied the effect of Lanthanum (La) addition and high temperature storage on the microstructure and microhardness of Sn-3.5Ag solder joints. Their experimental results confirms that addition of La refine the solder microstructure. They explained that during solidification of the solder, LaSn_3 compounds form at the beginning and provide extra nucleation sites for Ag_3Sn IMC to grow resulting a refine microstructure. Addition of La was also found to reduce the thickness of IMC layer after soldering as well as isothermal aging. They also reported that La addition helps to improve microhardness and thermal resistance of solder joints.

Hao, et al. [71] studied the effects rare earth element Er addition on the evolution of microstructure of lead free eutectic SAC (Sn-3.8Ag-0.7Cu) solder joints during isothermal aging. Aging was conducted at 170 °C for 4 different holding periods (i.e. 0, 200, 500 and 1000 hours). The authors measured the thickness of IMC layer of Sn-3.8Ag-0.7Cu and Sn-3.8Ag-0.7Cu-0.15Er alloy after different aging duration. They found that Er addition reduces the thickness of IMC layer in as reflowed condition and also significantly reduces the growth during aging. They argued that Er combines with Sn to form ErSn_3 IMC and reduces the activity of Sn which subsequently suppress the formation of Cu_6Sn_5 IMC layer. They also observed that ErSn_3 IMCs formed during solidification of solder act

as a heterogeneous nucleation site for Ag_3Sn and Cu_6Sn_5 precipitates. The increase in nucleation sites results a refinement of Ag_3Sn and Cu_6Sn_5 particles. Addition of Er also found to make the microstructure more uniform and reduce the coarsening rate of the IMCs during isothermal aging.

Witkin [72] and Delhaise et al. [73] studied the effect of aging of Bi doped SAC alloys. In both study, the authors reported an elimination or at least decline of aging induced reduction in SAC-Bi alloys.

2.5 Cyclic Stress-Strain Behavior of Solder Materials

The stress-strain behavior obtained from a monotonic tension or compression tests can be quite different from that obtained under cyclic loading. This was first observed during the late nineteenth century by Bauschinger [36]. His experiments indicated that the yield strength in tension or compression was reduced after applying a load of the opposite sign that caused inelastic deformation. Thus, one single reversal of inelastic strain can change the stress-strain behavior of metals. In the early 1960s, Morrow [37] reviewed several experimental techniques and methods for interpreting results and also illustrated typical phenomenological cyclic-dependent deformation and fracture behavior. He observed that a stable hysteresis loop quickly developed under cyclic conditions. Based on this observation, he also developed a descriptive theory of fatigue based upon cumulative plastic strain energy as a criterion for fatigue damage, and elastic strain energy as a criterion for fracture. He demonstrated quantitatively that the fatigue properties of a metal can be related to its cyclic stress-strain properties.

Many prior researchers have studied the cyclic stress-strain behavior of solder materials. Models to estimate fatigue life of solder alloys under thermal cycling or thermomechanical fatigue loading have been developed in several investigations [74-81]. For example, Guo, et al. [77] investigated the evolution of the hysteresis loops for both thermomechanical fatigue and isothermal fatigue for 63Sn-37Pb solder, and also compared the hysteresis loops found at various failure cycles. They performed mechanical cyclic tests at 25 °C and 80 °C under strain control for strain limits of 0.3% to 3%. For thermal cycling, they tested between 25 °C and 80 °C, with a 120 second ramp. They also developed a mathematical correlation between the sizes of the isothermal hysteresis loop and thermal cycling hysteresis loop. Busso, et al. [74] studied cyclic stress-strain behavior of 60Sn-40Pb solder using cyclic torsion tests performed on a feedback controlled servo-hydraulic testing machine. They also developed a viscoplastic constitutive model that included a strong Bauschinger effect exhibited by solder during cyclic loading. Frear, et al. [76] observed that under conditions of cyclic strain, the microstructure of 60Sn-40Pb alloy experienced a heterogeneous coarsening. They then developed a microstructure based finite element simulation methodology for the estimation of solder joint fatigue life. Yang, et al. [81] investigated cyclic stress-strain behavior of 63Sn-37Pb solder under triangular waveform, trapezoid waveform, and multi-level dwell time waveform cyclic loading.

Measurement of deformations during thermal cycling and stress-strain hysteresis in solder joints have also been examined. For example, Hall [82, 83] experimented the stress-strain hysteresis of a leadless ceramic chip carrier (LCC) mounted to an organic PCB during the thermal cycling. Another research conducted by Pao [84-86] evaluated the thermo-mechanical hysteresis of different Sn-based alloys using a 2-beam geometry. In

addition, Haacke, et al. [87] characterized both the thermomechanical fatigue and hysteresis for Sn-Pb solder. For lead free solders, Raeder and co-workers [88] have investigated the thermomechanical deformation behavior of Sn-Bi eutectic solder under cyclic loading, and the findings were quantified using the plastic strain energy density. Later, Dusek, et al. [89] explored the cyclic stress-strain behavior and hysteresis loop evolution for Sn-Ag-Cu (SAC) lead free solders during isothermal fatigue at several different temperatures ($T = 25, 30, 60, \text{ and } 125\text{ }^{\circ}\text{C}$) and found that the number of cycles to fatigue failure increased with the increment of temperatures for the same stress level. They have also claimed this phenomenon attributed to the use of slow cycling, where creep and stress relaxation play dominant roles. Hu, et al. [90] reported that under constant strain range, the stress range decreased during cyclic testing of Sn-Pb solder. They also observed the fifth hysteresis loops at $22\text{ }^{\circ}\text{C}$ for total strain ranges of 1%, 2%, and 5%, and reported that with increasing strain range, the area of the hysteresis loops increased. The stress range was found to have decreased by 85% as the temperature was increased from $22\text{ }^{\circ}\text{C}$ to $50\text{ }^{\circ}\text{C}$. Shi, et al. [91] studied the effects of frequency and temperature on hysteresis loops during cyclic loading. They reported that the area of the hysteresis loops reduced with a decrease of the frequency from 1 Hz to 0.0001 Hz, and that the area of hysteresis loop also decreased with the increasing testing temperature from $25\text{ }^{\circ}\text{C}$ to $150\text{ }^{\circ}\text{C}$. Kanchanomai, et al. [92] have performed uniaxial fatigue tests on Sn-Ag solder, while Pang and coworkers [93] have performed uniaxial fatigue tests on SAC387 lead free solder. Mustafa, et al. [46] investigated the aging induced evolution of the cyclic stress-strain and fatigue properties of bulk lead-free SAC solder specimens.

2.6 Isothermal and Thermomechanical Fatigue Criteria for Solder Materials

Several previous studies have been conducted on the fatigue behavior of lead free solders. Herkommer, et al. [94] have developed a damage model that is capable of estimating material behavior under both mechanical shear cycling and thermal cycling loading conditions. Zhang and Dasgupta [95, 96] have discussed the mechanical and thermal cycling durability of selected lead-free solders (Sn3.9Ag0.6Cu, Sn3.5Ag, and Sn0.7Cu), and developed a mechanical fatigue damage criterion. They demonstrated good correlation between estimations of their criterion and experimental cycles to failure under different strain rates and temperature conditions. Whitelaw, et al. [97] have determined the parameters of the Bonder-Partom model from uniaxial cyclic stress-strain tests for both lead-free and Sn-Pb solders. They have also verified their model under isothermal mechanical cycling conditions. Korhonen, et al. [98] conducted uniaxial cyclic tests at various temperatures for near eutectic Sn-Ag-Cu alloy to understand the isothermal fatigue behavior. Nucleation and growth mechanisms of fatigue damage were also observed using optical microscopy, scanning electron microscopy (SEM), and the Electron Back-Scattering Diffraction (EBSD) method. Fatigue failure behavior and cyclic creep deformation were observed by Shang, et al. [99], and they related the fatigue behavior to cyclic stress amplitude, number of cycles, stress ratio, frequency, temperature, alloy composition, and microstructure for different lead-free solders. Both uniaxial cyclic tests and shear cyclic tests were carried out by Andersson, et al. [100] to establish a comparison between the isothermal mechanical fatigue properties of lead free solder joints and bulk solders. The fatigue ductility exponent in the Coffin-Manson law for a Sn-Ag-Cu micro-solder joint was investigated by Kanda, et al. [101] in terms of the cyclic strain-hardening

property and inelastic strain energy in fracture for isothermal fatigue. They established a relationship between the equivalent inelastic strain range and the fatigue life under triangular and trapezoidal waves for both as-soldered and aged specimens. Shang, et al. [99] reviewed the recent fatigue studies of Sn-rich Pb-free solder alloys to provide an overview of the current understanding of cyclic deformation, cyclic softening, fatigue crack initiation, fatigue crack growth, and fatigue life behavior in those alloys. Fossum, et al. [102] proposed a practical viscoplastic damage model to characterize 95.5Sn-3.9Ag-0.6Cu lead free solder under cyclic thermomechanical loading conditions. Isothermal low cycle mechanical fatigue tests of solder joints for 63Sn-37Pb, Sn-3.7Ag, Sn-4.0Ag-0.5Cu and Sn-8Zn-3Bi solders were performed by Andersson, et al. [103]. They found that Sn-4.0Ag-0.5Cu lead free solder exhibited the best isothermal fatigue properties. Kanchanomai, et al. [92] performed low cycle fatigue tests of Sn-3.5Ag eutectic lead free solder under various temperatures and frequencies, and then used three different estimation models including the Coffin-Manson model, Smith-Topper model, and Morrow model. Lau, et al. [104] investigated the thermal cycling fatigue life of lead-free solder joints in a plastic ball grid array (PBGA) package assembly. They concluded that the thermal-fatigue life of the solder joints would be over-estimated based on a pure shear theory. In addition to pure shear, bending, twisting and expansion/contraction deformations were needed to be included. Pang, et al. [93] investigated isothermal low cycle fatigue for 95.5Sn-3.8Ag-0.7Cu bulk solder specimens over a range of test temperatures (-40, 25, 75 and 125 °C) and frequencies (1Hz, 0.01 Hz and 0.001 Hz). They proposed frequency modified Coffin-Manson model and Morrow model relationships for 95.5Sn-3.8Ag-0.7Cu lead free solder at 125 °C and over the entire range of test frequencies.

2.7 Effects of Thermomechanical Cycling on Solder Properties

The effects of mechanical cycling on the microstructure of eutectic Sn-Pb and Lead free solder joints have been explored by several researchers. Chen, et al. [105] studied the microstructure evolution of Sn-Ag-based solder interconnects during thermal cycling. They noted during the cycling, recrystallization was observed and Ag_3Sn IMC particles segregated in those regions. Berthou, et al. [106] investigated microstructure of SAC joints when subjected to thermal cycling and thermal loading, and stated that huge stresses were developed in areas where tin grain recrystallization occurred. This resulted in the joints failing by thermomechanical cycling fatigue. During thermal cycling, Yin and coauthors [107] noticed that recrystallization of β -Sn phases occurred, and there was coarsening of precipitates in the solder joints. Coarsening of the IMC particles during cycling has also been observed by other researchers [108-110]. Thermal cycling has also been found to induce significant changes in the mechanical properties of SAC solders due to damage accumulation [108-114]. Changes in elastic modulus [113], hardness [111], reliability [114], creep behavior [108, 109], and constitutive response [112] have been reported.

Matin, et al. [115, 116] studied the effects of both mechanical and thermal loadings on SAC solder joints using Optical Microscopy (OM) and Scanning Electron Microscopy (SEM). It was observed that for thermal loads, damage propagated through the grain boundaries, while for the pure mechanical load, the damage was seen to have occurred between slip bands and eutectic regions in the form of micro-cracks and voids. Many researchers have also investigated the evolution of mechanical properties of solder joint materials under thermal cycling or mechanical cycling conditions. Chen, et al. [105] reported that the hardness of SAC305 decreased by about 39% and 20%, respectively, in

the recrystallized and non-recrystallized regions after 2815 thermal cycles between 0 °C and 100 °C. Dutta and coworkers [108, 109] studied the effects of TMC induced microstructural coarsening on the creep behavior of lead free solders. They reported that the creep rate increases with the precipitate size proportionately when the stresses are low. On the other hand, precipitate coarsening changes the threshold stress for particle-limited creep when the stresses are high. Erinc, et al. [113] found the global elastic modulus was declining for the three SAC405 samples of different thickness as the thermal cycling progressed (-40 °C to 125 °C). Furthermore, a significant reduction in board-level drop reliability with increased thermal cycling was reported in [114].

2.8 Effects of Aging on the Microstructure of Solder

Due to their low melting temperatures, solders are exposed to high homologous temperatures in most product applications. Thus, there is a continuous state of active diffusion processes in the solder alloys, and their microstructures are inherently unstable and will continually evolve during normal operating temperature conditions of electronic packaging assemblies. High temperature storage, which is also known as isothermal aging, of the solder alloys causes a significant change in the microstructure leading to a degradation of mechanical properties. In recent days, electronic industries are moving towards lead free solders due to the growing concern about environment. Effect of aging in lead free solder is even more significant. Sahaym et al. [117] examined the evolution of microstructure of bulk SAC105 and SAC305 solder during isothermal aging at 150 °C. They observed the changes of identical region after 4 different aging durations (0 hr., 110 hr., 194 hr., and 310 hr.). They reported that after 310 hours of aging, the average size of IMC precipitates has increased from 0.35 μm to 2.5 μm and the average grain size has

increase from 4.5 μm to 7.5 μm . The authors also observed that a small percentage ($\sim 10\%$) of $\beta\text{-Sn}$ grains, especially those near the eutectic region, has went through recrystallization during isothermal aging. They claimed the stress on the $\beta\text{-Sn}$ grains, due to the growth of IMC particle, is responsible for the recrystallization. The extent of recrystallization was less in SAC105 than in SAC305 due to the relative difference in IMC volume fraction.

Maleki et al. [118] studied the evolution of microstructure of SAC405 and pure Sn during isothermal aging at 150 $^{\circ}\text{C}$ after 144 hours and 296 hours. They also performed Mechanical testing (shear test) to correlate changes in microstructure and mechanical properties. Sample size was approximately $1 \times 0.3 \times 0.3$ mm and it was attached to Cu-pad. They reported that in as-reflowed condition, the microstructure of SAC405 was consisted of ~ 70 vol% of eutectic phase and ~ 30 vol% of $\beta\text{-Sn}$ dendrite, whereas the mean diameter of IMC particles and interparticle distance was ~ 250 nm and ~ 630 nm, respectively. Average particle size increases with aging time due to Ostwald ripening. During IMC growth, they attributed bulk diffusion to be the main rate controlling mechanism. On the other hand, aspect ratio decreases and interparticle spacing increases with aging due to the driving force to reduce surface energy. Electron backscatter diffraction (EBSD) analysis confirmed the presence of large grains (~ 200 μm) in SAC405 solder before aging. After 296 hours of aging at 150 $^{\circ}\text{C}$ they didn't find any significant change in grain size and orientation. On the other hand, pure tin has a fine grain (~ 10 μm) microstructure before aging which grows significantly during aging and become ~ 200 μm after aging. They attributed the reduction of mechanical properties during aging for SAC 405 to the IMC's coarsening whereas for pure Sn to the grain coarsening.

Telang et al. [119] worked on the effects of aging at 150 °C on microstructure, especially grain size and grain-boundary misorientation, of several alloys including Pure Sn (ingot and reflowed), Eutectic Sn-3.5Ag (ingot), Eutectic Sn-3.8Ag-0.7Cu (ingot), Sn-1.6Ag (solder ball), Sn-3.0Ag (solder ball), and Sn-3.0Ag-0.6Cu (solder ball). For Sn-3.5Ag alloy they studied three different aging conditions (i.e. 0, 200, and 400 hours.) whereas for rest of the alloys they studied two different aging conditions (i.e. 0. and 200 hours). Before aging, grain size of pure Sn ingot and Reflowed Pure Sn was 50-150 μm (equiaxed) and 100-250 μm (equiaxed), respectively. After aging for 200 hours, the grain size of pure Sn for both condition was $>500 \mu\text{m}$ with irregular shape. After comparing the microstructure of eutectic Sn-Ag (3.5% Ag) ingot with pure Sn ingot, they found that Sn-Ag alloy has a much finer (10-30 μm) grain size, which was very stable. Aging of Ag-Sn alloy did not cause any significant grain growth due to the pinning effect of Ag_3Sn IMC particles. On the other hand, the grain size of SAC (Sn-3.8Ag-0.7Cu) ingot was similar to that of Sn-Ag ingot (10-30 μm). But, aging for 200 hours caused a significant growth in the grain size ($\sim 120 \mu\text{m}$) of the alloy. From their experimental observations the authors also have discussed the effect of Ag and Cu on preferred grain orientation and grain size.

Allen et al. [120] studied two near eutectic lead free SAC solders (bulk Sn-3.5Ag-0.9Cu and SAC405 joint). They used 3 different aging temperatures (152, 177, and 201 °C) and 5 different aging durations (0, 1, 2, 4, and 8 weeks). They measured the density of IMC particle after different aging conditions and based on their experimental results they conclude that the rate controlling mechanism for coarsening is volume diffusion ($n=3$). They also reported that the coarsening kinetics of eutectic SAC solder is slower than that of eutectic Sn-Pb solders. Kumar et al. [121] worked with SAC105 and SAC305 alloys.

For isothermal aging experiments, they polished a bulk reflowed solder samples and then aged the samples at 150 °C in high vacuum (to prevent oxidation). After different aging intervals (i.e. 0, 110, 194, 220, and 330 hours) they captured the SEM image of the same region, of any particular sample, to quantify the coarsening behavior of the IMC's. They found that the growth rate of Cu_6Sn_5 particles are much faster than Ag_3Sn particles, due to the higher diffusivity of Cu than Ag in Sn matrix. Besides, the fraction of Ag_3Sn particles was significantly higher than Cu_6Sn_5 particles. Hence, they decided to focus their study on the coarsening of Ag_3Sn particles only. They estimated coarsening rate of Ag_3Sn particles, during isothermal aging, and then compared with the experimental observations. They conclude that the model could efficiently measure thermomechanical history during isothermal aging and thermomechanical cycling (TMC) below 200 cycles. After 200 cycles the IMC particles undergo dissolution and re-precipitation/redistribution which can't be captured properly by this model. In addition to coarsening, during isothermal aging, they also had observed recrystallization near the eutectic region. They explained that the growth of IMC particles might play an important role in recrystallization.

Besides bulk solder samples, researchers have also explored the effect of aging on the actual solder joints. Chauhan et al. [122] monitored the effect of isothermal aging at 100 °C on phase coarsening and evolution of SAC305 solder joint. They used image processing software to quantify size, interparticle spacing and volume fraction of Ag_3Sn and Cu_6Sn_5 IMC's. Impact of these changes on secondary creep response was modeled using multiscale creep model. The authors performed their experiments in 4 different aging conditions (0, 24, 600, and 1000 hours.) and presented average results of 3 samples (solder joint) for each aging condition. They found that the size of Ag_3Sn increases monotonically

with increasing aging time whereas the size of Cu_6Sn_5 decreased after 600 hours of aging. They didn't find any significant change in Sn grain morphology after 1000 hours (41 days) of aging.

Yang et al. [123] prepared solder joints using 2 different soldering methods (laser and infrared soldering) and captured the evolution of microstructure during aging up to $190\text{ }^\circ\text{C}$ for times up to 300 days. They reported that the evolution is consisted of the growth of IMC's and the Cu-Sn layer near the interface. Although they had studied two different initial microstructure, obtained from 2 different soldering methods, aging at $190\text{ }^\circ\text{C}$ caused the final microstructures to be the same. Chiu et al. [124] examined the effect of aging time and temperature on the board level reliability during the drop test of lead free SAC solder joints. They reported that Kirkendall void formation, at the interface of Cu pad and Cu_3Sn IMC, is the main reason for getting lower drop reliability during drop test. Fix et al. [125] explored the effect of aging time (0 to 1000 hours) and temperatures (125 to $175\text{ }^\circ\text{C}$) on the microstructure of SAC405 solder joint. They have confirmed a significant growth of Ag_3Sn and Cu_6Sn_5 IMCs with aging time. The authors modeled phase growth based on Ostwald ripening mechanism. They conclude that a growth exponent $n = 3$, which indicated volume diffusion as the growth rate controlling mechanism, matches very well with the experimental growth rate data. The effect of aging at $150\text{ }^\circ\text{C}$ on the evolution of eutectic Sn-Ag solder joint for up to 800 hours was studied by Ahat et al. [126]. From experimental results, they conclude that the thickness of IMC layer increases linearly with square root of aging time. Choi et al. [127] investigated the effect of different soldering and aging time on the interface layer of Sn-3.5Ag solder and Cu substrate. They used the same solder joint to age at $130\text{ }^\circ\text{C}$ for different time duration up to 800 hours. They found

that thickness of IMC layer continue to grow with increasing aging duration. However, the growth behavior of IMC layer during aging strongly depends on the initial morphology and hence on the soldering time.

Akhtar et al. [128] studied the evolution of microstructure near the interface of a solder joint during isothermal aging at 150 °C, for four different aging duration (0, 250, 500, and 1000 hours). They used SAC305 solder joint (ball diameter 500 µm) with two different surface finish (Immersion Gold (ImAu) and Immersion Tin (Sn)). Image analysis software ImageJ was used to measure the thickness of the IMC layer. They also calculated the activation energy based on the measured thickness data. They found that the thickness of the IMC layer increases with increasing aging time. Besides, the morphology of the IMC layer, for both surface finishes, change from scallop type to layer type after the aging treatment. Based on their calculations they found that the activation energy of SAC305/ImSn system was less than SAC305/ImAu system resulting a higher IMC layer growth rate in SAC305/ImSn system.

A comparative study was performed by Berthou et al. [106] where the authors compared the failure mechanism of BGA packages with SAC305 solder joints subjected to two different conditions: (a) accelerated thermal cycling (ATC) between -55 to 125 °C and (b) thermal storage at 80, 125, and 150 °C for 1000 hours. During ATC, they found recrystallization of big Sn grains to small grains in the regions of high stress accumulation. Cracks initiates and grows at interface of the new recrystallized grains. On the other hand, thermal storage for 1000 hours caused a significant growth of IMC layer near the interface as well as the IMCs in the bulk. They didn't reported any recrystallization after thermal storage.

2.9 Summary

In this chapter, the existing literature on the effects of aging on the mechanical properties and the microstructure of lead free solder was extensively discussed. The mechanical properties of a solder are strongly influenced by its microstructure, which is controlled by its thermal history including its solidification rate and thermal exposures after solidification. Aging of lead free solders leads to reductions in their constitutive and failure behaviors. For example, research in the literature has shown that aging leads to large decreases in solder material properties including shear strength, elastic modulus, nanoindentation joint modulus and hardness, high strain rate mechanical behavior, creep response, and Anand model parameters. Other studies have shown that aging causes severe reductions in uniaxial cyclic stress-strain curves and fatigue life, shear cyclic stress-strain curves and fatigue life, fracture behavior, drop reliability, and thermal cycling reliability.

Dopants have been found to strongly influence the properties and behaviors of lead free solders. For Example, Bi helps to reduce solidification temperature, increases strength by means of precipitation hardening, helps to reduce IMC (Intermetallic Compound) layer thickness, and also reduce aging induced reduction of mechanical properties in lead free solder materials. Ni helps to improve thermal fatigue life and drop test performance by refining Sn grain size and reducing the IMC layer formation near the Cu pad.

Previous studies on cyclic stress-strain behavior of solder alloys were also discussed. Several experimental investigations, and developed theories for cyclic stress strain behavior of different solder alloys were presented for various testing conditions. Studies on the dependence of hysteresis loop shape and size for different frequencies and testing temperatures were also discussed. It has been demonstrated that the area of the

hysteresis loop decreases with increasing frequency of cyclic loading, whereas the area of the hysteresis loop decreases with the increasing testing temperature. It is also reported in the literature that with increasing strain range, the area of the hysteresis loop increases. Several attempts have also been made to establish a correlation between isothermal mechanical loading hysteresis loops and thermal cycling hysteresis loops for lead free solders.

Lastly, evolution of microstructure and mechanical properties of solder materials subjected to cyclic loading were discussed. Several fatigue life estimation models such as, the Coffin-Manson model, Smith-Topper model, and Morrow model have been used by researchers. Fatigue tests have been performed for the solder alloys under strain controlled uniaxial, shear, and torsional loadings. Nucleation and growth mechanisms of fatigue damage were also observed using optical microscopy, scanning electron microscopy (SEM), and the Electron Back-Scattering Diffraction (EBSD) method. Finally, a short discussion of the literature regarding aging effects on fatigue behavior for solder materials has been presented. The changes in solder mechanical behavior are a result of the evolution of the SAC solder microstructure that occurs during aging. The most well-known and widely observed changes are coarsening of the Ag_3Sn and Cu_6Sn_5 intermetallic compounds (IMCs) present in the eutectic regions between beta-Sn dendrites. Several researchers have proposed empirical models to describe the growth of these secondary phase particles as a function of aging temperature and aging time, and related this growth to mechanical property changes.

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Introduction

The specimen preparation and testing techniques are presented in this chapter. The mechanical characterization of solder alloy is typically performed by uniaxial loading of machined or mold cast samples, or by shear loading of joint samples. In some parts of this study, mold cast samples using some high precision rectangular cross-section glass tubes have been tested under uniaxial loading to characterize mechanically. However, the thin flat specimens tend to buckle during compression in fatigue testing. In order to prevent buckling, the circular cross-section specimens were also introduced, and fixtures were designed to grip the cylindrical specimens. The mechanical testing was performed using a tension/torsion thermo-mechanical test system. After testing, empirical models were utilized to represent the stress-strain, creep, and cyclic stress-strain experimental data. For the microstructural analysis, the rectangular cross-sectional solder specimens were polished and then the microstructures were examined using SEM or polarized light microscope. Lead free SAC and SAC+X solder balls were polished and prepared. The solder balls were then subjected to nanoindentation tests and sheared for various durations. Sandwiched SAC305 solder joints were also prepared and polished to observe any changes in the grain morphology when subjected to shear mechanical cycling at either room temperature or elevated temperature.

3.2 Uniaxial Test Sample Preparation

Initially, bulk solder material was cut into pieces by a shear cutter machine. Then, the small pieces were melted in a quartz crucible using circular heating elements (Figure

3.1), which is controlled by a digital controller using the feedback from a thermocouple attached on the crucible. Using vacuum suction process, uniaxial test specimens of lead free solder alloy were formed with controlled microstructure in glass tubes. The melted solder material was sucked into the glass tube by inserting one end into the molten solder in the crucible and then applying suction to the other end using a rubber tube connected to a vacuum suction pump. A manually controlled regulator on the vacuum line regulates the amount of solder drawn into the tube. When the tube contains the desired amount of solder, it was solidified through quenching in a water bath at room temperature.

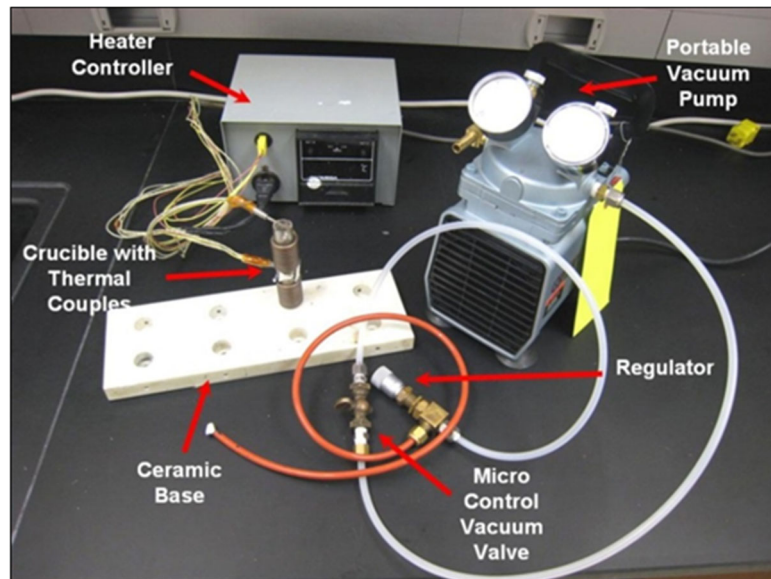


Figure 3.1 Equipment used for Specimen Preparation

After that, the tensile specimens were prepared using two different solidification profiles.

- 1) Water quenched (WQ) solidification profile, leading to fine microstructures and the upper limits of the mechanical properties for each alloy.

2) Reflowed (RF) solidification profile, leading to a coarse microstructure very similar to an actual solder joints. The solder test specimens were passed through a controlled heating and cooling chamber using a SMT (surface mount technology) reflow oven.

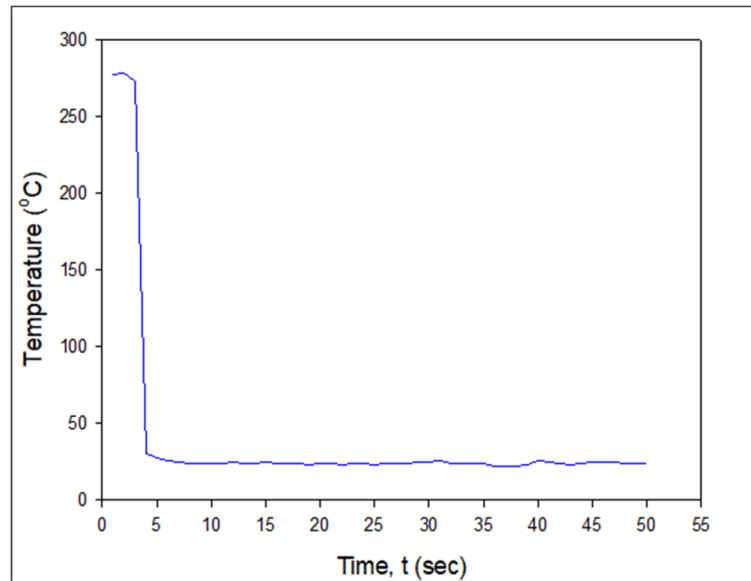


Figure 3.2 Water Quenched (WQ) Cooling Profiles

The temperature vs. time variations for the WQ profile is shown in Figure 3.2. For the samples with reflowed profile, test specimens were initially prepared using WQ profile and then the samples within the glass tubes were sent through a 9 zone Heller 1800EXL reflow oven (Figure 3.3).



Figure 3.3 Heller 1800EXL Reflow Oven

Inside the oven, solder samples were re-melted and experienced to a pre-set temperature profile which is very similar to that used for the actual solder joints. The reflow temperature profile used in this study is presented in Figure 3.4.

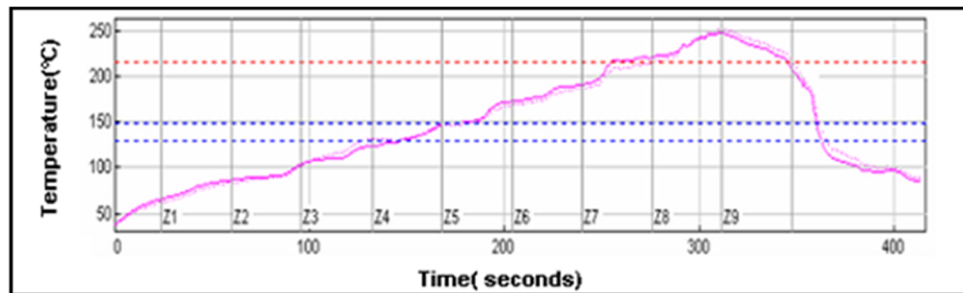


Figure 3.4 Reflow (RF) Cooling Profiles

A typical glass tubes filled with solder and final solder samples after extraction from the glass tubes are shown in Figure 3.5. Glass has lower coefficient of thermal expansion (CTE) compared to solder. As a result, for some solder alloy and cooling rate combinations, solidified solder samples were easily pulled out from the glass tube due to the difference is the CTE of glass and solder. Another way followed to extract the solder

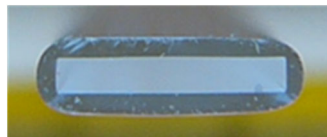
sample from the glass tube is by carefully breaking the glass. The tubes in this work had a length of 120 mm, and a cross-sectional area of 3.0 x 0.5 mm. A thickness of 0.5 mm was chosen since it matches the height of typical BGA solder joints. The nominal dimensions of the final test samples were 80 x 3 x 0.5 mm. The specimens were stored in a low temperature freezer after the water quenched/reflow process to minimize any aging effects. The solder microstructure has been verified to be consistent throughout a specimen volume, and from specimen to specimen by cross-sectioning. A micro-focus x-ray system was used to inspect the samples for the presence of flaws (e.g. notches and external indentations) and/or internal voids (non-visible). Specimens with no flaws and voids were generated using proper experimental techniques, and Figure 3.6 illustrates x-rays scans for good and poor specimens.



(a) Within Glass Tubes



(b) After Extraction



(c) Cross-Section

Figure 3.5 Solder Uniaxial Test Specimens

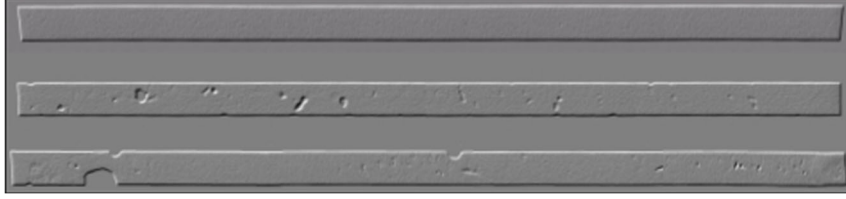


Figure 3.6 X-Ray Inspection of Solder Test Specimens (Good and Bad Samples)

3.3 Uniaxial Tensile Testing System

The tension/torsion thermo-mechanical test system (Wisdom Technology MT-200) used to perform the stress-strain tests in this study is presented in Figure 3.7. This instrument is optimized for loading small specimens such as thin films, solder joints, gold wire, fibers, etc. It provides an axial displacement resolution of 0.1 micron. Samples can be tested over a temperature range of -185 to +300 °C using supplemental environmental chambers added to the system.

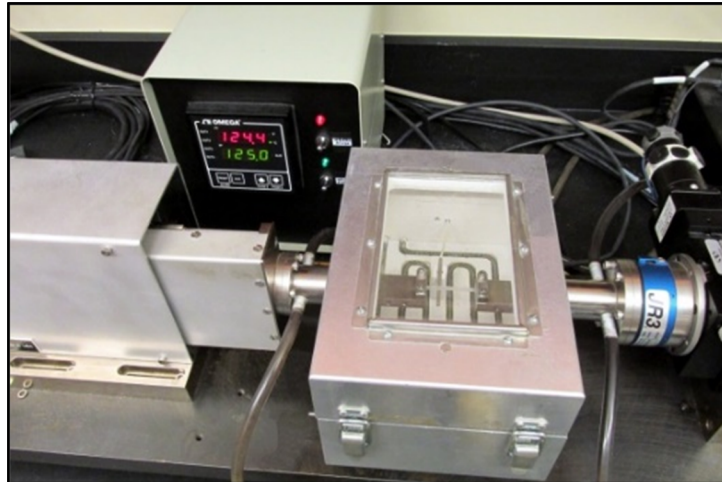


Figure 3.7 Mechanical Test System with Uniaxial Sample

Forces and displacements were measured in the uniaxial tests, and the axial stress and axial strain were calculated using

$$\sigma = \frac{F}{A} \quad \varepsilon = \frac{\Delta L}{L} = \frac{\delta}{L} \quad (3.1)$$

Where, F is the measured uniaxial force, δ is the measured crosshead displacement, σ is the uniaxial stress, ϵ is the uniaxial strain, A is the original cross-sectional area, and L is the chosen specimen gage length (initial length between the grips). In order to ensure a uniaxial stress state, the gage length of the specimen was kept as 60 mm (thus the length to width ratio was 20 to 1).

3.4 Typical Stress-Strain Data Processing

A typical SAC solder tensile stress strain curve is illustrated in Figure 3.8. The standard material properties are labelled on the graph including the effective elastic modulus E (initial slope of the stress-strain curve). This effective modulus is rate dependent since solder behavior is viscoplastic. The value of the effective modulus will become the true elastic modulus as the testing speed is increased to the limit of infinite strain rate. The yield stress σ_Y (YS) is defined using the typical definition of the stress level that results in a permanent strain of $\epsilon = .002 = 0.2\%$ upon unloading. The maximum (saturation) stress on the stress-strain curve is the ultimate tensile strength σ_u (UTS). As shown the figure, the stress-strain curve for the solder material has an elastic region at the beginning, a small transition region followed by a plastic region. As the strain becomes significantly high, localized deformation takes place which is also known as necking. Necking causes a visible reduction in cross-sectional area and a decline in the applied load, near the end of the stress strain curve, leading towards a rupture.

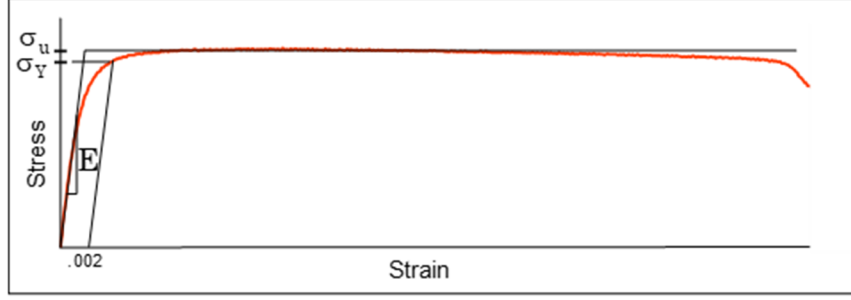


Figure 3.8 SAC Stress-Strain Curve and Material Properties

Figure 3.9 illustrates a typical set of 5 solder stress strain curves measured for the same alloy under similar environmental and aging conditions. In this work, a four parameter hyperbolic tangent empirical model has been used to represent the “average” stress-strain curve through a set of experimental data (red curve in Figure 3.9). Material constants C_1 , C_2 , C_3 , and C_4 are determined through regression fitting of the model to experimental data (Equation 3.2). The effective elastic modulus E at zero strain is calculated from the model constants using Equation 3.3.

$$\sigma = C_1 \tanh(C_2 \epsilon) + C_3 \tanh(C_4 \epsilon) \quad (3.2)$$

$$E = \sigma'(0) = C_1 C_2 + C_3 C_4 \quad (3.3)$$

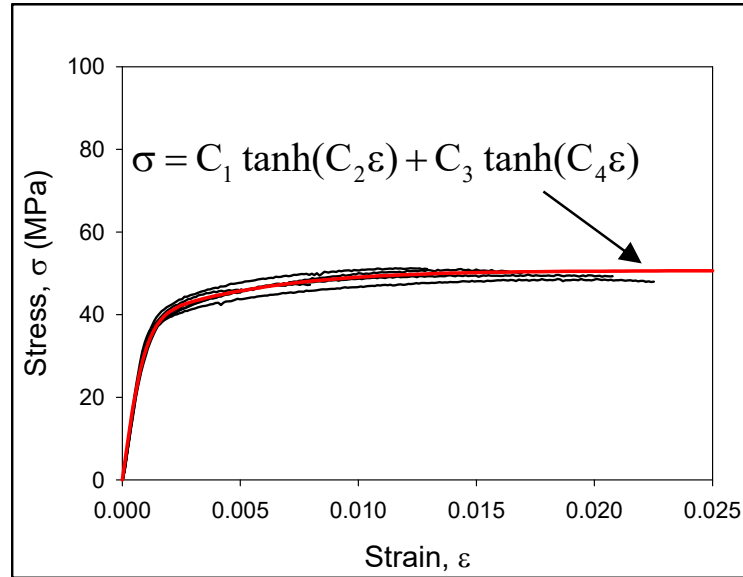


Figure 3.9 Empirical Model Fit to Solder Stress-Strain Curves

3.5 Typical Creep Data Processing

As shown in Figure 3.10, the typical solder creep curve can be divided into three stages, and the secondary (steady-state) region is often characterized by a long duration that is nearly linear. The constant slope is called secondary creep rate and is a key parameter used in finite element simulations for the estimation of solder reliability. For solder creep experiments, the four-parameter Burger's (spring-dashpot) model was found to fit the primary and secondary creep regions very well:

$$\sigma = C_1 \tanh(C_2 \epsilon) + C_3 \tanh(C_4 \epsilon) \quad (3.4)$$

From the strain versus time curve under constant stress, the “steady state” secondary creep rate (constant k_1) can be obtained.

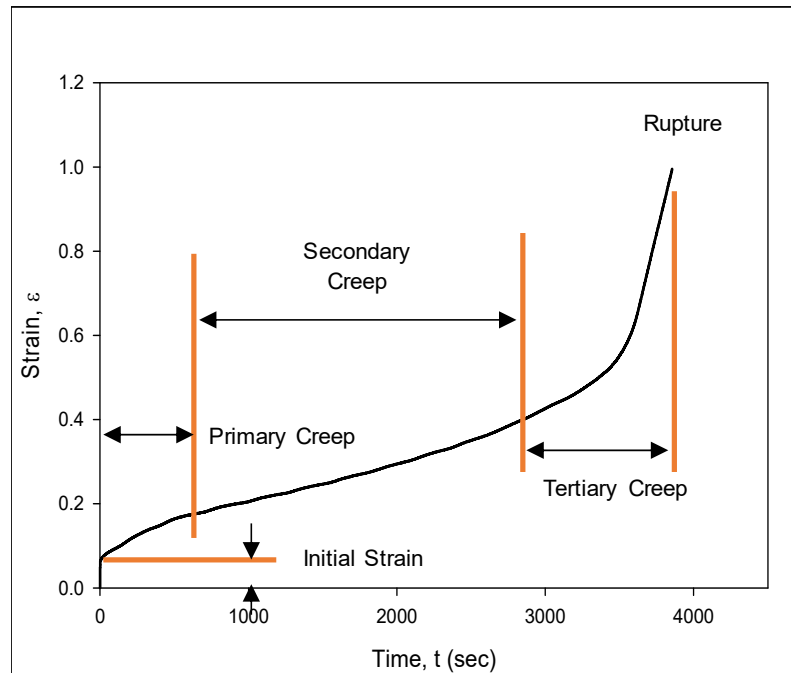


Figure 3.10 Typical Creep Curve for Solder

3.6 Sample Preparation for Cyclic Stress-Strain Test

For cyclic stress-strain testing, unlike prior studies that used rectangular cross-section glass tubes, glass tubes with circular cross-sections were adopted in this part to produce cylindrical specimens that are more resistant to buckling during the compression portions of cyclic stress-strain testing. Buckling is the bending of a slender element when subjected to compression, which leads to a sideways deflection. If buckling occurs during fatigue testing, it can be told from the test sample, corresponding hysteresis loop and cycles to failure. The resistance to buckling can be significantly improved by using the cylindrical specimens in proper size. Cylindrical glass tubes of inner diameter 1.2 mm were used in this case. For cyclic stress-strain tests, specimens were formed in circular cross-section glass tubes using the same vacuum suction process as shown in Figure 3.1. The reflow profile was chosen to closely mimic profiles used for BGA assemblies, so that the obtained microstructures were similar to those found in typical solder joints. Typical cylindrical glass tube assembly filled with solder and a final extracted specimen are shown in Figure 3.11. The samples were initially cooled in a water bath and were later reflowed.

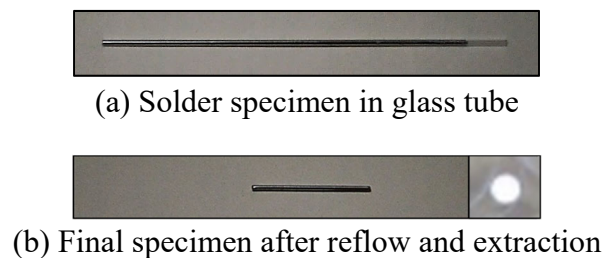


Figure 3.11 Cylindrical solder test specimen

3.7 Cyclic Stress-Strain Testing

The universal micro-tension/torsion thermo-mechanical (Wisdom Technology MT-200) testing machine shown in Figure 3.7 was used to perform the cyclic testing in

this work. Figure 3.12 shows the utilized specimen grips that contained V-shaped grooves that clamped the ends of the samples. A gage length of 5 mm was used for the tests, and the displacements could be controlled to a resolution of 0.1 micron. The samples were mechanically cycled at room temperature ($T = 25\text{ }^{\circ}\text{C}$) under strain control. For SAC305, a total strain range of $\varepsilon = \pm 0.01$ was used per cycle, while for SAC_Q, a total strain range of $\varepsilon = \pm 0.005$ was used per cycle. In both cases, a strain rate of $\dot{\varepsilon} = 0.002\text{ sec}^{-1}$ was utilized.

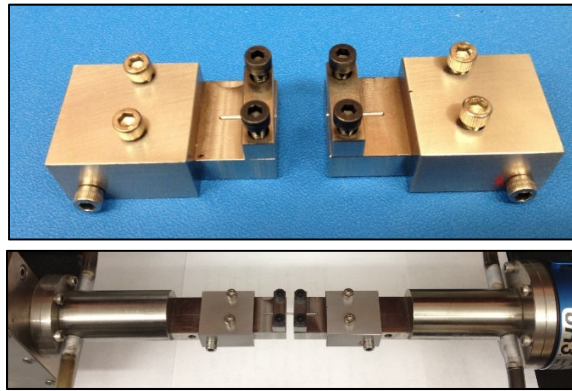


Figure 3.12 Fixtures with V-grooves used to grip the samples.

3.8 Cyclic Stress-Strain Data Processing

Typical measured cyclic shear stress–strain curves and hysteresis loops for a doped SAC solder specimen are shown in Figure 3.13. In this case, all samples were tested under strain controlled cycling with strain limits of ± 0.005 . Initially, a tensile loading was applied starting from a relaxed point (point A). Then, the loading was gradually increasing at a strain rate of 10^{-3} to generate the initial portion of the stress-strain curve until the strain reached $+0.005$ (point B). At that time, the direction of crosshead motion was reversed and the sample was then compressed until the strain reached -0.005 (point C). Again, the direction of loading was reversed and testing continued in tension until the strain again reached $+0.005$ (point B). Hence, the first hysteresis loop was ended. Further loops

(between points B and C) were created by continuing the alternating tensile and compressive loadings in a similar fashion.

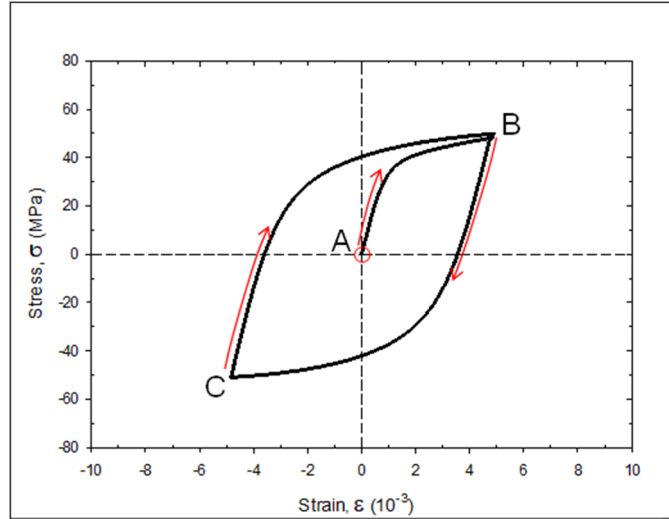


Figure 3.13 Typical Cyclic Stress-Strain Results for bulk samples (Strain Controlled)

A 4-parameter hyperbolic tangent model was used to process the cyclic stress-strain data. Two different equations of this hyperbolic tangent model were used to represent the tension portion (C to B, top) and the compression portion (B to C, bottom) of the stress-strain behavior in each cycle.

$$f_1(\varepsilon) = -A_1 \tanh(A_2(-\varepsilon + \varepsilon_2)) - A_3 \tanh(A_4(-\varepsilon + \varepsilon_2)) + \sigma_1 \quad (3.5)$$

$$f_2(\varepsilon) = B_1 \tanh(B_2(\varepsilon - \varepsilon_1)) + B_3 \tanh(B_4(\varepsilon - \varepsilon_1)) + \sigma_2 \quad (3.6)$$

$$\varepsilon_1 \leq \varepsilon \leq \varepsilon_2$$

Where the empirical fits $f_1(\varepsilon)$ for the compression loading region (bottom of the hysteresis loop) and $f_2(\varepsilon)$ for the tensile region (top of the hysteresis loop). From the stress-strain data for each cycle, the constants in the empirical models of eq. (3.5) and (3.6) can

be determined through a nonlinear regression analysis. The associated hysteresis loop area can then be calculated using the eq. (3.7).

$$\Delta W = \int_{\varepsilon_1}^{\varepsilon_2} [f_2(\varepsilon) - f_1(\varepsilon)] d\varepsilon \quad (3.7)$$

This area represents the energy density dissipated per cycle during the cyclic loading.

From the recorded cyclic stress-strain curves, the areas of the hysteresis loops are calculated using the procedure outlined above. The plastic strain ranges and peak stresses are determined as illustrated in Figure 3.14. The plastic strain range is equal to the total strain range minus the elastic strain range, which is the distance between the two strain axis intercepts on the graph. The peak stress is simply the maximum stress recorded during the cycle. The hysteresis loop area and plastic strain range are usually considered as fatigue damage driving forces, and are widely used in models to estimate fatigue life of solders.

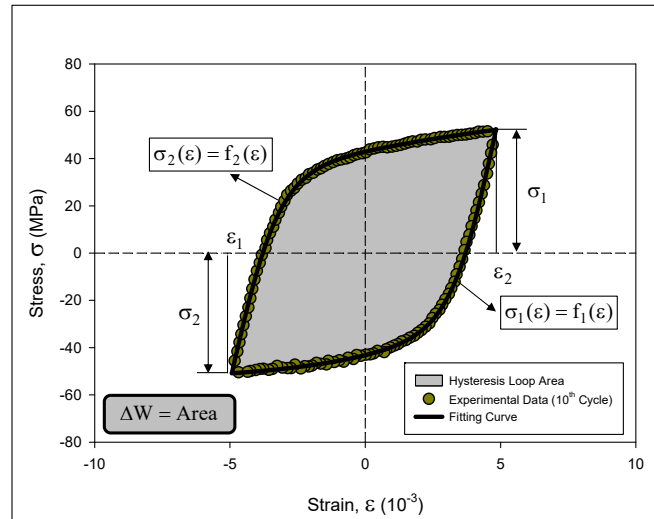


Figure 3.14 Cyclic stress-strain curve and hysteresis loop for a SAC solder.

To study the effects of cycling on damage accumulation and changes in the constitutive behavior of SAC305, SAC_Q and Innolot, uniaxial samples were prepared and subjected to various durations of prior isothermal mechanical cycling (e.g. 0, 50, 100, 300,

600, 900, 1200 cycles) that were below the fatigue life of the material. These various amounts of prior cycling served to impart various levels of fatigue damage to the samples. The same strain ranges of $\varepsilon = \pm 0.01$ and $\varepsilon = \pm 0.005$ were used for SAC305 and SAC_Q & Innolot, respectively. After cycling (damage accumulation), the specimens were subjected to stress-strain or creep testing.

In all the cyclic studies conducted in this dissertation, the hysteresis curves that were plotted were extracted from a single sample for representative purposes. All the cyclic data, however, were reported from cyclic studies conducted on at least 5 samples.

3.9 Microstructural Study

Rectangular cross-sectioned samples were employed to use study the degradations in microstructure of lead free alloys when subjected to mechanical cycling at elevated temperatures. The same vacuum system method followed by the reflow procedure was utilized to produce the samples. The specimens had a nominal dimension of 30 x 3 x 0.5 mm. The samples were then attached to a dummy epoxy preform with the aid of a double sided tape. This was done to enable extraction the sample and conduct mechanical cyclic tests on it which not have been possible had the samples been potted in epoxy resin. Details of the preparation process included mechanical grinding with several SiC papers (#320, #400, #600, #800 and #1200) in a rotating metallographic disc as shown in Figure 3.15. The final polishing was conducted with 0.02 μm colloidal silica suspensions where BUEHLER MasterMet 2 was the abrasive solution and BUEHLER ChemoMet was the polishing cloth. This procedure resulted in mirror finish samples suitable for optical microscopy, Scanning Electron Microscopy (SEM), as well as nanoindentation. Microstructure analysis of the solder alloys was performed using a Zeiss Polarized Optical

Microscope (Figure 3.16), a JEOL JSM 7000F Field Emission SEM (Figure 3.17 (a)), and a Zeiss Crossbeam 550 SEM (Figure 3.17 (b)). In addition, EDS (Energy-Dispersive X-ray Spectroscopy) was employed to explore the chemical composition of different phases in the microstructure.



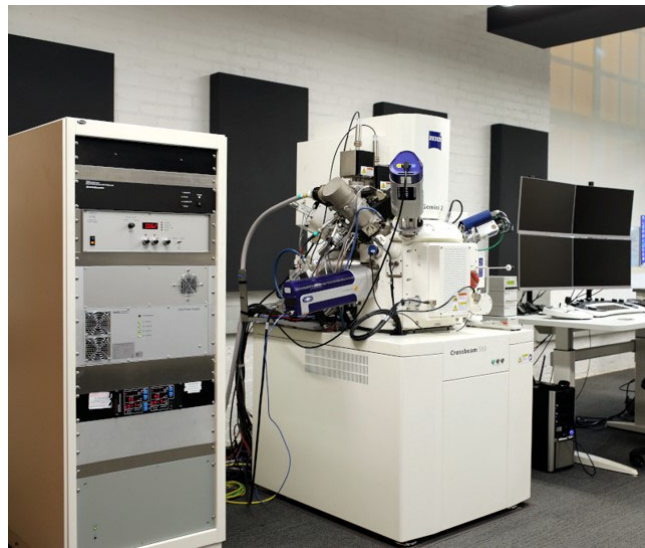
Figure 3.15 Grinding and Polishing Machine



Figure 3.16 Zeiss Polarized Optical Microscope



(a) JEOL JSM-7000F Field Emission SEM



(b) Zeiss Crossbeam 550 SEM

Figure 3.17 SEM Machines used

The prepared samples were then placed within the nanoindentation system (Figure 3.18), and several fixed square regions with dimensions of approximately $90 \times 90 \mu\text{m}$ were then selected in the polished region of each sample for subsequent SEM observations. To locate and track the selected fixed regions, they were demarcated using four nanoindentation marks placed at their corners using a Hysitron TI 950 nanoindentation system as shown in Figure 3.19. After polishing and applying the nanoindentation marks,

the specimens were carefully removed from the temporary carriers. The indentation marks were positioned close enough to enable region identification, but far enough away from the region to avoid influencing the microstructure evolution. Lastly, a field emission SEM was used to study the changes of solder microstructure caused by both fatigue cycling and aging.



Figure 3.18 Hysitron TI950 TriboIndenter

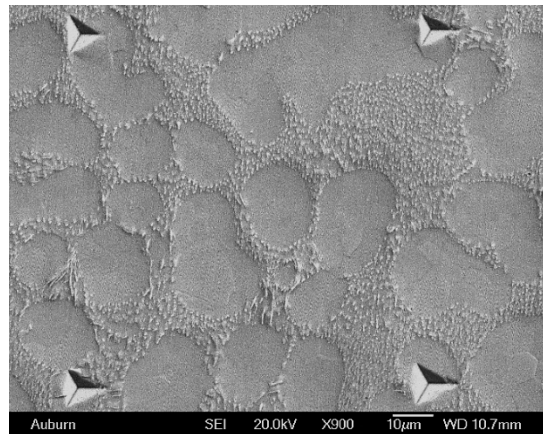


Figure 3.19 - Typical Fixed Region with Indentation Marks

3.10 Sample Preparation for Nanoindentation

For this study, SAC305 (96.5Sn-3.0Ag-0.5Cu) and SAC_Q (92.7Sn-3.4Ag-0.5Cu-3.3Bi) lead free solder joints were tested. The joints had a diameter of about 30mils (762 μm). Figure 3.20 shows an example of the test joints used for the experiments. The samples consisted of a 3 x 3 Ball Grid Array (BGA) of the lead free alloys to be tested. The substrates used in the specimens were FR-4 PCBs. Samples which had an Immersion Silver (ImAg) surface finish on the copper pads were chosen. Flux was deposited on the pads using a stencil. A reflow profile with a maximum temperature of 245 °C was obtained with the help of a Vitonics-Soltec convection oven. The profile also ensured that the assembly was kept above the liquidus temperature for about 45-60 seconds. The samples were then cleaned, dried and stored in a freezer at -40 °C to hinder any aging effects.

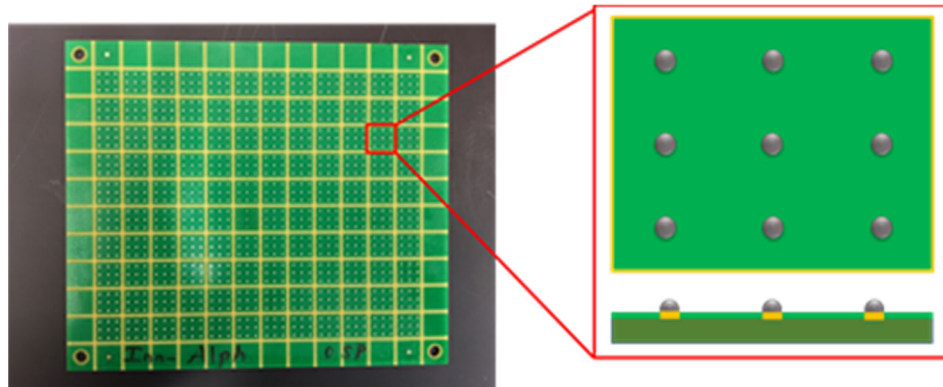


Figure 3.20 3x3 BGA Specimen Sample

Samples were covered with a tape and then confined in epoxy resin. The tape was used to prevent epoxy from entering the space between the individual joints which allows the cycling of each joint separately. The tape from these samples were removed by coarse grinding using a 320 grit SiC paper. Grinding papers up to 1200 grit were then used to reduce the scratches on the samples. The samples were then finally polished a 0.02 μm

colloidal silica suspension to flatten the top surface of the solder joints and making the joints suitable for nanoindentation tests. All the polished joints were viewed under a Polarized Light Microscope to verify that all experiments were conducted on single grain joints. The Micromechanical Tester shown in Figure 3.21 was used to cycle the individual solder joint. The cyclic shear loading was conducted with a peak load of 600 gf (equivalent to a stress of about 24MPa). The joints were individually cycled in the shear direction for various durations of 50, 100, 150, 200 and 300 cycles. After every selected cyclic condition, the sample was removed and the evolution in Elastic Modulus, Hardness and Creep Strain Rate of each joint was characterized using the nanoindentation machine.

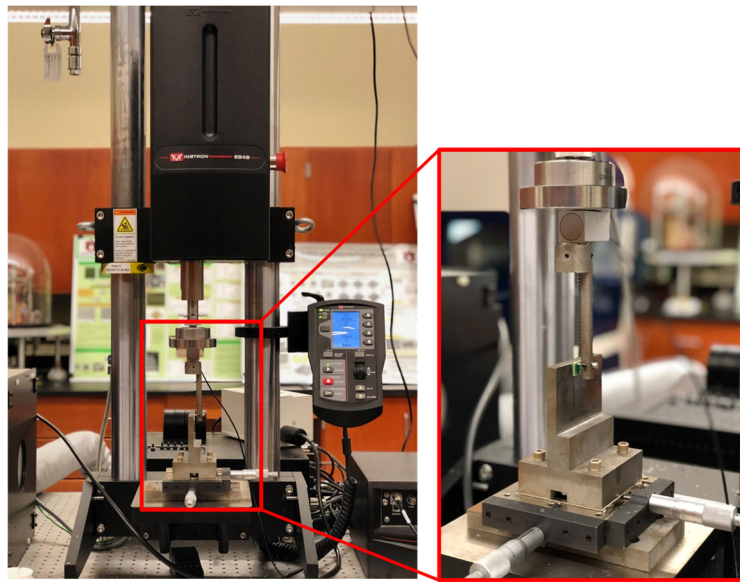


Figure 3.21 Micromechanical Tester used for the Mechanical Cycling Tests

3.11 Nanoindentation Machine and Test Procedures

The nanoindentation machine utilized a probe with a Berkovich geometric tip to record the changes in load against indentation displacement. The recorded data was measured in the direction normal to the polished face. At least 5 indents were made on every solder joint for each set of conditions. Through trial and error, it was observed that

a peak holding load of 30 mN was ideal for the nanoindentation tests. Using this high load not only prevented creep effects but also ensured that the indents made were deep enough to prevent surface effects while also ensuring these large indents covered all the solder phases present. A schematic depicting all the experimental techniques described above and used for this study has been presented in Figure 3.22.

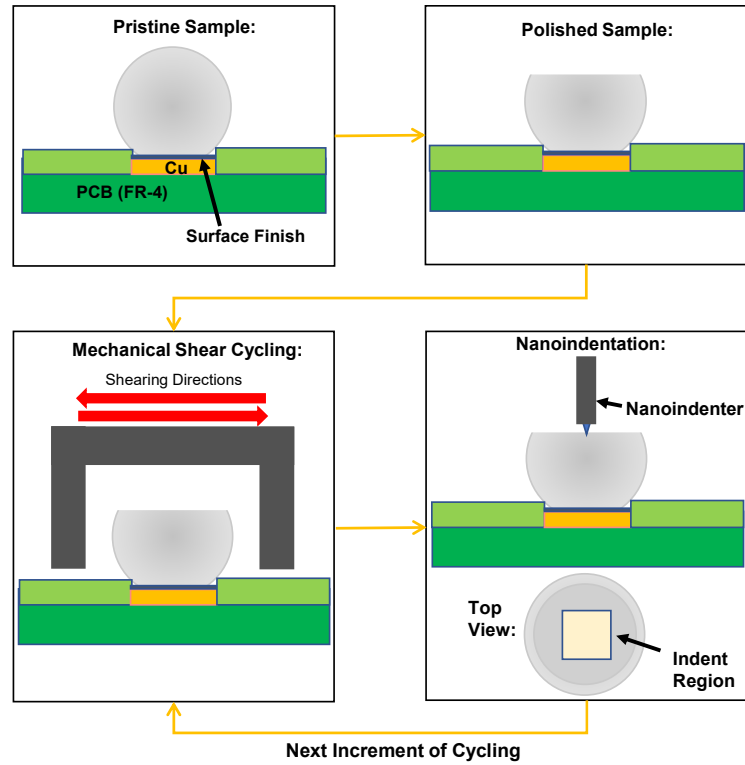


Figure 3.22 Schematic showing the experimental procedure

3.12 Measurement of Elastic Modulus and Hardness

A typical load (P) versus displacement (h) curve, obtained after a nanoindentation test, is presented in Figure 3.23. This curve has three different segments where the first, second, and third segments represent the displacements during the loading, holding and unloading period, respectively.

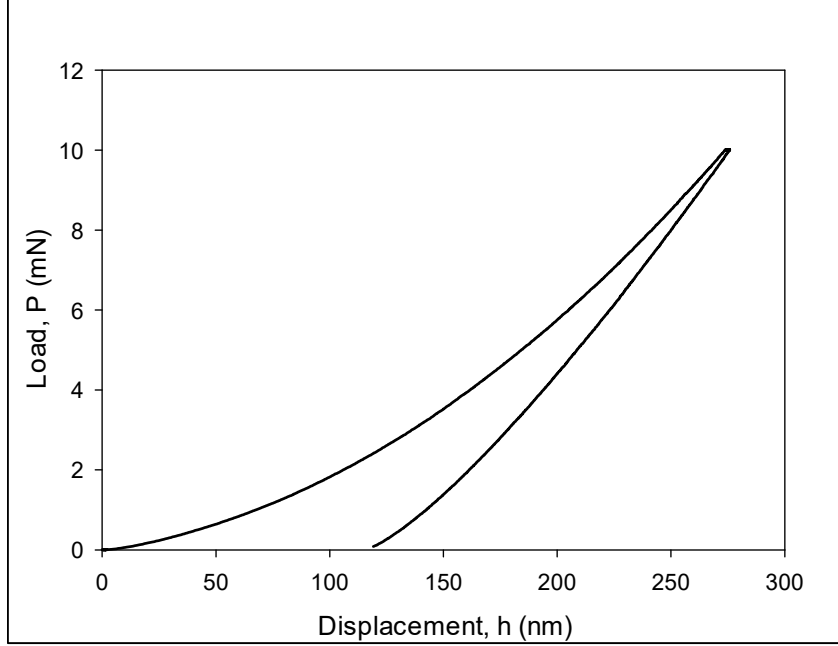


Figure 3.23 An example of the load-displacement curve obtained after nanoindentation testing.

During a nanoindentation experiment, the effects of the non-rigidity of an indenter, during a nanoindentation experiment, can be addressed by introducing a term called reduced modulus (E_r) through the following equation:

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i} \quad (3.8)$$

Where, E is the elastic modulus of the test specimen, E_i is the known elastic modulus of the indenter tip material, ν and ν_i are the Poisson's ratio of the test specimen and the indenter tip, respectively. Typically reduced modulus was measured from an indentation experiment using the following equation:

$$E_r = \frac{\sqrt{\pi}}{2} \times \frac{S}{\sqrt{A}} \quad (3.9)$$

Where, S is the stiffness of the test specimen at the maximum load and A is the projected contact area at the maximum load. Stiffness was determined from the initial slope ($\frac{dP}{dh}$) of

the unloading segment of a load displacement curve. The contact area A was measured following the technique proposed by Oliver and Pharr [129] where they assumed the contact area to be a function of contact depth. For an ideal Berkovich tip, it can be expressed as

$$A(h_c) = 24.5h_c^2 \quad (3.10)$$

In order to address any deviation from the ideal Berkovich geometry due to tip blunting, a modified version of equation 3.6 was utilized.

$$A(h_c) = 24.5h_c^2 + C_1h_c^1 + C_2h_c^{1/2} + C_3h_c^{1/4} + C_4h_c^{1/8} + C_5h_c^{1/16} \quad (3.11)$$

Where, C_1 , C_2 , C_3 , C_4 , and C_5 are fitting constants. In order to determine the values of these constants, multiple indents were made at multiple depths on a quartz sample with known elastic modulus (69.6 GPa). The contact areas at the different known depths were determined from equation 3.5 and these values were plotted to get an A versus h_c plot. The values of constants C_1 to C_5 were determined by fitting the plot by Equation 3.11. Once the values of the constants are known for a particular tip geometry, equation 3.9 was used to determine the value of reduced modulus (E_r) for any unknown material and equation 3.8 was used to convert E_r to elastic modulus E .

Hardness is a material property that defines the resistance of the surface against plastic deformation. During a nanoindentation experiment, hardness H was determined by dividing the maximum load by the projected contact area.

$$H = \frac{P_{\max}}{A} \quad (3.12)$$

Researchers [130, 131] have developed an approximate relationship between hardness and yield strength of which is true for many metals.

$$H \approx 3\sigma_Y \quad \text{Or} \quad \sigma_Y \approx \frac{H}{3} \quad (3.13)$$

The above equation was used to determine stress during nanoindentation experiments.

3.13 Polishing of Sandwiched Solder Joints

Each sample that was for the study on the effect of shear cycling on the grain morphology consisted of 9 reflowed SAC305 sandwiched joints arranged in a 3x3 Ball Grid Array (BGA). The substrate in the samples were FR-4 PCBs. The joints in consideration had an average diameter of about 30 mils (approximately 762 μm) and reflowed on to copper pads with a nominal diameter of 20 mils (approximately 508 μm). The ball-to-ball pitch was about 3 mm. The average height of the joints was approximately 0.5 mm while the specimen had an overall height of about 2.5 mm. Fig. 3.24 represents one such sample specimen used in these experiments. An Immersion Silver surface finish was applied on the specimens.

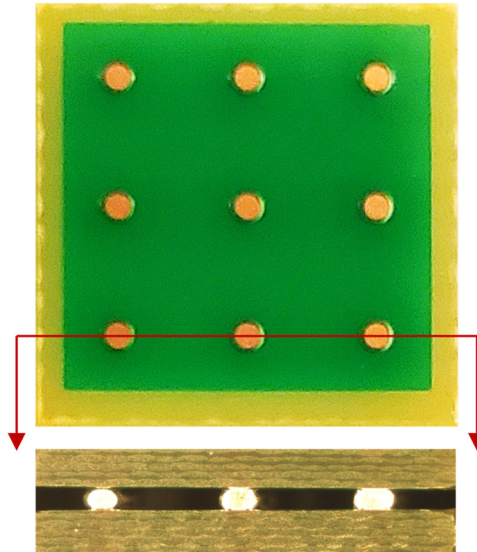


Figure 3.24 SAC305 Sandwiched BGA Samples

To observe the change in the microstructure of the SAC305 joints with mechanical cycling, the samples were first inserted into slots created in epoxy preforms. One such sample fit inside an epoxy preform is shown in Fig. 3.25. The slot created in the epoxy preforms had a similar thickness to that of the specimens to ensure that a tight fit is created for even polishing. This method would allow us to not only evenly support the sample for polishing, but also be able to extract the sample after polishing. The specimens were polished on either sides to verify that the load distribution on the sample would be equal on both sides.

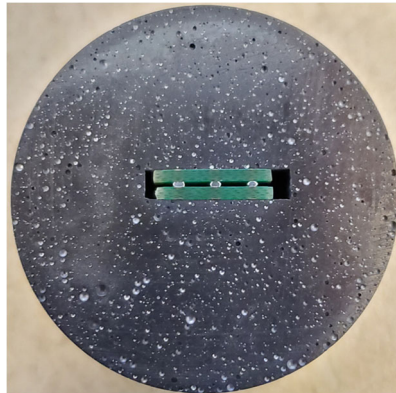


Figure 3.25 Polished SAC305 Joints in Slotted Epoxy Preforms

The polished sample was then extracted from the epoxy resin and cycled using the microtester shown in Fig. 3.7. The thermal chamber visible in Fig. 3.7 was used to maintain an isothermal environment to conduct the shear mechanical cycling. Fig. 3.26 shows a set of newly designed fixtures used to grip and cycles these BGA samples. The sample is tightened using screws at various locations to ensure the cycling is uniform. The left end of the microtester is attached to the linear actuator that allows the arm to move back and forth while the right end of the microtester is held stationary. The load cell is attached to the stationary arm of the microtester. The samples were cycled in a shear direction until

failure was noticed. The polarized optical microscope presented in Fig. 3.16 was used to observe changes in the grain morphology of the solder joints when subjected to isothermal mechanical cycling. The microstructures were recorded after selected cyclic intervals. Any aged samples that were used in this study were aged in an environmental chamber for 10 days at a temperature of 125 °C.

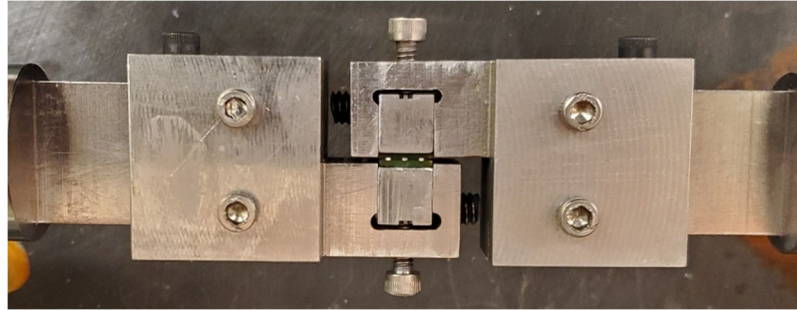


Figure 3.26 Newly Designed Fixtures to Grip Sandwiched Samples

3.14 Summary and Discussion

All the experimental procedures and the data processing steps were presented in this chapter. Micro-scale uniaxial tensile specimens were prepared in a circular shaped hollow glass tubes using a vacuum suction method. These uniaxial lead free solder test specimens were introduced to solve the buckling problem during compression in the cyclic or fatigue testing. Gripping fixtures were designed and added to the test system to grip the cylindrical specimens. After testing, empirical models were utilized to represent the stress strain, creep, and cyclic stress-strain experimental data.

To study the effects of mechanical cycling on the solder microstructure, during cyclic testing, SAC305, SAC Q, and Innolot uniaxial samples with various durations of prior mechanical cycling were prepared. After polishing, small indentation marks were

added to the cross-sections to facilitate locating the identical regions of interest. Then the microstructural changes in the fixed regions during cycling were examined using SEM.

In a latter part of this study, a unique specimen preparation procedure was to fabricate actual 3 x 3 BGA solder joint array specimens to explore the effect of shear mechanical cycling on damage accumulation in SAC305 and SAC_Q solder joints. The solder balls were cycled individually with a peak load of 600 gf by a Micromechanical tester for various durations (50, 100, 150, 200, 300 cycles), all of which were below the fatigue life of these joints. After each cycling duration, nanoindentation experiments were conducted on the samples to test for changes in elastic modulus, hardness, and creep response.

Sandwiched 3 x 3 SAC305 BGA solder joints were initially inserted in an epoxy preform and polished. The samples were polished on both sides to ensure an even load distribution. After the polishing process was completed, the samples were extracted and cycled in a shear direction using newly designed fixtures compatible with the micro-tension torsion machine used for the bulk solder experiments. Using an optical microscope, the changes in microstructure with shear mechanical cycling was observed using a polarized optical microscope. Aging and elevated temperature factors were also introduced to notice how they played an impact in the degradation of the microstructure. The microstructures were also checked for any signs of recrystallization along the crack path.

CHAPTER 4

EVOLUTION OF MECHANICAL PROPERTIES IN LEAD FREE AND DOPED LEAD FREE SOLDERS DURING CYCLIC LOADING

4.1 Introduction

While it has been known that the reversal of inelastic strain can change the stress-strain behavior of materials (Bauschinger effect), there have been very few prior studies on how the cycling changes the microstructure and reduces the mechanical properties of lead free solders during fatigue testing. In this chapter, we have explored the effects of mechanical cycling on the constitutive behavior (stress-strain and creep) of SAC305 and SAC_Q (SAC+Bi) in lead free solders during fatigue loading. The evolution of the cyclic stress-strain behavior (hysteresis loop area, plastic strain range, and peak stress) during the cycling tests has also been studied. Cylindrical uniaxial SAC305 and SAC_Q lead free solder test specimens have been prepared. The uniaxial specimens had a nominal diameter of 1.2 mm, and the gage length was 5 mm in this work. All the tests were conducted at room temperature (25 °C) at a strain rate of $\dot{\epsilon} = 0.002 \text{ sec}^{-1}$. Strain controlled method has been used in the cyclic testing, and the cyclic strain limits were ± 0.01 . To study the evolution of the cyclic stress-strain behavior, the specimens have been subjected to cyclic stress/strain loading at room temperature (25 °C) until fatigue failure. The cyclic stress-strain curves at after various durations of cycling have been examined. From the recorded cyclic stress-strain curves, the evolution of the solder hysteresis loops with number of cycles has been characterized. Also, the duration of cycling on the hysteresis loop area (plastic strain energy density), plastic strain range, and peak load have been studied.

To study the effects of cycling on damage accumulation and changes in the constitutive behavior of SAC305 and SAC_Q, uniaxial samples have been prepared and subjected to various durations of prior isothermal mechanical cycling (e.g. 0, 50, 100, 300, 600, 900, 1200 cycles) that were below the fatigue life of the material. These various amounts of prior cycling served to impart various levels of fatigue damage to the samples. The same strain ranges of $\varepsilon = \pm 0.01$ and $\varepsilon = \pm 0.005$ were used for SAC305 and SAC_Q, respectively. After cycling (damage accumulation), the specimens were subjected to stress-strain or creep testing. After fitting the experimental data from mechanical testing, we have been able to characterize the cycling induced evolution of several mechanical properties of SAC305 and SAC_Q lead free solders. The goal of the study is to explore damage accumulation that occurs during fatigue testing.

4.2 Evolution of Cyclic Stress-Strain Curves

To study the evolution of the cyclic stress-strain behavior with cycling, the specimens have been subjected to cyclic strain loading (± 0.01) at room temperature until fatigue failure occurred. Figure 6.1 shows a typical result for a SAC305 sample that was cycled with a total strain range of $\varepsilon = \pm 0.01$. The hysteresis loops for selected cycles ($N = 1, 50, 100, 200, 300 \dots, 1200$) are indicated. It is clear that the peak stress and hysteresis loop size evolve dramatically during the test. For example, the fall of the peak stress is shown in Figure 6.2. This response is typically divided up into three regions. Initially, the peak stress declines rapidly (primary region), followed by a steady state (secondary) region of constant slope. The curve then enters a tertiary stage, where specimen fracture occurs. The fatigue life is typically defined as the number of cycles needed to cause a preset drop in the peak stress (preset load drop). In this work, we selected a 50% decline to define

failure. With the selected strain range, the fatigue life of the SAC305 samples was approximately 1200 cycles.

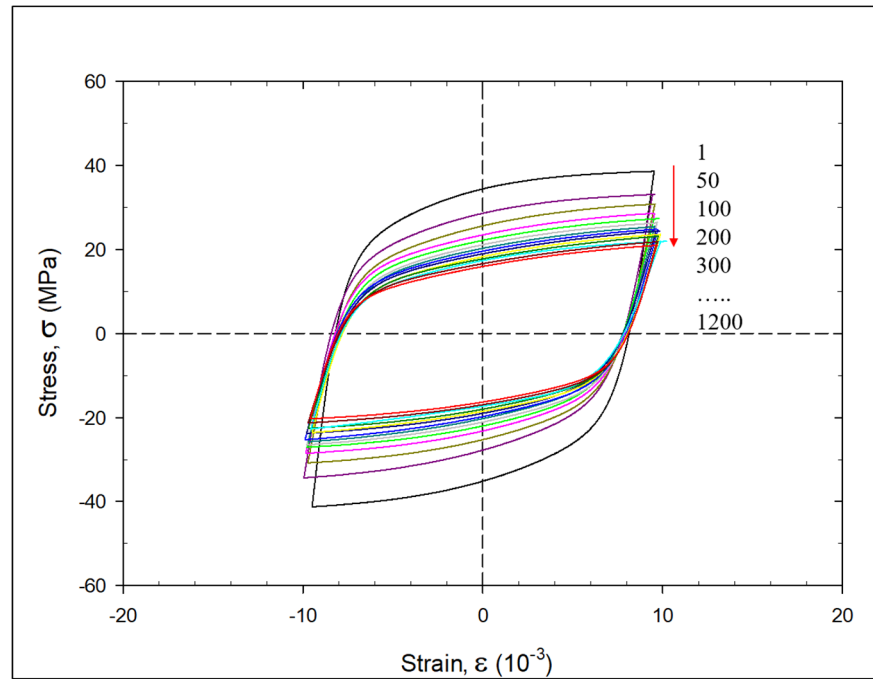


Figure 4.1 Evolution of the cyclic stress-strain curves during a cyclic test (SAC305)

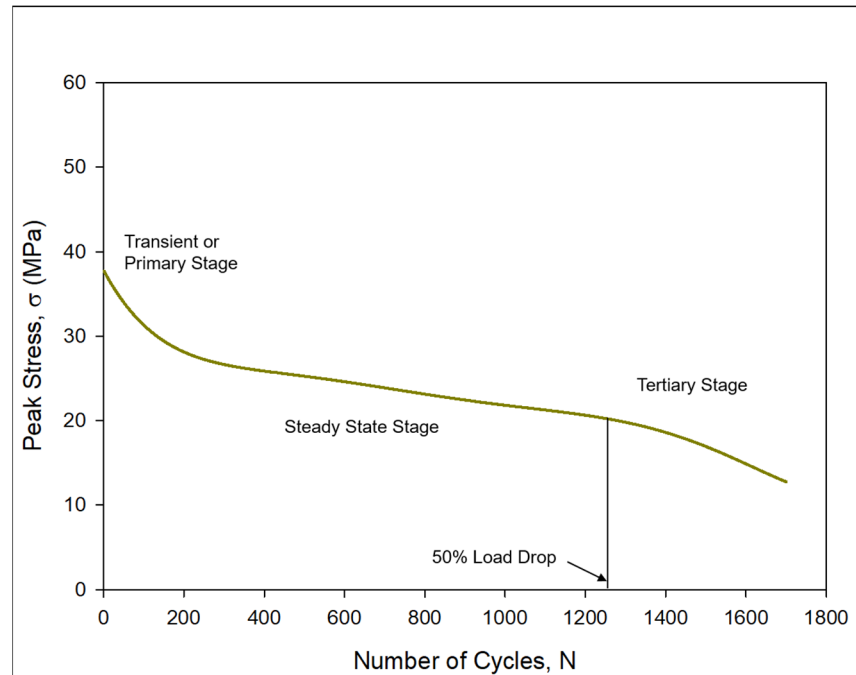


Figure 4.2 Decline of the peak stress during a fatigue test (SAC305)

For each cyclic test, the recorded hysteresis loops were processed at several cycle counts ($N = 0, 50, 100, 300, 600, 900$, and 1200 cycles) as described above, and hysteresis loop area, peak stress, and plastic strain range were calculated. The numerical results (averages for three tests) are shown in Table 4.1. It can be seen that the plastic strain (distance between horizontal axis intercepts) remains essentially constant during the cycling. Thus, the evolution in loop area is caused mainly by the reduction in peak stress as the strength of the material is degraded by damage accumulation in the specimen. For SAC305, the reductions in peak stress and hysteresis loop area were 48% and 56%, respectively, for the 1200 applied cycles.

Table 4.1 Cyclic Induced Changes of hysteresis loop area, peak stress, and plastic strain range (SAC305)

Cycling Duration (Cycles)	Loop Area (MJ/m ³)	Peak Stress (MPa)	Plastic Strain Range (%)
1	1.16	38.65	1.65
50	0.88	33.18	1.63
100	0.80	30.62	1.61
300	0.71	27.28	1.59
600	0.62	24.71	1.59
900	0.57	23.10	1.61
1200	0.51	20.18	1.61

Figure 4.3 shows analogous results for the cyclic stress strain evolution of a typical SAC_Q sample that was cycled with a total strain range of $\varepsilon = \pm 0.005$. Again, it is observed that the peak stress and hysteresis loop size evolve dramatically during the test, while the plastic strain range remains almost constant. The numerical results (averages for three tests) are shown in Table 4.2. For SAC_Q, the reductions in peak stress and hysteresis loop area were both 54%.

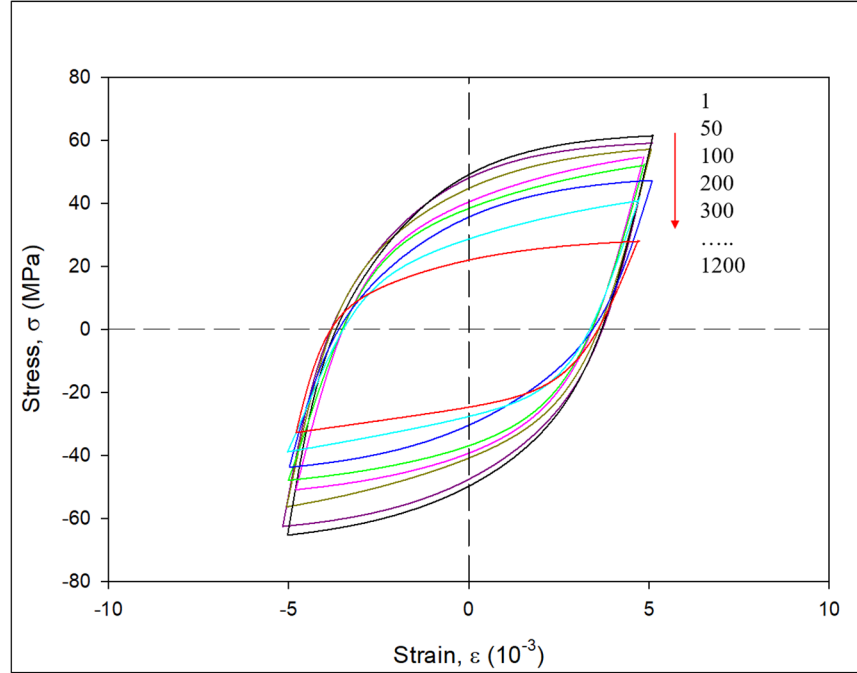


Figure 4.3 Evolution of the cyclic stress-strain curves during a cyclic test (SAC_Q)

Table 4.2 Cyclic Induced Changes of hysteresis loop area, peak stress, and plastic strain range (SAC_Q)

Cycling Duration (Cycles)	Loop Area (MJ/m ³)	Peak Stress (MPa)	Plastic Strain Range (%)
1	0.71	61.39	0.58
50	0.60	57.56	0.59
100	0.54	54.38	0.59
300	0.51	49.27	0.60
600	0.46	46.62	0.61
900	0.40	40.45	0.62
1200	0.33	28.06	0.62

Figure 4.4 demonstrates similar hysteresis loops and extracted properties that were obtained for Innolot samples cycled at a strain range of $\epsilon = \pm 0.005$. It was observed again that the plastic strain range was fairly constant. The numerical values of the extracted properties for Innolot have been tabulated in Table 4.3. After 1200 cycles, the peak stress and hysteresis loop area decreased by about 54% and 45%, respectively, for Innolot.

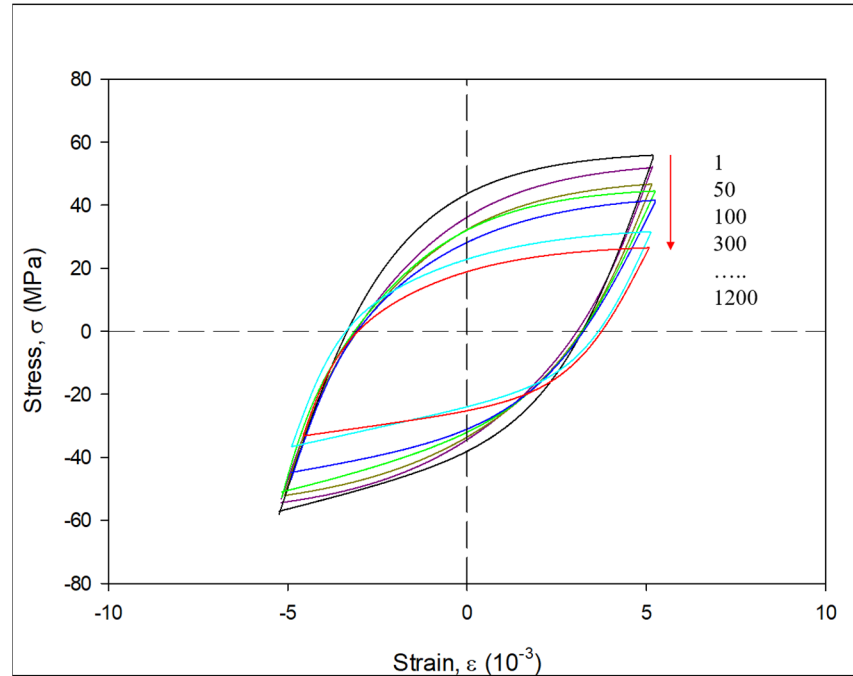


Figure 4.4 Evolution of the cyclic stress-strain curves during a cyclic test (Innolot)

Table 4.3 Cyclic Induced Changes of hysteresis loop area, peak stress, and plastic strain range (Innolot)

Cycling Duration (Cycles)	Loop Area (MJ/m ³)	Peak Stress (MPa)	Plastic Strain Range (%)
1	0.58	58.09	0.66
50	0.51	54.36	0.64
100	0.48	52.10	0.64
300	0.47	50.98	0.64
600	0.45	44.78	0.65
900	0.35	36.63	0.69
1200	0.32	26.54	0.68

The numerical values for SAC305, SAC_Q and Innolot cannot be directly compared since different applied strain ranges were used for the three materials resulting in different values of the initial hysteresis loop size and initial plastic strain range during the first cycle. In order to directly compare the performance of the materials, it is necessary to know the controlling parameter for fatigue damage accumulation. The choice of this

parameter is still often considered an open question. However, it is regularly taken to be the hysteresis loop area (ΔW) (Morrow fatigue model), or the plastic strain range (Coffin-Manson fatigue model). In the future, by modifying the additional applied total strain ranges for all three solder materials, an attempt to match the amount of damage applied during the first cycle (as modeled using either ΔW or the plastic strain range) will be investigated. With such an approach, one will be in a better position to compare the observed reductions and damage accumulations for the three materials.

4.3 Evolution of Stress-Strain Behavior with Mechanical Cycling

The next step of the study was to investigate the effects of the damage accumulation caused by mechanical cycling on the evolution of the tensile stress-strain and creep behaviors of the lead free alloys. Uniaxial samples were prepared as described above, and then subjected to various durations of prior isothermal mechanical cycling (e.g. 0, 50, 100, 300, 600, 900, and 1200 cycles) that were below the fatigue life of the materials. The cycled samples were then subsequently subjected to stress-strain or creep testing.

Figure 4.5 illustrates the evolution occurring in the tensile stress-strain curve of SAC305 solder due to various durations of prior mechanical cycling. The curves in Figure 4.5 are fits of eq. (3.2) to the data from five experimental tension tests performed for each level of cycling induced damage. It is easily observed that both the modulus (initial slope) and strength (maximum stress) were greatly reduced due to the prior cycling. The extracted values of the Elastic modulus, Ultimate Tensile Strength (UTS), and yield stress are tabulated in Table 4.4. For SAC305, the reductions in modulus, UTS, and yield stress were 62%, 39%, and 44%, respectively, for the 1200 applied cycles.

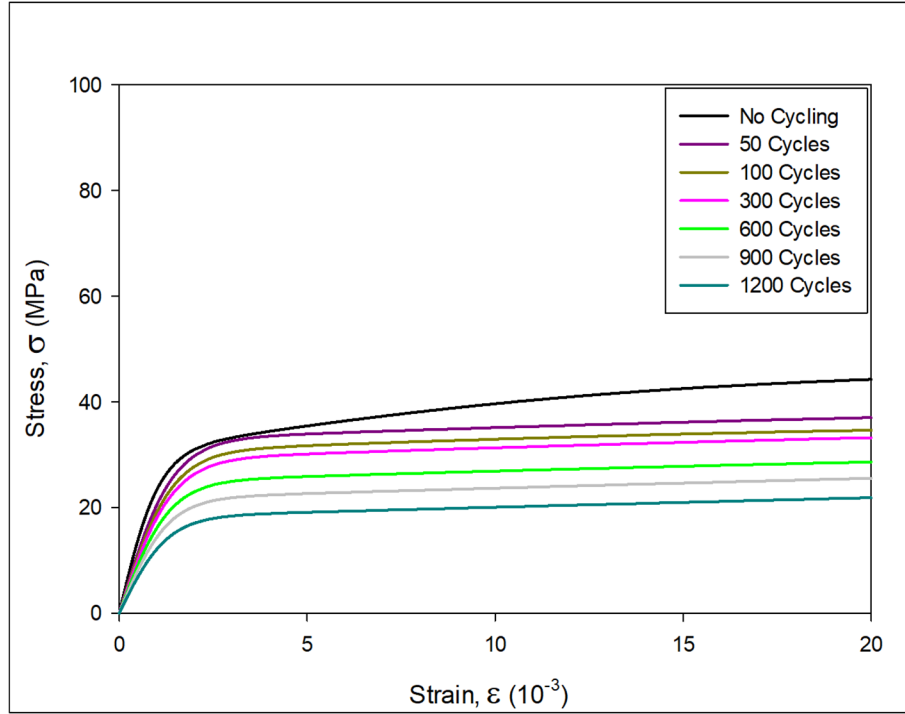


Figure 4.5 Stress-Strain Curve Evolution with Cycling (SAC305)

Table 4.4 Evolution of Material Properties with Cycling (SAC305)

No. of Cycles Prior to Testing	Elastic Modulus (GPa)	Ultimate Tensile Strength (MPa)	Yield Stress (MPa)
0	37.72	48.19	33.09
50	30.35	40.23	32.95
100	23.89	38.22	31.77
300	21.89	37.15	29.23
600	19.06	32.12	25.18
900	18.88	31.03	22.04
1200	14.48	29.46	18.56

Figure 4.6 shows analogous changes occurring in the tensile stress-strain curve of SAC_Q solder due to various durations of prior mechanical cycling. Again, it is observed that the mechanical properties were all greatly reduced due to the prior cycling. The extracted values of the Elastic modulus, Ultimate Tensile Strength (UTS), and yield stress are tabulated in Table 4.5. For SAC_Q, the reductions in modulus, UTS, and yield stress

were 28%, 37%, and 40%, respectively, for 600 applied cycles. Similarly, the elastic modulus, UTS, and yield stress reduced by 59%, 49% and 51%, respectively, for the Innolot specimens after 1200 cycles. The evolution of the tensile curves of Innolot with various prior mechanical cycles has been demonstrated in Fig. 4.7 while the extracted numerical values of the tensile curves for Innolot has been tabulated in Table 4.5.

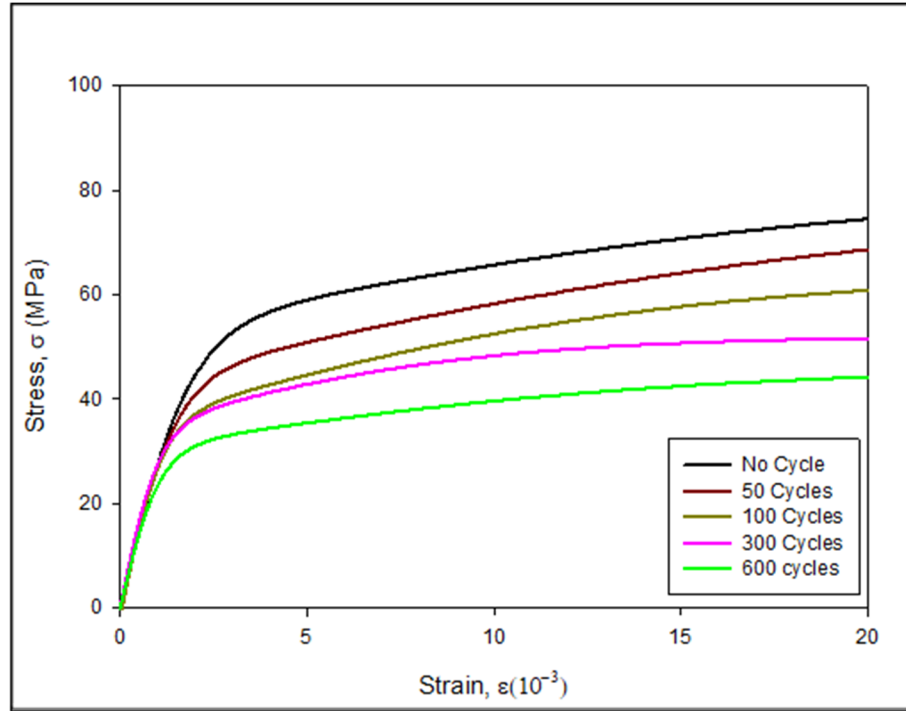


Figure 4.6 Stress-Strain Curve Evolution with Cycling (SAC_Q)

Table 4.5 Evolution of Material Properties with Cycling (SAC_Q)

No. of Cycles	Elastic Modulus (GPa)	Ultimate Tensile Strength (MPa)	Yield Stress (MPa)
0	39.26	74.31	49.63
50	33.14	67.22	41.49
100	32.67	62.16	36.28
300	31.85	55.73	34.15
600	28.44	46.54	29.67

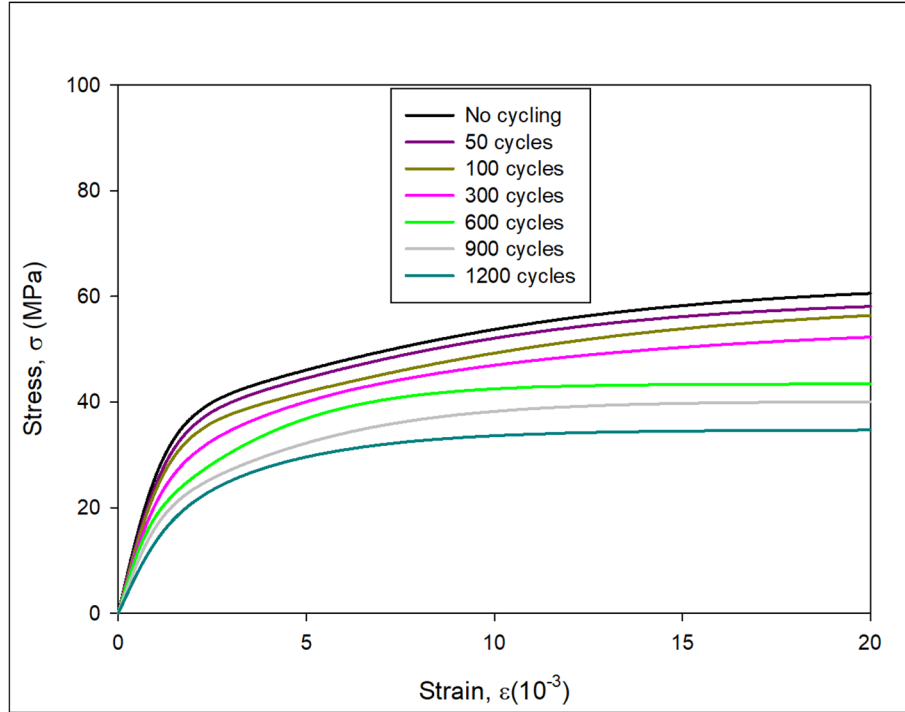


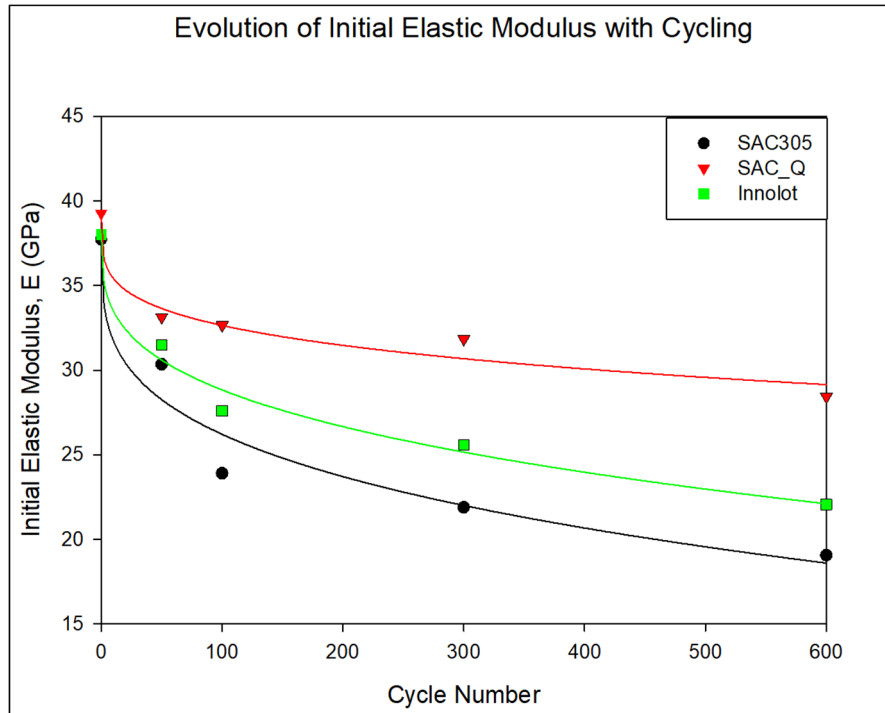
Figure 4.7 Stress-Strain Curve Evolution with Cycling (Innolot)

Table 4.6 Evolution of Material Properties with Cycling (Innolot)

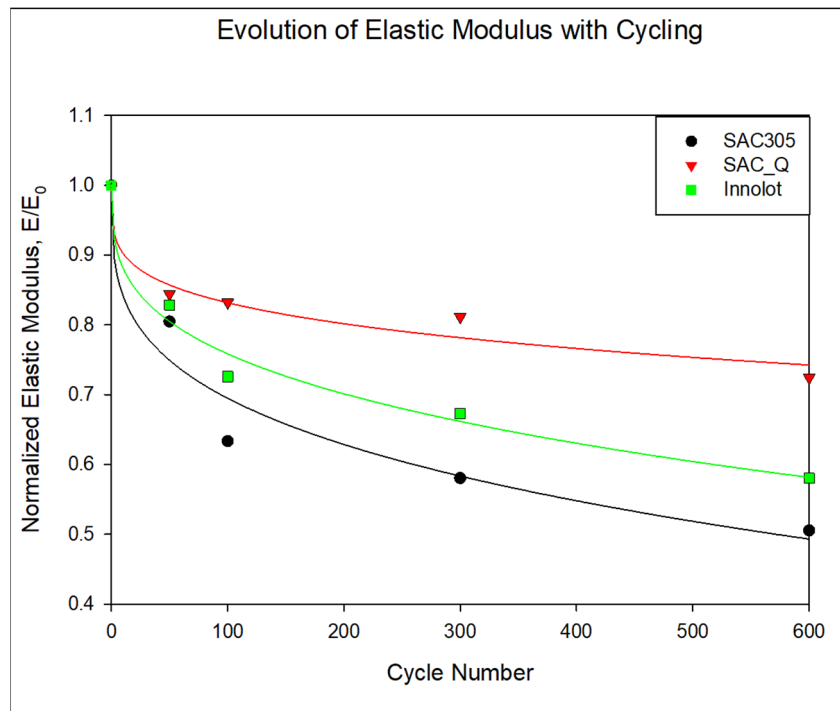
No. of Cycles Prior to Testing	Initial Elastic Modulus (GPa)	Ultimate Tensile Strength (MPa)	Yield Stress (MPa)
0	38.02	68.33	49.15
50	31.50	63.15	42.56
100	27.60	58.74	38.41
300	25.58	54.56	36.09
600	22.06	43.42	30.02
900	19.05	40.05	27.21
1200	15.68	34.66	23.95

Plots of the evolution of the initial Elastic Modulus, Ultimate Tensile Strength (UTS), and the Yield Stress of all three alloys with the progress in mechanical cycling have been presented in Figs. 4.8 – 4.10 respectively. From Fig. 8, we can infer that after the initial 50 cycles, all the alloys had a similar steep reduction in elastic modulus. Cycling

beyond this duration resulted in a much steadier and slower decrease in the Elastic modulus. SAC_Q showed the least change in elastic modulus, while SAC305 showed a significant decline in elastic modulus at the end of 600 cycles. The evolution of UTS with duration of cycling had a very different trend (Fig. 4.9), than that observed with elastic modulus. After the first 100 cycles, Innolot had experienced the least decline in UTS, while SAC305 had the highest decline in UTS. However, after the end of 600 cycles, the SAC_Q samples had the largest decline while SAC305 samples had the least drop in UTS. The magnitude of the UTS values of SAC_Q was still the highest among the three alloys despite demonstrating the largest decline in UTS after 600 cycles. By observing Fig. 4.10, we were able to deduce that all three alloys showed a large decline in yield stress magnitudes after the first 100 cycles, beyond which a steady decline in yield stress was observed for all the alloys. In this case, SAC305 displayed the least decline in yield stress, while SAC_Q had the largest decline in yield stress. Innolot, however, demonstrated the highest yield stress magnitude throughout the tests as can be observed from Fig. 4.10.

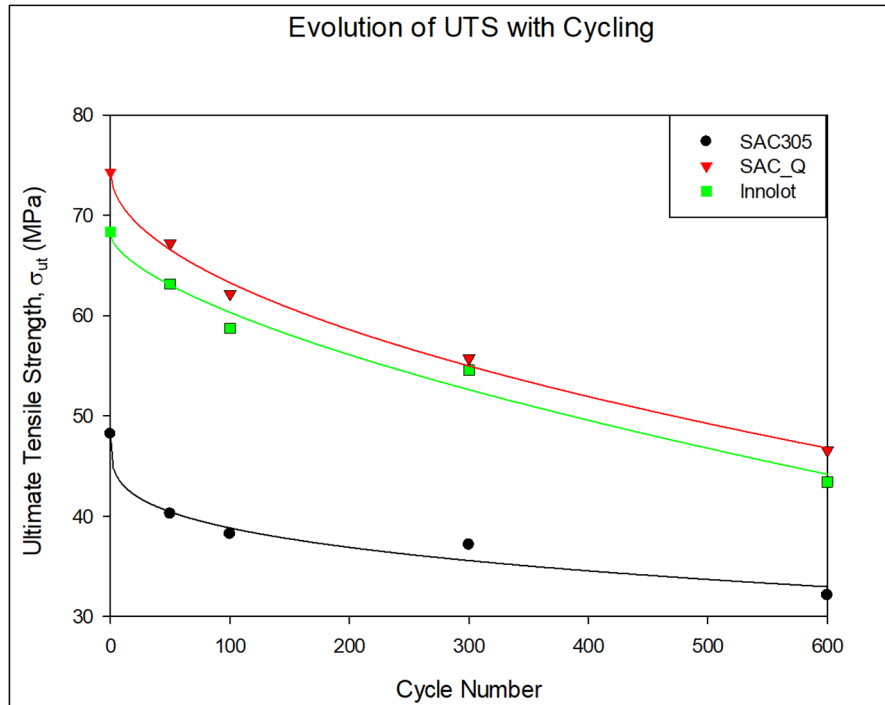


(a) Initial Elastic Modulus

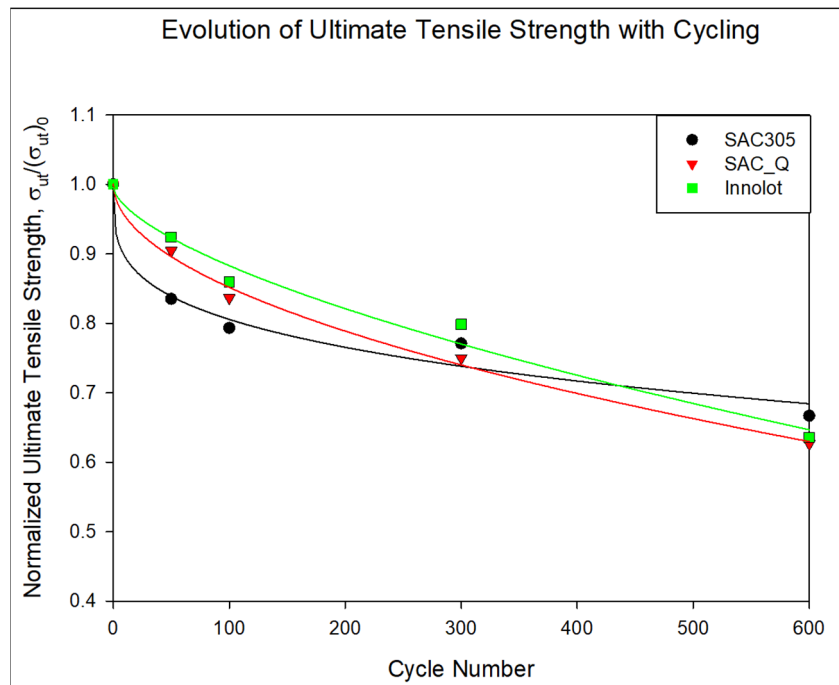


(b) Normalized Initial Elastic Modulus

Figure 4.8 Evolution of Initial Elastic Modulus with Cycling

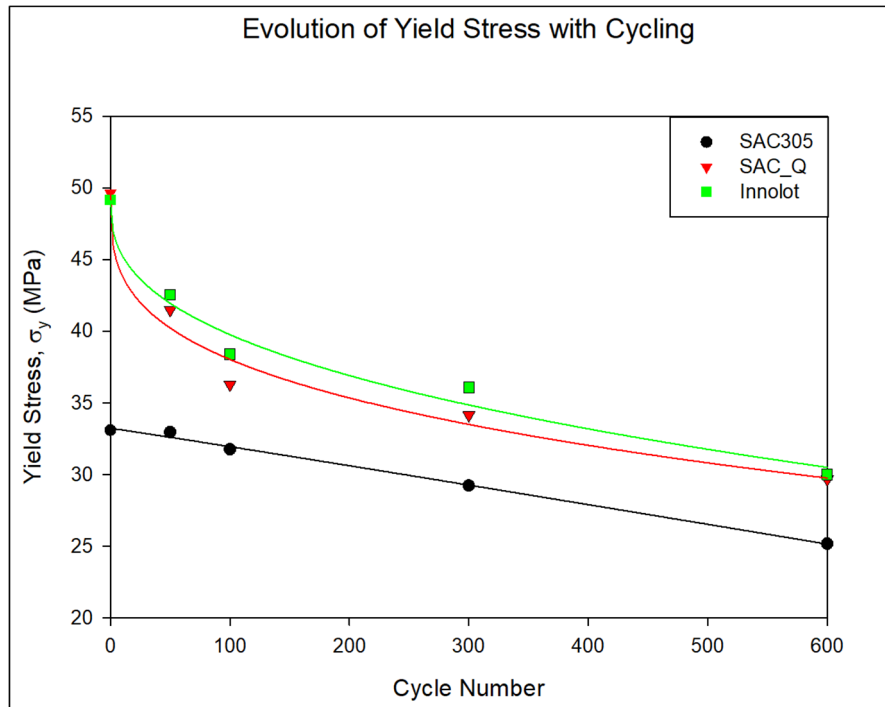


(a) Ultimate Tensile Strength (UTS)

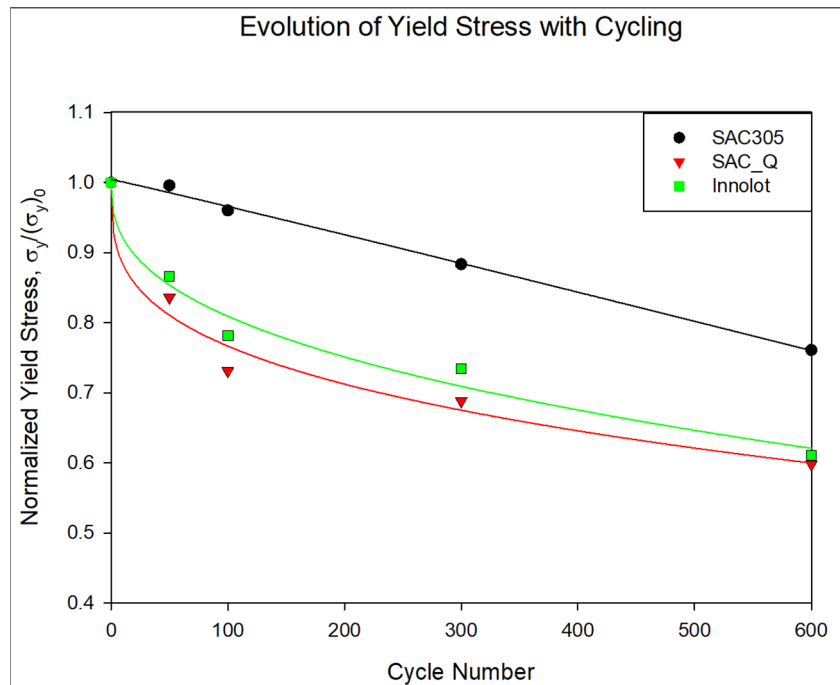


(b) Normalized Ultimate Tensile Strength (UTS)

Figure 4.9 Evolution of Ultimate Tensile Strength (UTS) with Cycling



(a) Yield Stress



(b) Normalized Yield Stress

Figure 4.10 Evolution of Yield Stress with Cycling

4.4 Evolution of Creep Behavior with Mechanical Cycling

Figs. 4.11 – 4.13 illustrate the creep response evolution with prior mechanical cycling of SAC305, SAC_Q, and Innolot samples, respectively. It is evident from the figures that mechanically induced damage accumulation has a more significant effect on the creep resistance than the tensile properties. The effect on creep behavior is seen to be similar to the effects brought about by aging. All the alloys showed a similar trend. With an increase in prior cycling duration, there was an increase in the initial strain jump followed by an increase in the slope of the steady region, suggesting an increase in the secondary creep rate as well.

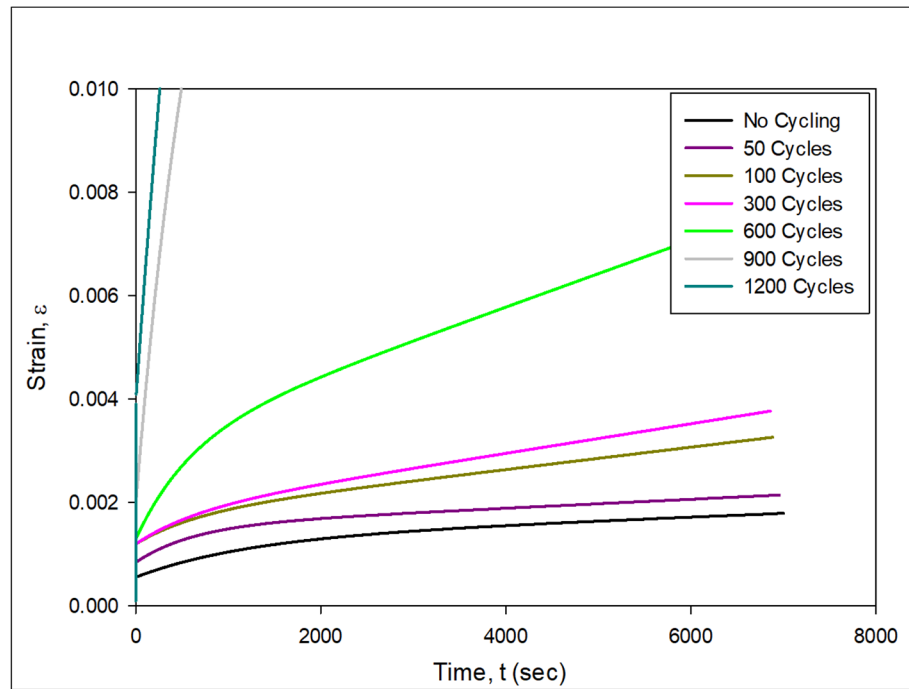


Figure 4.11 Creep Curve Evolution with Cycling (SAC305)

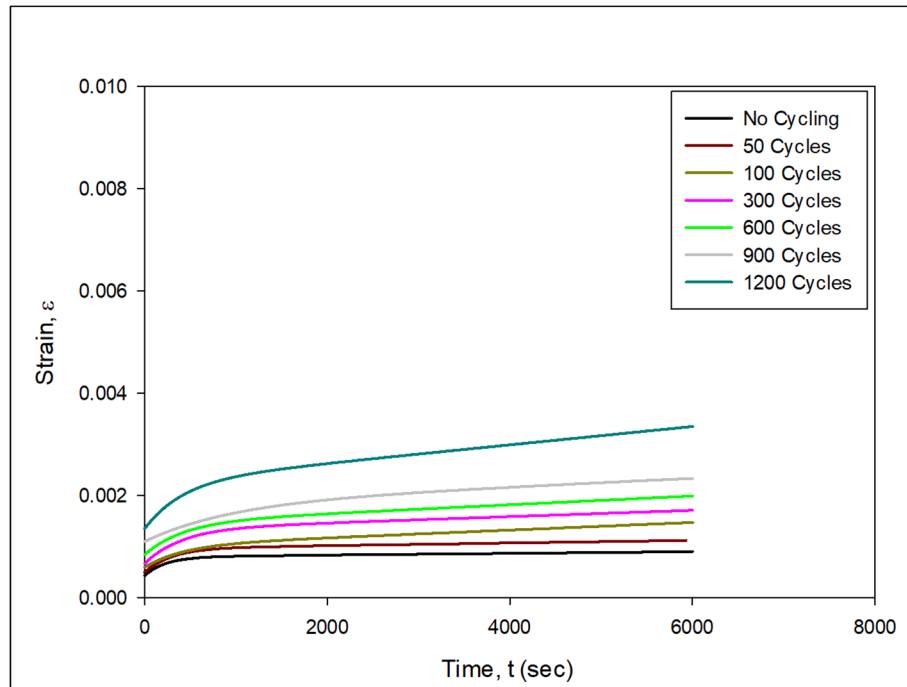


Figure 4.12 Creep Curve Evolution with Cycling (SAC_Q)

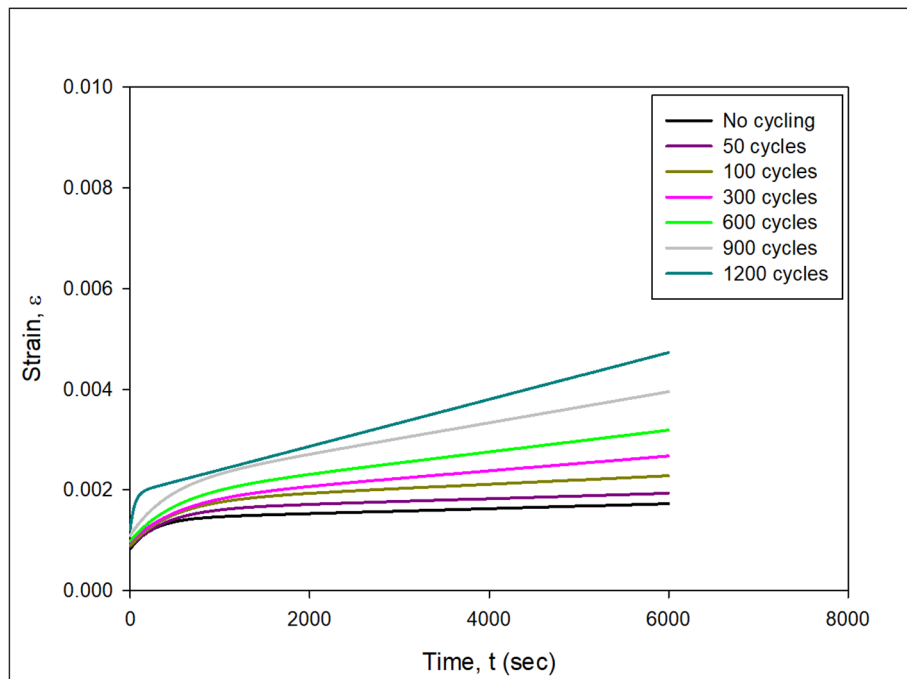
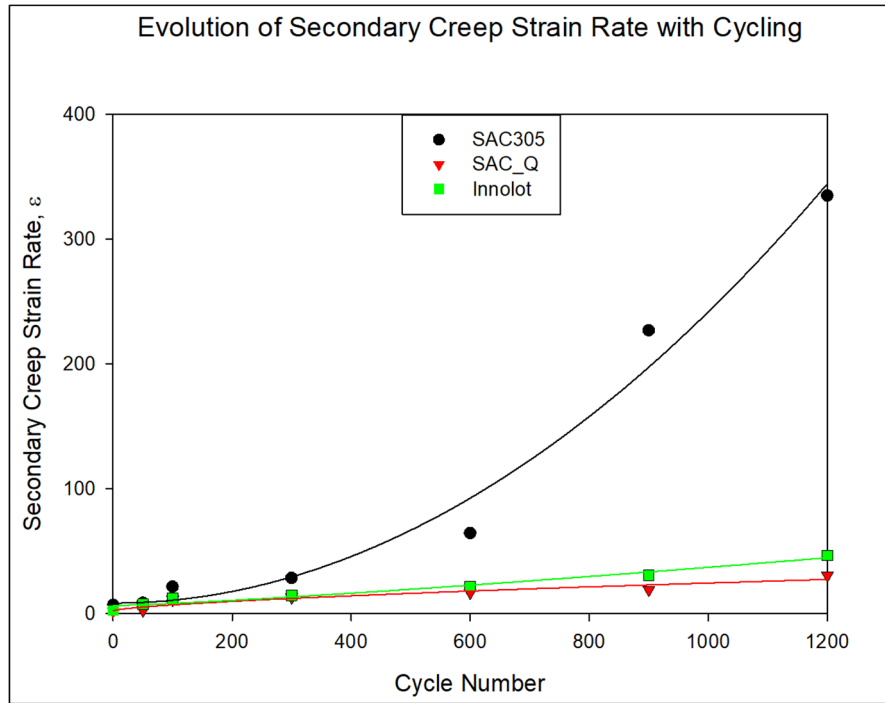


Figure 4.13 Creep Curve Evolution with Cycling (Innolot)

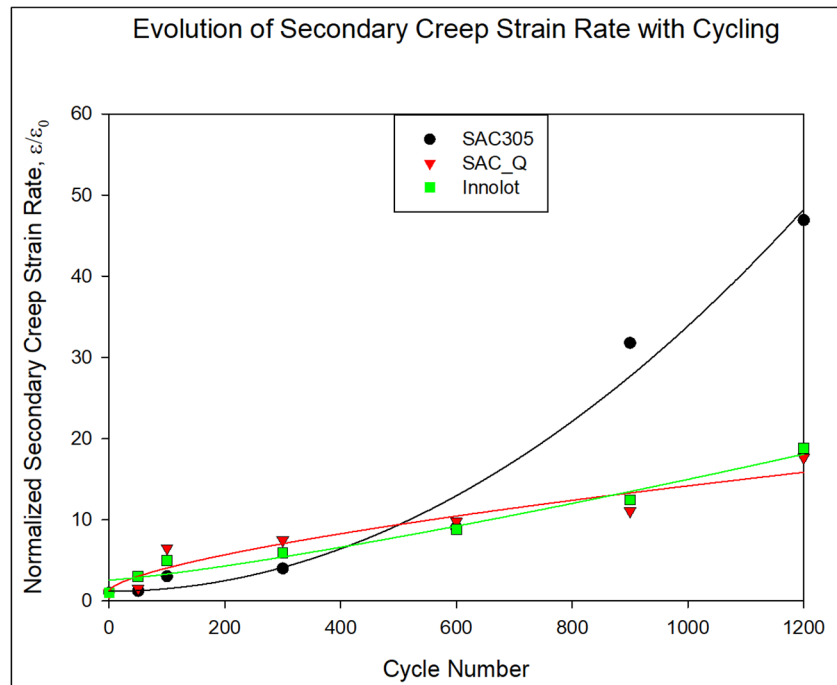
The extracted values were then tabulated in Table 4.7, while Fig. 4.14 displays the evolution of the secondary creep strain rate with number of cycles. It was observed from Fig. 4.14 that prior to 600 cycles, SAC_Q samples experienced the largest secondary creep rate and SAC305 samples the least. The scenario, however, from 600 cycles to 1200 cycles, was the complete opposite of what we saw previously. By the end of 1200 cycles, the secondary creep strain rate increased by about 46X for SAC305, by about 18X for SAC_Q, and by approximately 19X for Innolot. From all the obtained results, we can see that mechanical cycling reduces not only the cyclic properties of the alloys, but also their constitutive properties. The reduction in these properties can be explained by observing the changes in microstructure with mechanical cycling which will be covered in the following section.

Table 4.7 Evolution of Secondary Creep Strain Rate with Prior Cycling

No. of Cycles Prior to Testing	Secondary Creep Strain Rate ($\times 10^{-8} \text{ sec}^{-1}$)		
	SAC305	SAC_Q	Innolot
0	7.14	1.73	2.48
50	8.61	2.62	7.46
100	21.6	11.26	12.33
300	28.5	12.96	14.69
600	64.5	16.90	21.77
900	227	19.13	30.84
1200	335	30.54	46.63



(a) Secondary Creep Strain Rate



(b) Normalized Secondary Creep Strain Rate

Figure 4.14 Evolution of Secondary Creep Strain Rate with Cycling

4.5 Evolution of Microstructure with Mechanical Cycling

The initial stages of fatigue damage in a material are primary surface phenomena [132]. There is preferential nucleation of fatigue cracks at or near the surface due to the ease of inelastic deformation and the development of slip line steps (intrusions and extrusions) on the surface. Thus, our initial investigations on microstructural evolution during cyclic loading have concentrated on studying the specimen surface. Using the procedures discussed in the previous chapter, special polished test specimens were prepared with nanoindentation demarcations (Fig. 3.19). This facilitated the monitoring of small fixed regions of interest in the solder specimens as the cycling progressed. For the microstructure portion of this study, a total strain range of $\epsilon = \pm 0.005$ was used per cycle for all the three materials. All the samples were cycled at room temperature. Figs. 4.15, 4.16, and 4.17 show typical non-cycled microstructures for the SAC305, SAC_Q, and Innolot specimens, respectively. In all three micrographs, there are β -Sn dendrites surrounded by eutectic regions containing Ag₃Sn and Cu₆Sn₅ IMC particles. The IMC particles provide pinning points that help mitigate and block dislocation movements. The dendrites in the SAC_Q and Innolot samples were somewhat larger than those in the SAC305 samples. In addition, the doping of Bi in SAC_Q led to a dispersion of Bi rich IMC particles, which appeared white in SEM micrographs using back scattered electrons [133]. Bismuth has a high solid solubility in Sn, which leads to an improved strength of the Sn matrix by the solid solution strengthening (SSS) mechanism. For the SAC305 specimens, it was observed that the cycling induced damage consisted primarily of small intergranular cracks forming along the subgrain boundaries within dendrites.

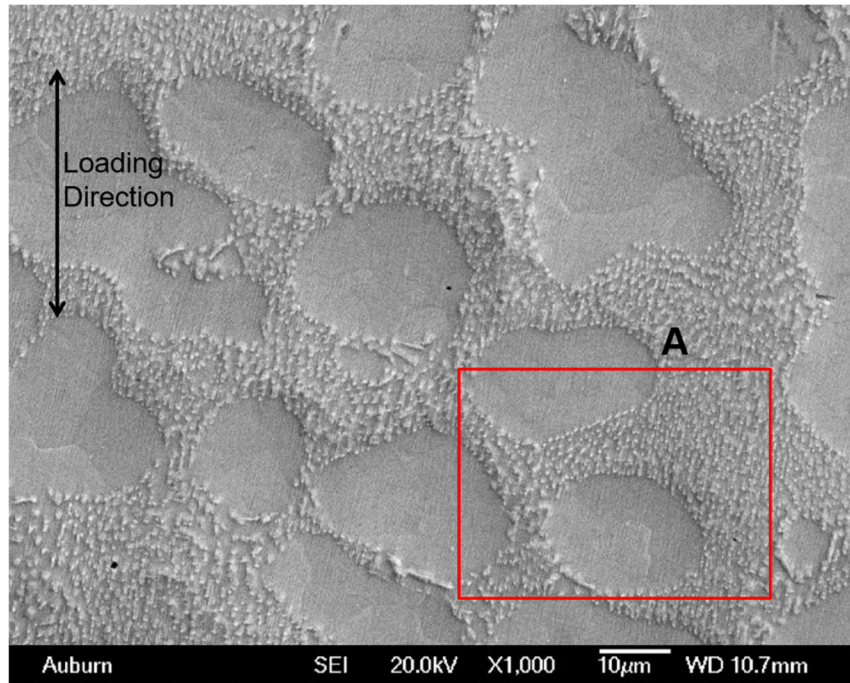


Figure 4.15 Typical Microstructure of Non-Cycled SAC305 Specimen

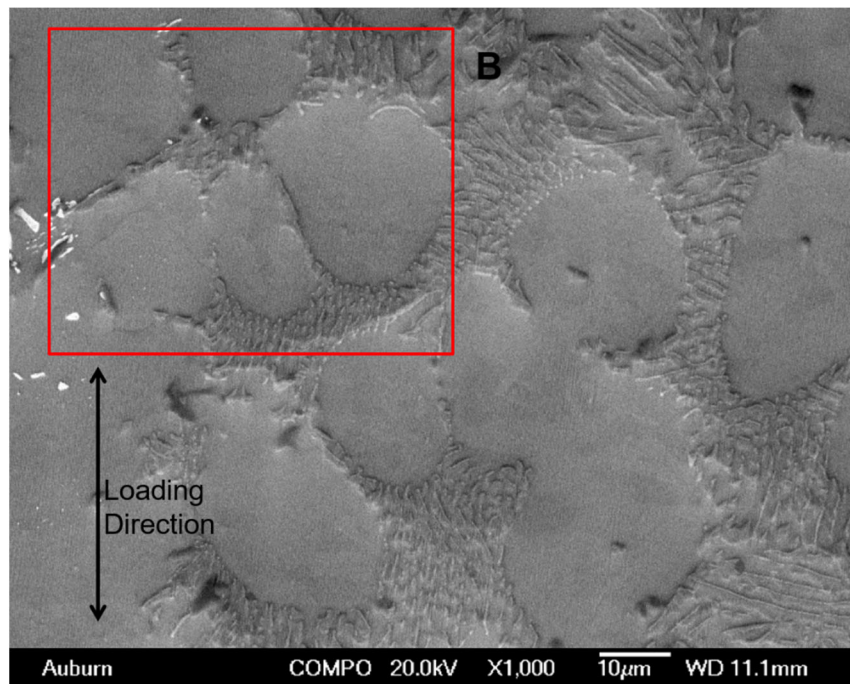


Figure 4.16 Typical Microstructure of Non-Cycled SAC_Q Specimen

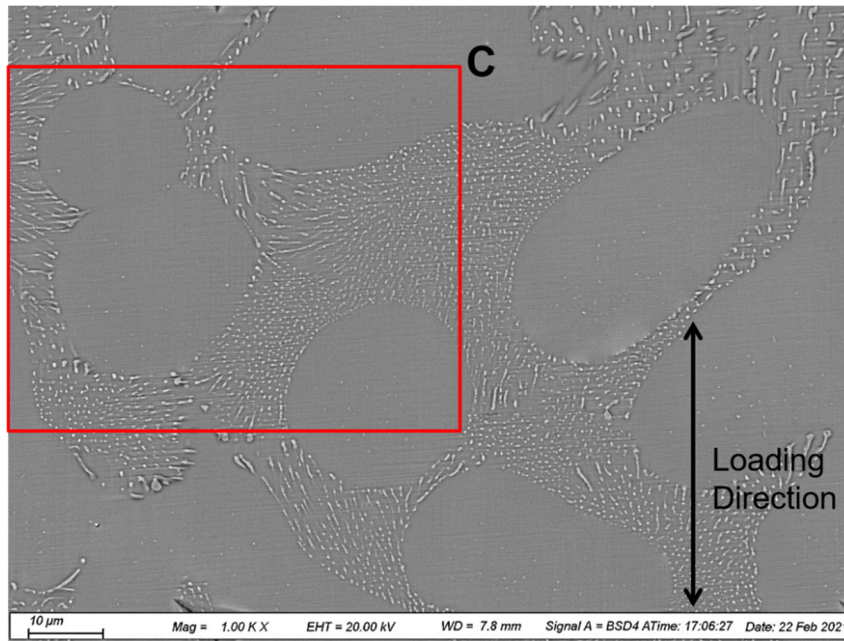


Figure 4.17 Typical Microstructure of Non-Cycled Innolot Specimen

These cracks continued to grow as the cycling progressed, resulting in a weakening of the dendrite structure, and eventually to the formation of large trans-granular cracks. The distribution and size of the intermetallic particles in the inter-dendritic regions were observed to remain essentially unchanged. For the SAC_Q specimens, inter-granular cracking was also observed, but to a lesser extent. There were also abundant slip lines visible within the dendrites of the SAC_Q specimens. In particular, slip line steps (intrusions and extrusions) that look like material wrinkles/ripples formed at the surface of the dendrites. As cycling progressed, these slip line steps were also sources of crack nucleation. With higher cycle counts, slip lines were also visible in the SAC305 specimens. For Innolot specimens, microcracks were also observed in the β -Sn dendrites. These microcracks were however found to be along the direction of the slip lines. As with the case for SAC_Q specimens, slip lines were evident in the microstructure from the

beginning of the cycling progress. These slip lines were also sources of crack nucleation for the Innolot samples. As the cycling progressed, the intensity and number of slip lines also increased. The crack propagation with cycling was also along these slip lines, with the cracks coalescing to form a larger trans-granular crack at higher durations of cycling. Example micrographs illustrating the evolution of damage during cyclic loading are shown in Figs. 4.18, 4.19 and 4.20 for SAC305, SAC_Q and Innolot specimens, respectively. The regions that were monitored were regions A, B, and C (red boxes) shown in Figs 4.15 – 4.17. For each region, the microstructures are shown for 0, 25, 50, 75, 100, 200, and 300 loading cycles. For all three materials, the initial micro cracks formed within the first 50 loading cycles. These cracks continued to grow as the cycling continued, resulting in a weakening of the dendrite structure, and eventually to the formation of large transgranular cracks. Thus, one can infer that the reductions in the various mechanical properties discussed above were due to this type of damage accumulation. In both alloys, no major changes were observed in the eutectic region or the IMCs, suggesting that they do not play significant roles in the propagation of the microcracks. The lack of clarity in the images at higher cycles was due to the sample surfaces developing non-flat topography. Significant numbers of slip lines were visible in the SAC_Q and Innolot microstructure just after 25 cycles, while they became prominently visible after 100 cycles for SAC305. This phenomenon was probably due to the larger amount of energy stored within the doped alloys than in SAC305 when cycled at the same strain range. Despite the deceiving appearances, the microstructures do not get better with mechanical cycling. This is largely due to a difference in the levels of contrast and brightness between the previous micrograph

making it look like the microstructure gets better with mechanical cycling, but it is the contrary that occurs.

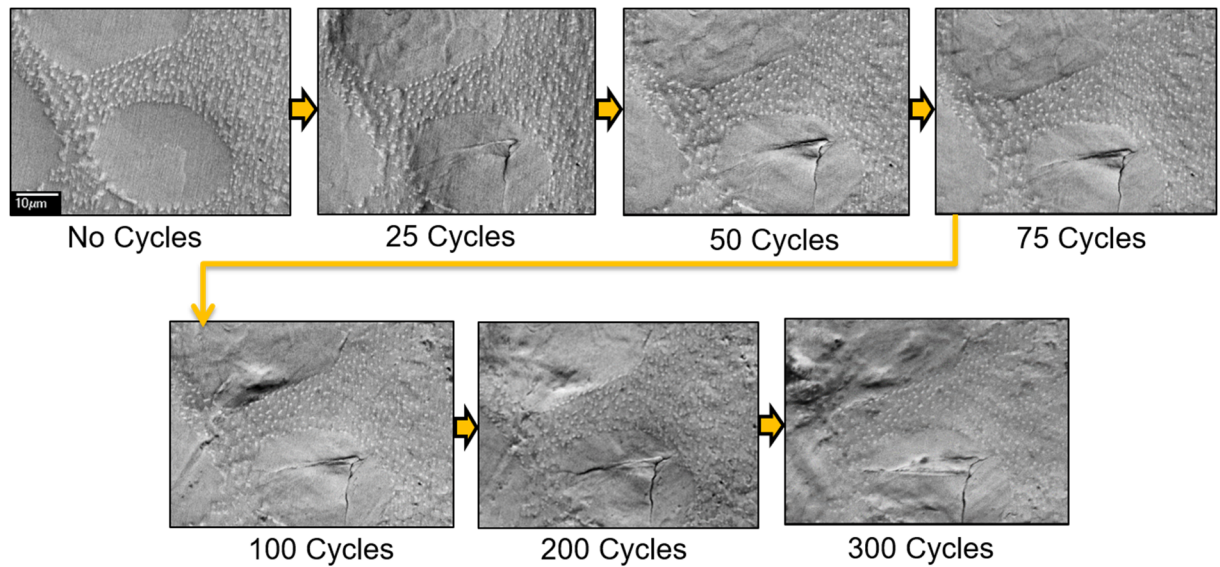


Figure 4.18 Evolution of Microstructure with Mechanical Cycling (SAC305 – Region A)

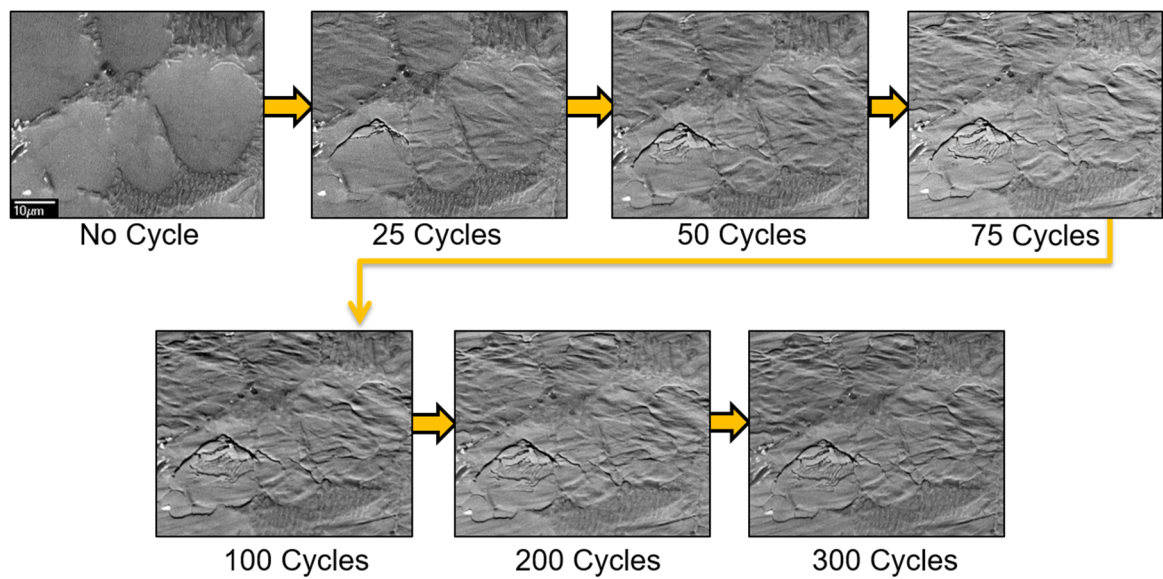


Figure 4.19 Evolution of Microstructure with Mechanical Cycling (SAC_Q – Region B)

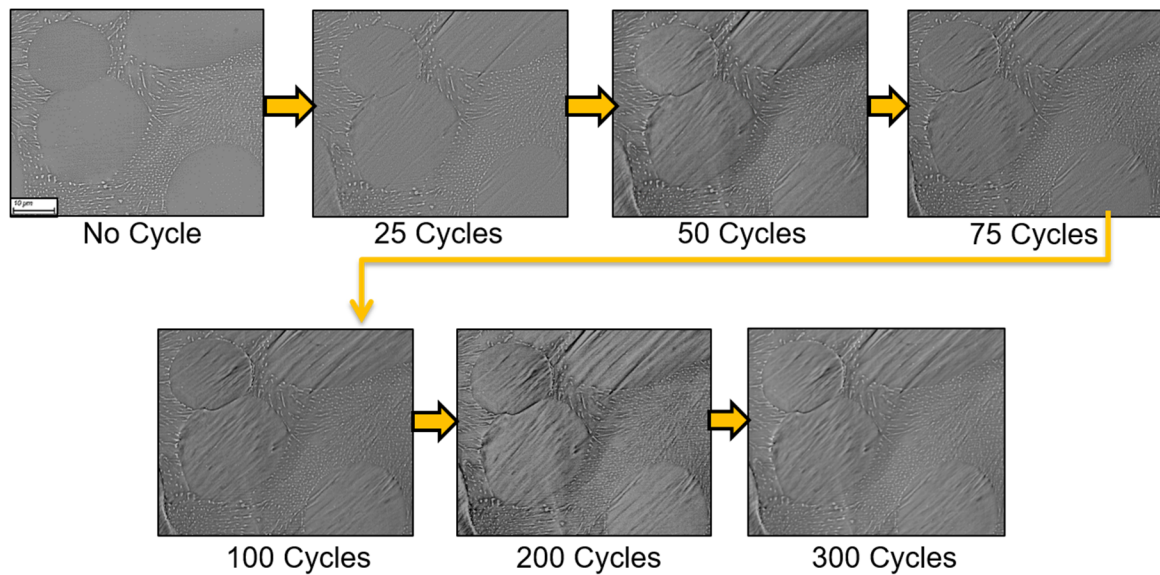


Figure 4.20 Evolution of Microstructure with Mechanical Cycling (Innolot – Region C)

4.6 Summary and Discussion

In this chapter, the effects of isothermal mechanical cycling on damage accumulation in lead free SAC305, SAC_Q (SAC+Bi), and Innolot (6 Element Alloy) lead free bulk solder alloys have been explored. In particular, we have examined both the evolution of the solder microstructure and the reductions occurring in the solder constitutive behavior (stress-strain and creep) as cycling progresses during fatigue testing. Such knowledge is necessary to develop accurate fatigue criteria for lead free solder alloys. In addition to quantifying damage accumulation during cyclic testing, we have also measured the evolution of the cyclic stress-strain behavior (hysteresis loop area, plastic strain range, and peak stress) during single fatigue tests run to failure. For the mechanical cycling experiments, cylindrical uniaxial SAC305 and SAC_Q lead free solder test specimens have been prepared using a vacuum suction reflow process within glass tubes. To study the evolution of the cyclic stress-strain behavior during a single fatigue test, the

uniaxial specimens have been subjected to cyclic stress/strain loading at constant temperature until fatigue failure. The cyclic stress-strain curves after various durations of cycling have been extracted, and a four parameter hyperbolic tangent empirical model has been used to fit the entire cyclic stress-strain curve at each chosen increment of cycling. From the recorded cyclic stress-strain curves, the evolution of the solder hysteresis loops with number of cycles has been characterized. Also, the dependencies of the hysteresis loop area (plastic strain energy density), plastic strain range, and peak stress on the duration of cycling have been studied. The first two parameters are often considered to be driving forces for fatigue damage, and used in fatigue life estimation models, such as Morrow model and Coffin-Manson model. Near fatigue failure, the decreases in loop area and peak stress were similar for all the three alloys. The plastic strain range remained essentially constant during the cycling for both materials. To study the effects of cycling on damage accumulation and changes in the constitutive behavior of SAC305, SAC_Q, and InnoLot, uniaxial samples have been prepared and subjected to various durations of prior isothermal mechanical cycling (e.g. 0, 50, 100, 300, 600, 900, 1200 cycles) that were below the fatigue life of the materials. Thus, the prior cycled samples contain different amounts of cycling induced material damage. The cycled samples were then subsequently subjected to stress-strain or creep testing. Using the data from the various tests, we have been able to characterize and quantify the cycling induced damage through the observed reductions of several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) with the amount of prior cycling. The decrease in the creep strain rate was especially significant for both materials due to exacerbated grain boundary sliding from intergranular fatigue cracking. The cycling induced material property evolutions observed

in the two lead free alloys have been correlated with the observed changes in their microstructures that occurred during the cycling. Special polished test specimens were prepared so that the microstructural evolution of a small fixed region in the solder specimen could be followed as the cycling progressed. It was observed that the cycling induced damage consisted primarily of small intergranular cracks forming along the subgrain boundaries within dendrites. These cracks continued to grow as the cycling continued, resulting in a weakening of the dendrite structure, and eventually to the formation of large transgranular cracks. The distribution and size of the intermetallic particles in the interdendritic regions were observed to remain essentially unchanged.

CHAPTER 5

EFFECT OF ELEVATED TEMPERATURE CYCLING ON THE PROPERTIES OF LEAD FREE SOLDER ALLOYS

5.1 Introduction

In the previous chapter, the accumulation of damage in several solder materials (SAC305, SAC+Bi, and SAC+Bi-Ni-Sb) during mechanical cycling at room temperature was investigated. Circular cross-sectioned solder specimens were first reflowed, and these samples were then mechanically cycled for various durations using a Micro-Mechanical tester. Cyclic stress-strain, monotonic stress-strain, and creep tests were then conducted on the prior cycled samples. The cyclic stress-strain curves obtained were studied to observe the evolution of hysteresis loop properties (peak stress, hysteresis loop area, and plastic strain range) with mechanical cycling. The monotonic stress-strain and creep test data were plotted, and several mechanical properties were characterized for various levels of cycling. Using the data from these tests, we have been able to characterize and quantify the cycling induced damage through the observed reductions of several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) with the amount of prior cycling. All of the mechanical cyclic testing in our prior work and the work of others has been performed at room temperature ($T = 25\text{ }^{\circ}\text{C}$), and thus cannot be easily extended to thermal cycling applications.

In this chapter, we have extended our prior study to examine mechanical cycling at elevated temperature. In particular, we have examined lead free solders subjected to high temperature mechanical cycling at $T = 100\text{ }^{\circ}\text{C}$. Two solder alloys, SAC305 and SAC+Bi

(SAC_Q), have been investigated. The findings for samples cycled at $T = 100\text{ }^{\circ}\text{C}$ will be demonstrated, and compared to the analogous results obtained for cycling at $T = 25\text{ }^{\circ}\text{C}$.

Uniaxial tensile tests and creep tests at room temperature were also conducted on specimens that had been previously mechanically cycled for various durations (e.g. 0, 50, 100, 300, 600 cycles) at high temperature. This allowed us to study the changes in the constitutive behavior of the solder alloys that occurred during the high temperature mechanical cycling due to the fatigue damage that builds up in the specimens. In particular, the reductions of the elastic modulus, yield strength, ultimate strength, and the secondary creep strain rate with duration of high temperature mechanical cycling were evaluated. These values were compared to the analogous results obtained for room temperature cycling in our previous study.

The reductions in the properties that occur during mechanical cycling were also correlated with the corresponding changes in the microstructure of the specimens. Rectangular cross-sectioned samples of the two lead free solder alloys were polished and selected regions indented to track the changes in the microstructure of a fixed region with mechanical cycling at $T = 100\text{ }^{\circ}\text{C}$. Using the results of this study, we are working to develop better fatigue criteria for lead free solders which are subjected to variable temperature applications.

5.2 Evolution of Cyclic Stress-Strain Curves

In this section, the evolution in the cyclic stress-strain curves is presented. The samples were subjected to mechanical cycling at a constant strain range of $\varepsilon = \pm 0.01$. The results obtained for the cyclic tests conducted at room temperature were discussed in the previous chapter. The hysteresis loops obtained for SAC305 samples cycled at $100\text{ }^{\circ}\text{C}$

have been presented in Figure 5.1. The cycling duration for which the curves were generated have also been indicated in the graph. Both curves (cycling at 25 °C and cycling at 100 °C) exhibited the same trend for the decline in peak stress with progression of cycling. The peak stress for both the cases was observed to have a rapid decline initially, followed by a steady decrease in the peak stress. Prior to failure, the samples experience another drastic decrease in peak stress. A 50% decline in initial peak stress was considered the failure criteria in this study.

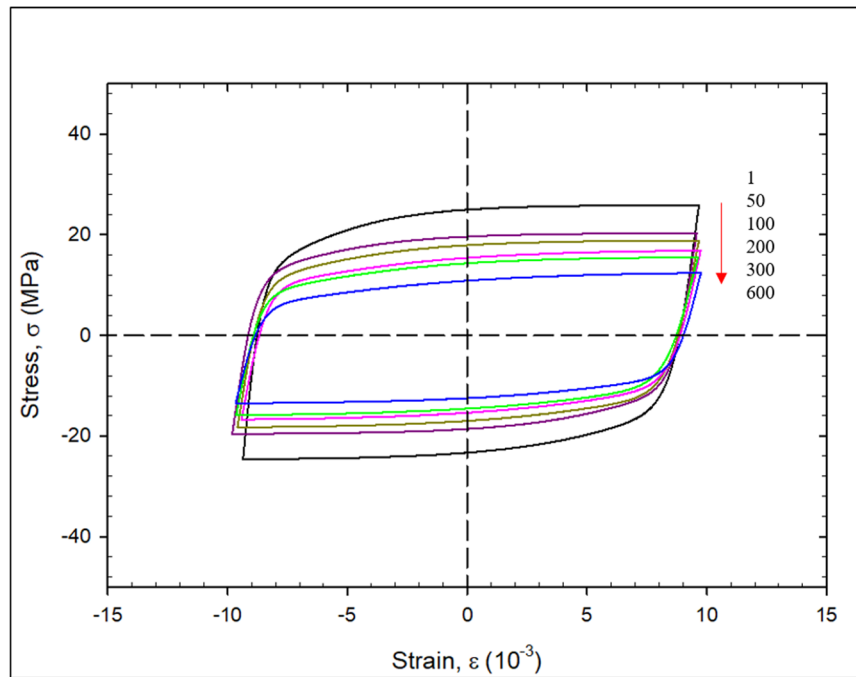


Figure 5.1 Changes in hysteresis loops of SAC305 cycled at T = 100 °C

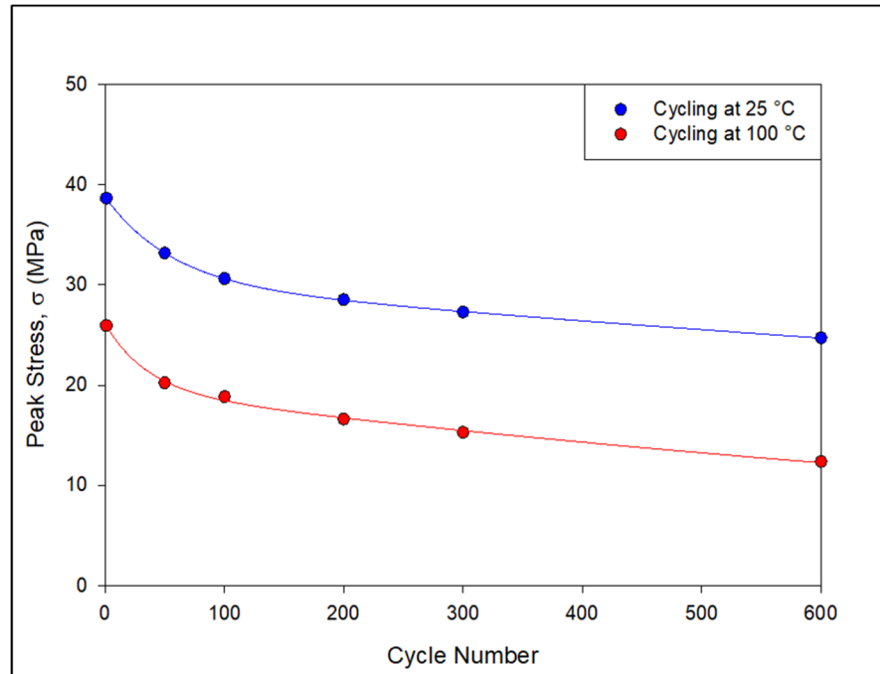
Three cyclic parameters (peak stress, hysteresis loop area, and plastic strain range) were evaluated from the hysteresis loops obtained at various cycle numbers (N= 0, 50, 100, 200, 300, and 600). The plastic strain range (distance between the two strain axis intercepts) was observed to remain approximately constant with cycling. The loop area is an indication of the plastic energy dissipated with each mechanical cycle. This was

calculated from the area between the curves. The hysteresis loop area evolved with each cycle. This was primarily due to the decrease in peak stress as the plastic strain range remained unchanged. The decrease in peak stress is brought about by the damage accumulation within the sample from the applied plastic work. The numerical values of the three cyclic parameters obtained are tabulated in Table 5.1.

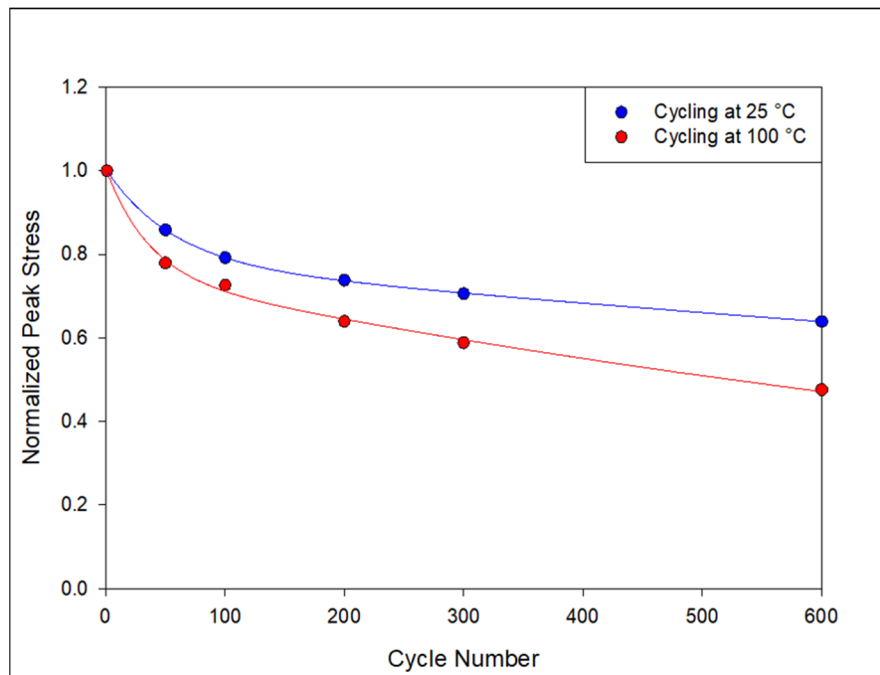
Table 5.1 Cyclic Induced Changes of hysteresis loop area, peak stress, and plastic strain range (SAC305)

Number of Cycles	Loop Area (MJ/m ³)		Peak Stress (MPa)		Plastic Strain Range (%)	
	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C
1	1.16	0.81	38.65	25.94	1.65	1.78
50	0.88	0.64	33.18	20.22	1.63	1.78
100	0.80	0.59	30.62	18.83	1.61	1.78
200	0.74	0.53	28.52	16.59	1.62	1.76
300	0.71	0.49	27.28	15.27	1.59	1.76
600	0.62	0.39	24.71	12.35	1.59	1.76

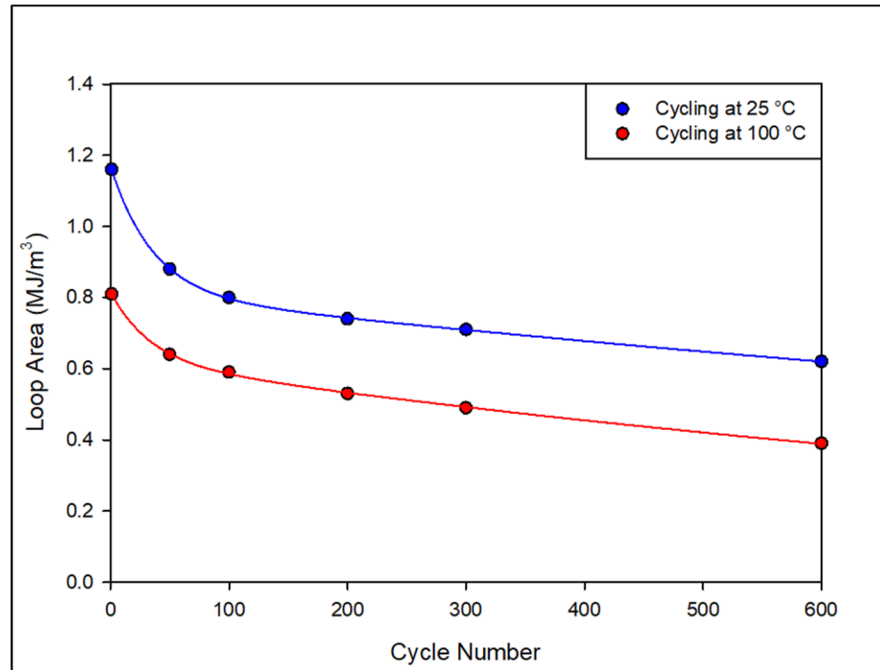
By comparing the results in Table 5.1, it was noted that the elevated temperature had an instant effect on the magnitude of the peak stress for both alloys and this reduction was more severe for SAC305. Cycling at 100 °C also reduces the fatigue life as elevated temperature cycling introduces a larger damage accumulation when cycled at the same strain range. This is evident from the results discussed in the following sections and from the microstructural study. The average fatigue life of SAC305 specimens decreased from about 1228 cycles when cycled at 25 °C to about 637 cycles when cycled at 100 °C. The hysteresis loop area evolution exhibited a similar trend as that observed with the peak stress when cycled at a higher temperature. Graphical comparisons of these evolutions are illustrated in Fig. 5.2.



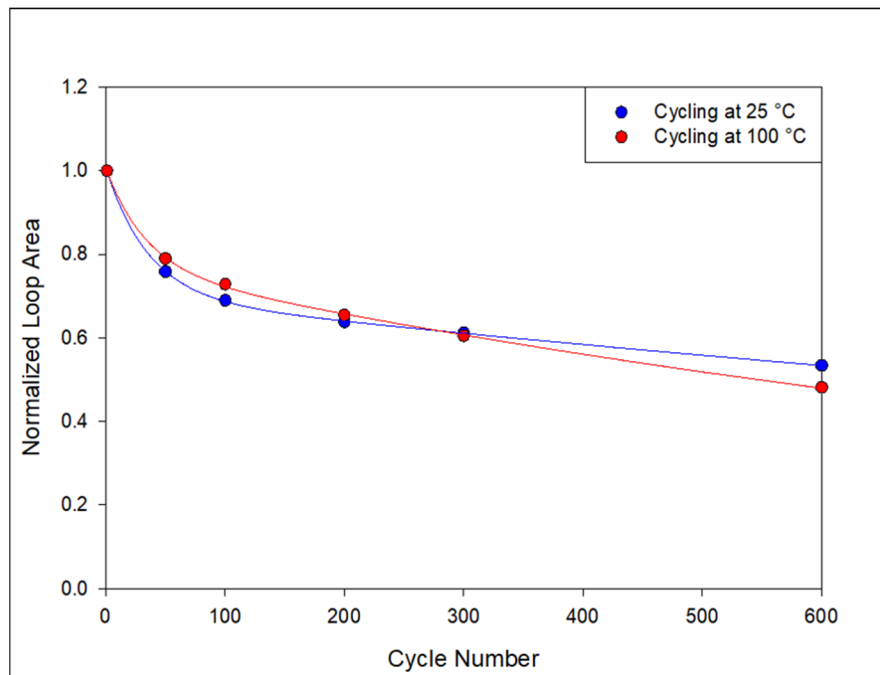
a) Reduction of Peak Stress with Cycling



b) Reduction of Normalized Peak Stress with Cycling



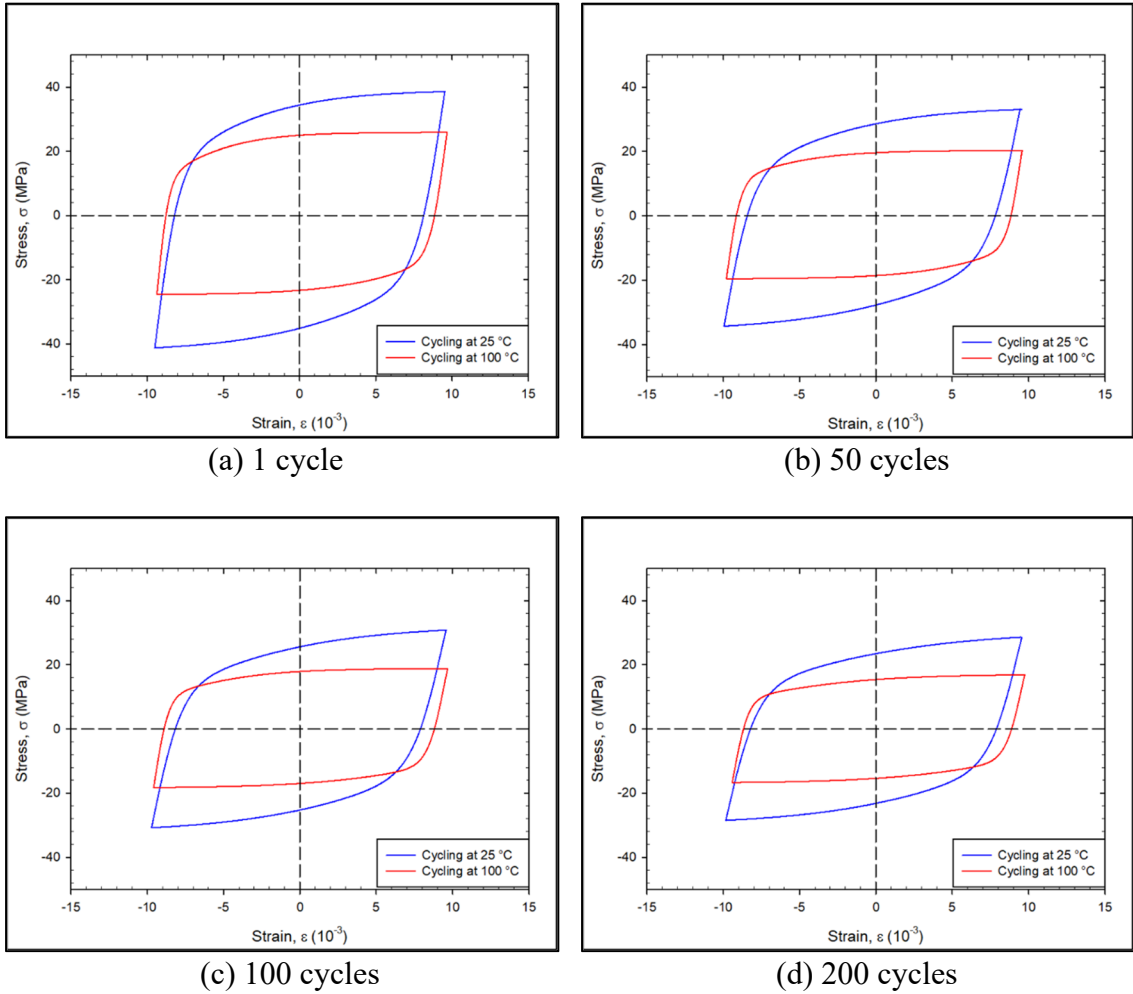
c) Reduction of Loop Area with Cycling



d) Reduction of Normalized Loop Area with Cycling

Figure 5.2 Reduction of Properties with Isothermal Mechanical Cycling (SAC305)

Figure 5.3 consists of a set of curve which show the evolution in the hysteresis loops with number of cycles for both the cycling temperatures. The blue curves represent the hysteresis curves obtained when SAC305 samples were cycled at room temperature, while the red curves represent the hysteresis loops obtained when SAC305 samples were cycled at an elevated temperature of 100 °C.



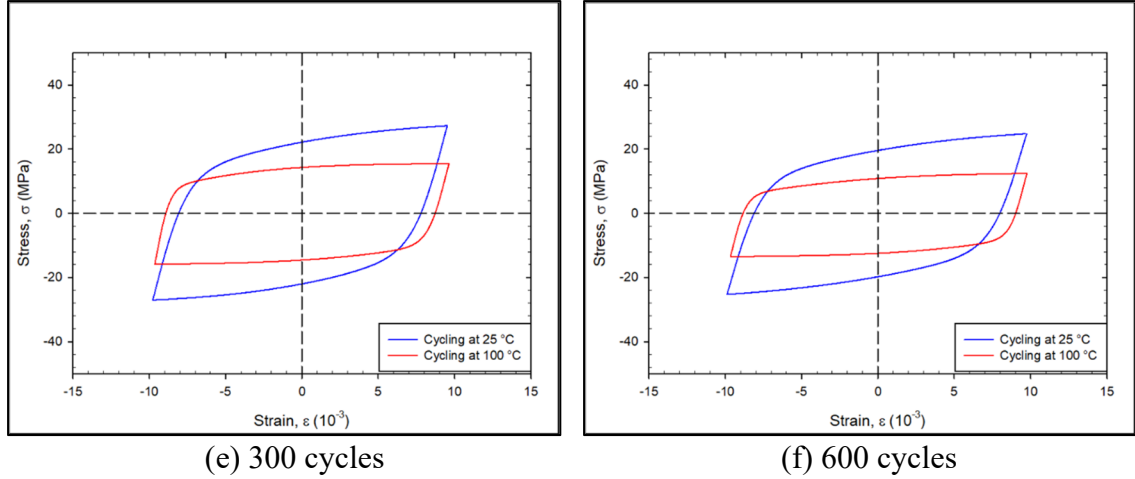


Figure 5.3 Hysteresis Loop Evolution of SAC305 with Number of cycles

Analogous curves obtained when mechanically cycling SAC_Q specimens with the same strain range at 100 °C are presented in Fig. 5.4. Straight away, we observe the higher peak stress achieved by SAC_Q. This indicates the higher strength achieved by Bismuth doped SAC alloys even at higher operating temperatures. A similar trend in evolution of peak stress was obtained for SAC_Q as that observed for SAC305. The plastic strain range for SAC_Q specimens also remained fairly constant throughout the cyclic test, but its magnitude was lower than the value obtained for the SAC305 samples. The numerical values of the three cyclic parameters obtained for SAC305 and SAC_Q specimens cycled at 100 °C are tabulated in Table 5.2. Graphical comparisons of these evolutions are illustrated in Fig. 5.5. From the data obtained, we can see that SAC_Q samples had almost 1.5 times larger loop area than SAC 305 samples when initially cycled at the same strain range at a temperature of 100 °C. The decrease in the loop area for SAC305 specimens was larger than the SAC_Q specimens, such that by the end of the 300 cycles, SAC_Q specimens had almost twice the loop area magnitude than the SAC305 specimens. The initial peak stress was observed to be almost twice the magnitude for SAC_Q and the decrease in peak stress had also declined less for the SAC_Q alloy. Overall, the evolution

in hysteresis loop area and peak stress exhibited a similar trend for the cycling of both the alloys at an elevated temperature of 100 °C. The SAC_Q samples, however, do not reach 600 cycles as the SAC305 samples. The reason behind this is the larger amount of plastic energy dissipated per cycle for SAC_Q samples. Another observation was that the decline in loop area of SAC_Q samples prior to failure was very sharp unlike that observed for SAC305 samples where a gradual decrease in loop area was obtained up to failure. All these results indicate the better cyclic performance of SAC_Q when cycled at elevated temperatures. To better compare the fatigue damage accumulation between SAC305 and SAC_Q alloys cycled at an elevated temperature, monotonic tensile tests, creep tests, and microstructural changes in a fixed location will be investigated and discussed in the following sections of this chapter.

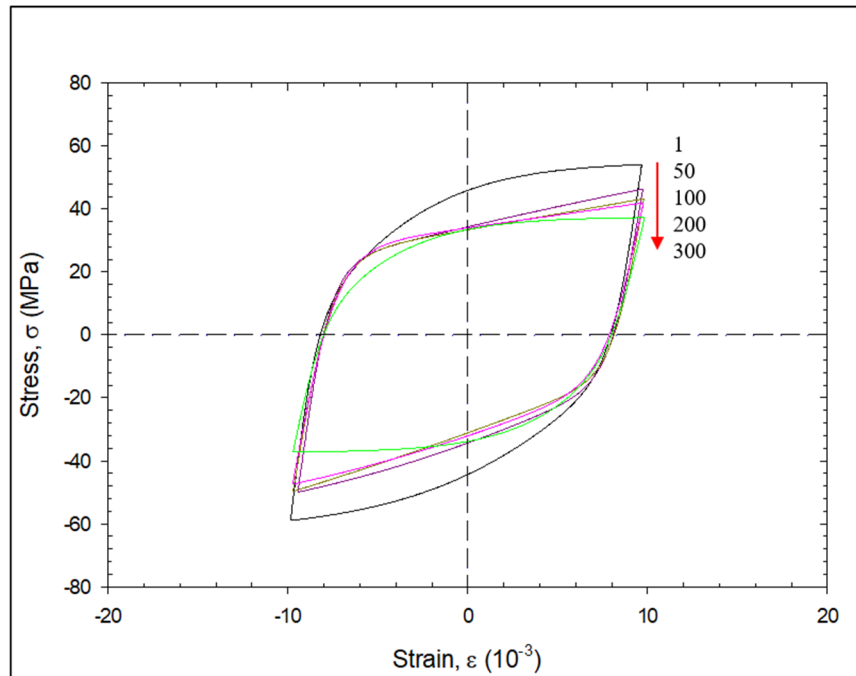
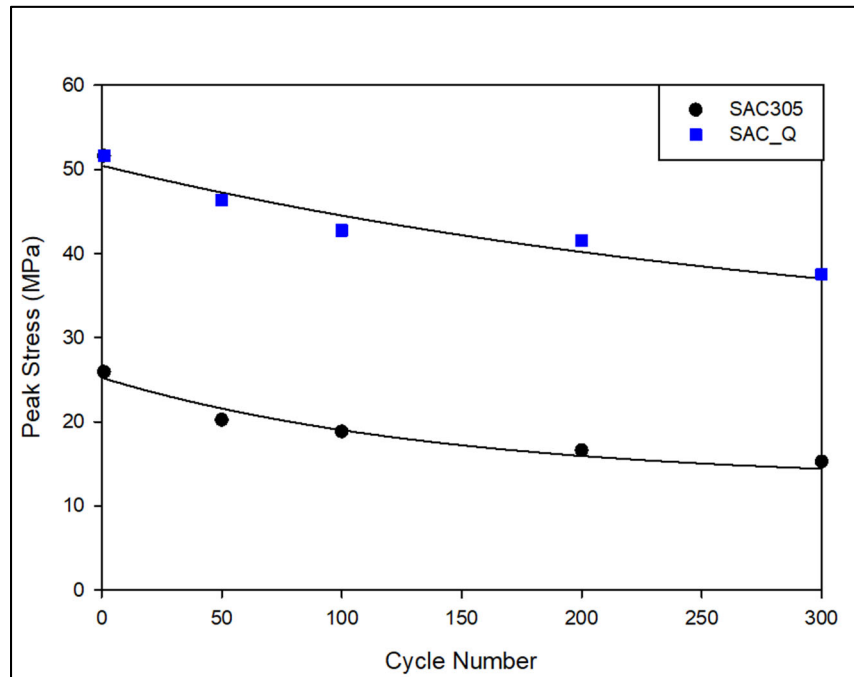


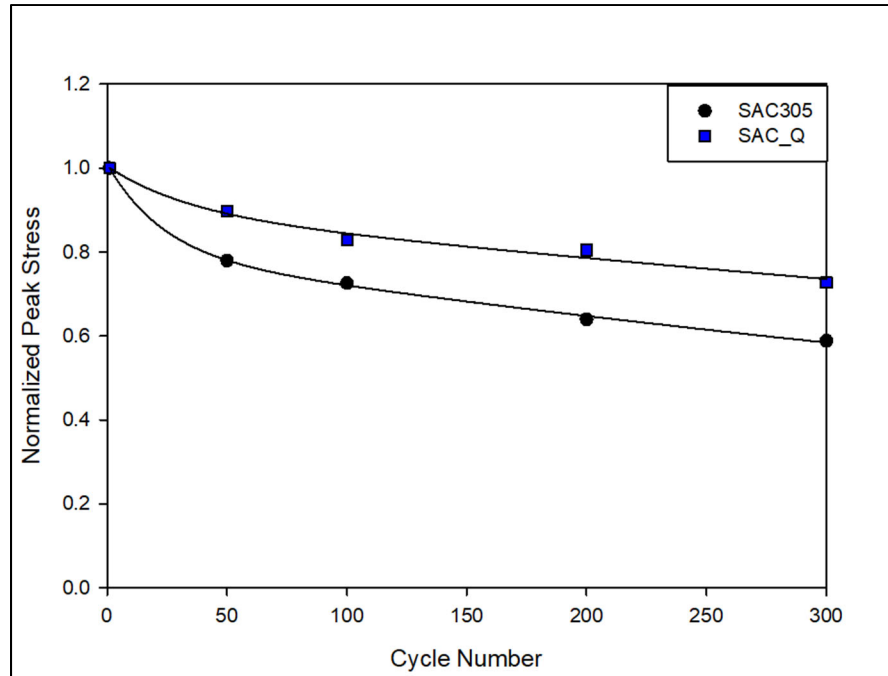
Figure 5.4 Changes in hysteresis loops of SAC_Q cycled at T = 100 °C

Table 5.2 Measured changes in loop area, peak stress, and plastic strain range during a cyclic test at 100 °C

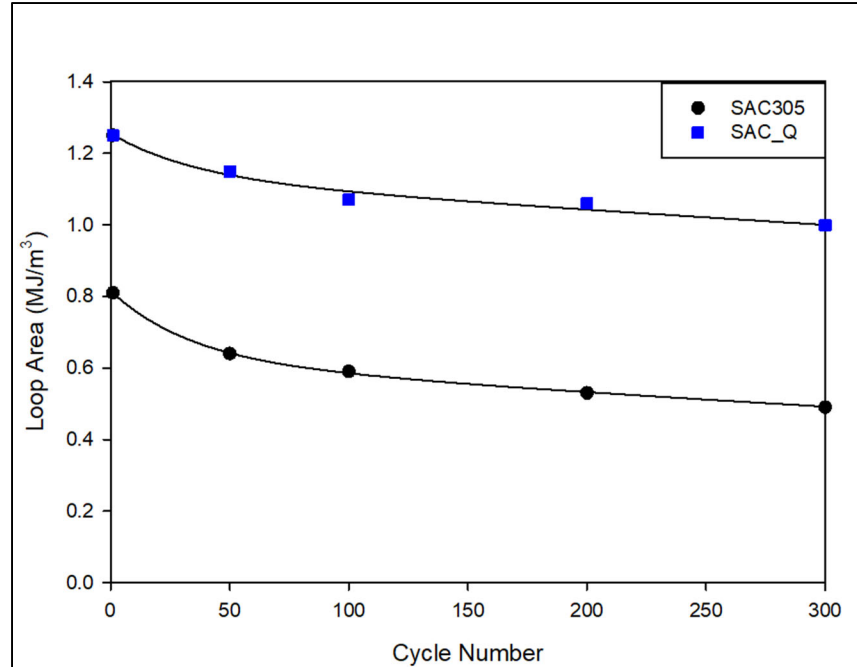
Number of Cycles	Loop Area (MJ/m ³)		Peak Stress (MPa)		Plastic Strain Range (%)	
	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C
1	0.81	1.25	25.94	51.62	1.78	1.60
50	0.64	1.15	20.22	46.35	1.78	1.64
100	0.59	1.07	18.83	42.76	1.78	1.61
200	0.53	1.06	16.59	41.55	1.76	1.63
300	0.49	1.00	15.27	37.53	1.76	1.62



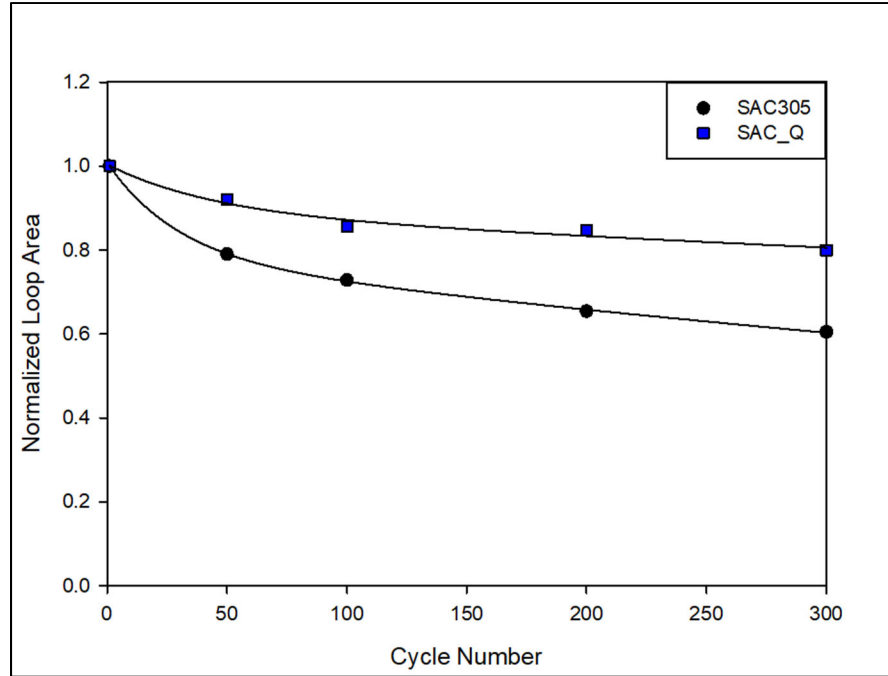
a) Reduction of Peak Stress with Cycling



b) Reduction of Normalized Peak Stress with Cycling



c) Reduction of Loop Area with Cycling



d) Reduction of Normalized Loop Area with Cycling

Figure 5.5 Reduction of Properties of SAC305 with Isothermal Mechanical Cycling

5.3 Evolution of Stress-Strain Behavior with Mechanical Cycling

This section focuses on quantifying the damage accumulation in the lead free alloys when they are mechanical cycled at an elevated temperature. This comprised of initially subjecting the uniaxial samples to prior mechanical cycles of durations above the average fatigue failure life of the alloys (e.g. 0, 50, 100, 200, 300, and 600 cycles). Once the specimens were cycled up to either of the aforementioned cycle number at 100 °C, they were stored in a freezer at -40 °C to prevent any aging effects. These samples were then subjected to monotonic tensile tests at 25 °C.

Figure 5.6 displays the measured evolution of the tensile stress-strain curves obtained for 100 °C pre-cycled SAC305 samples. Similar monotonic tensile stress-strain

curves obtained for 25 °C pre-cycled SAC305 samples was presented in Figure 4.5. The stress-strain data from five experimental tests were fit with a four parameter hyperbolic tangent model to generate an average stress-strain curve for each set of prior cycling conditions. Both curves show a similar trend. Cycling the samples for 50 cycles was sufficient to drastically reduce the Elastic modulus (initial slope) and the ultimate tensile strength (UTS, peak stress); the effect being less prominent when the samples were cycled at 25 °C. Table 5.3 summarizes the calculated values of the effective elastic modulus (E), Ultimate Tensile Strength (UTS), and yield stress (YS) for SAC305 specimens pre-cycled at both of the temperatures. The values suggest that increased damage accumulation occurs when lead free alloys are cycled at high temperatures. The changes in the elastic modulus and the UTS have been graphically presented in Figure 5.7. It was observed that cycling SAC305 at 25 °C for 600 cycles caused a reduction of 49%, 33%, and 24% in Elastic modulus, UTS, and Yield Stress, respectively. Similarly, cycling the SAC305 specimens at 100 °C for 600 cycles resulted in a decrease of 66%, 40%, and 39% in Elastic modulus, UTS, and Yield Stress, respectively. Figure 5.8 consists of a set of curves which show the evolution in tensile curves with number of cycles for both the cycling temperatures. The blue and red curves represent the tensile curves obtained when SAC305 samples were cycled at 25 °C and 100 °C, respectively, while the black curve is the tensile curve obtained for pristine SAC305 samples.

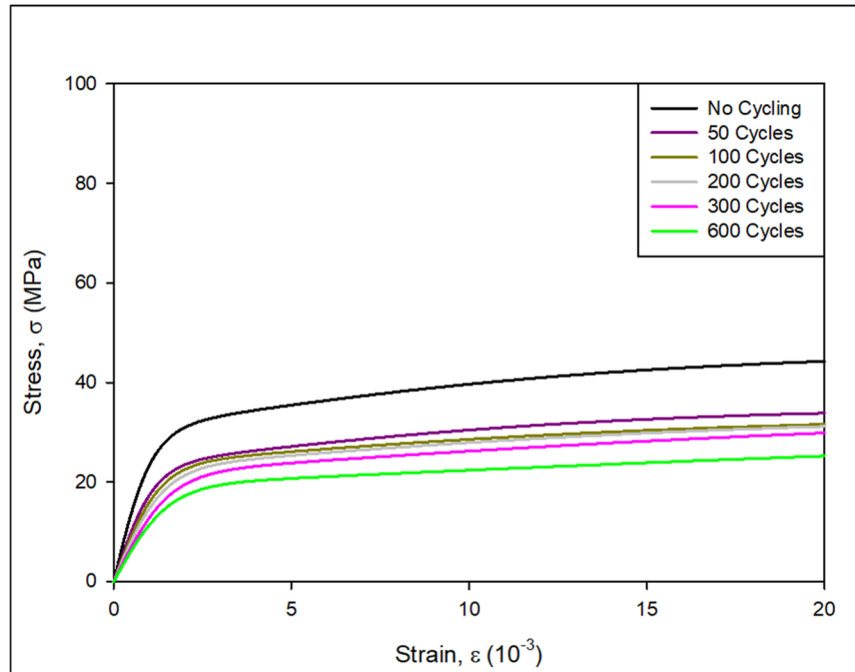
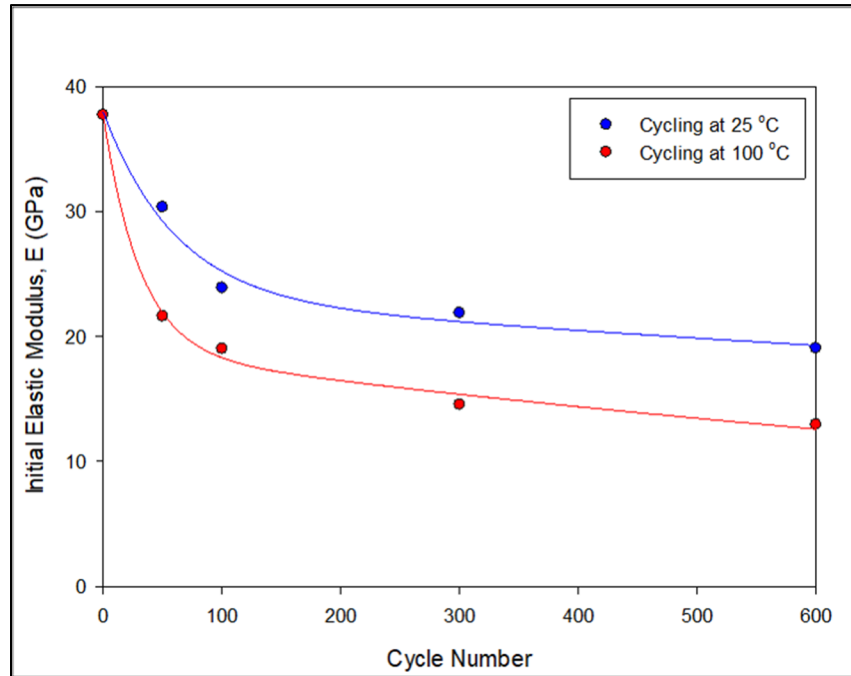


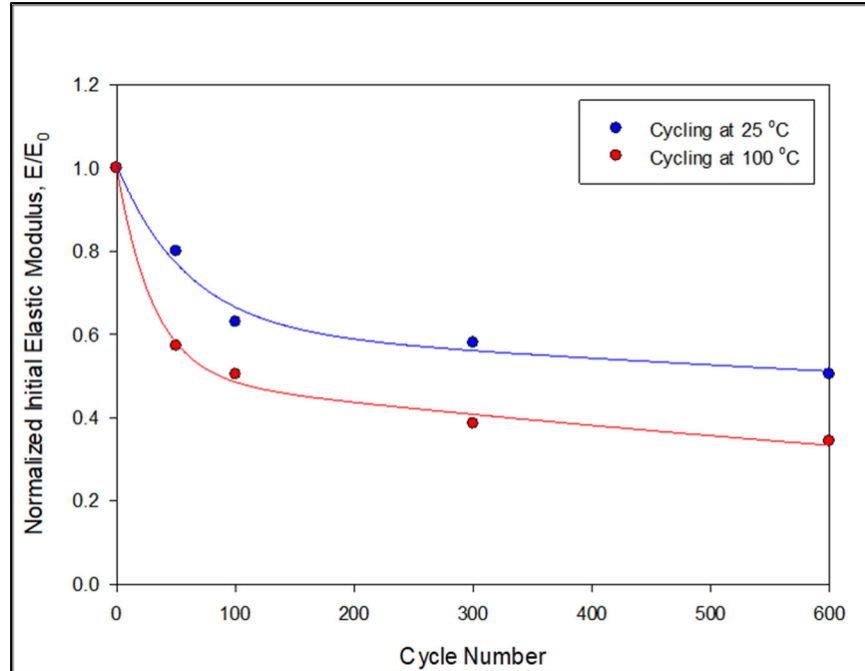
Figure 5.6 Evolution of tensile curves of SAC305 cycled at $T = 100\text{ }^{\circ}\text{C}$

Table 5.3 Evolution of Tensile Material Properties with Isothermal Mechanical Cycling (SAC305)

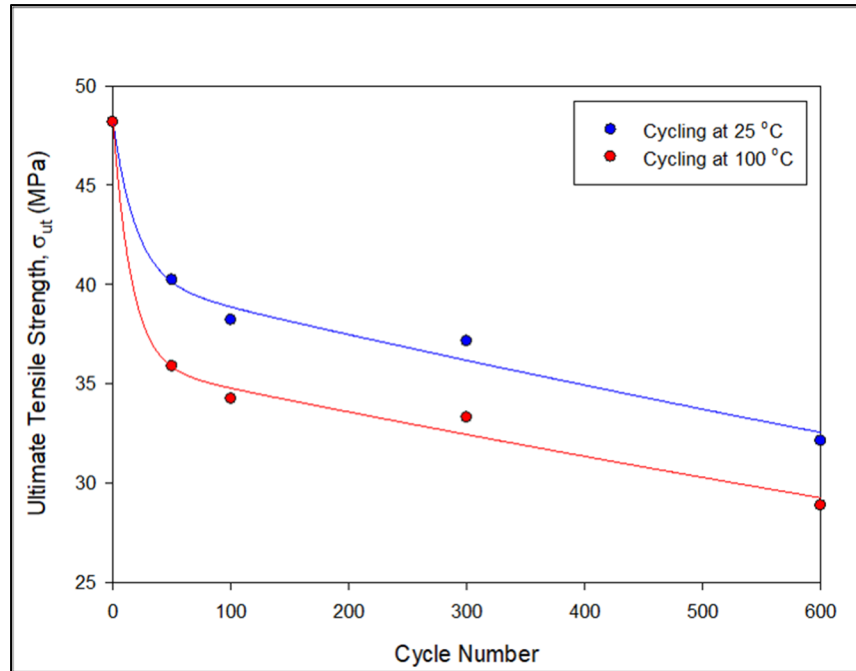
Number of Cycles	Effective Elastic Modulus (GPa)		Ultimate Tensile Strength (MPa)		Yield Stress (MPa)	
	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C	SAC305 Cycled at 25 °C	SAC305 Cycled at 100 °C
0	37.72	37.72	48.19	48.19	33.09	33.09
50	30.35	21.63	40.23	35.89	32.95	25.64
100	23.89	19.02	38.22	34.24	31.77	24.88
300	21.89	14.55	37.15	33.30	29.23	22.85
600	19.06	12.95	32.12	28.87	25.18	20.02



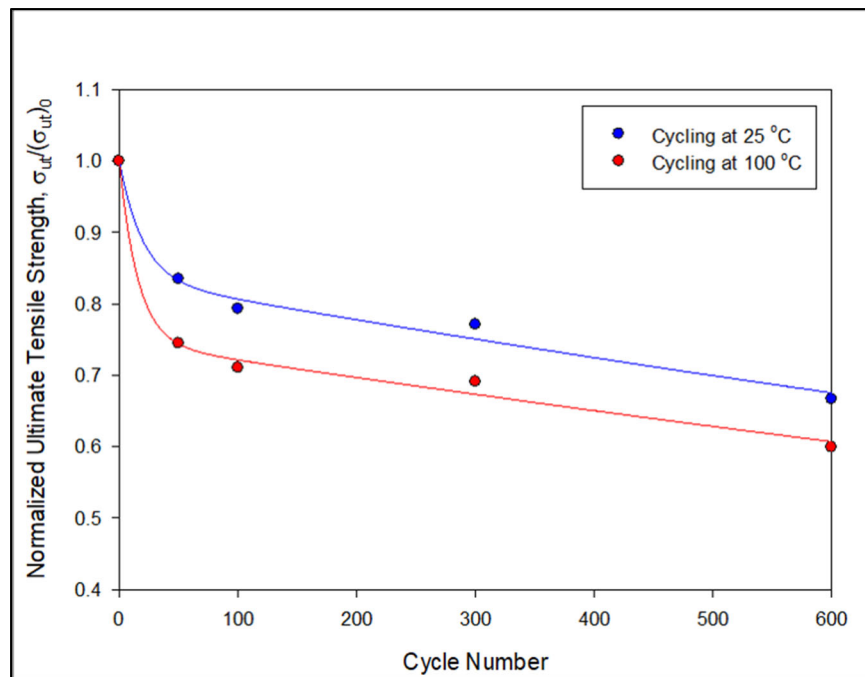
a) Reduction of Initial Elastic Modulus with Cycling



b) Reduction of Normalized Initial Elastic Modulus with Cycling



c) Reduction of Ultimate Tensile Strength with Cycling



d) Reduction of Normalized Ultimate Tensile Strength with Cycling

Figure 5.7 Reduction of Tensile Properties of SAC305 with Mechanical Cycling

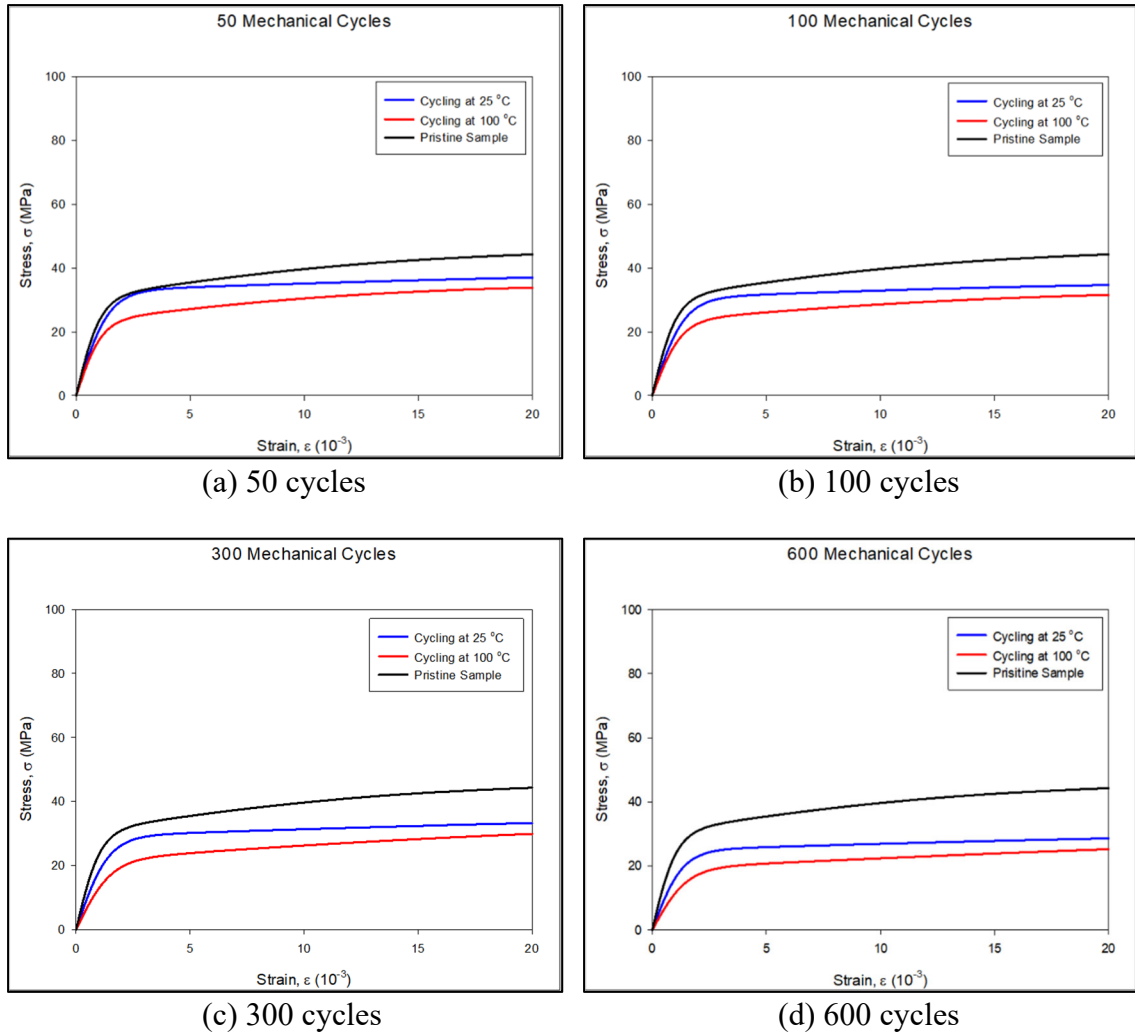


Figure 5.8 Tensile Stress-Strain Curve Evolution of SAC305 with Number of cycles

Fig. 5.9 represents the measured evolution of the tensile stress-strain curves obtained for 100 °C pre-cycled SAC_Q samples. Comparing this graph to Fig. 5.6 for SAC305, we notice that both curves exhibit a similar trend in the evolution of the curves. Cycling the samples for 50 cycles at 100 °C was sufficient to drastically reduce the Elastic modulus (initial slope) and the ultimate tensile strength (UTS, peak stress); the effect being less prominent in the doped alloy. Table 5.4 summarizes the calculated values of the elastic modulus, UTS and the yield stress for both alloys at the specified pre-cycle for a cycling

temperature of 100 °C. It was observed that cycling SAC_Q at 100 °C for 600 cycles caused a reduction of 49%, 33%, and 24% in Elastic modulus, UTS, and Yield Stress, respectively. The changes in the elastic modulus and the UTS for SAC_Q cycled at 100 °C have been graphically presented in Figure 5.10.

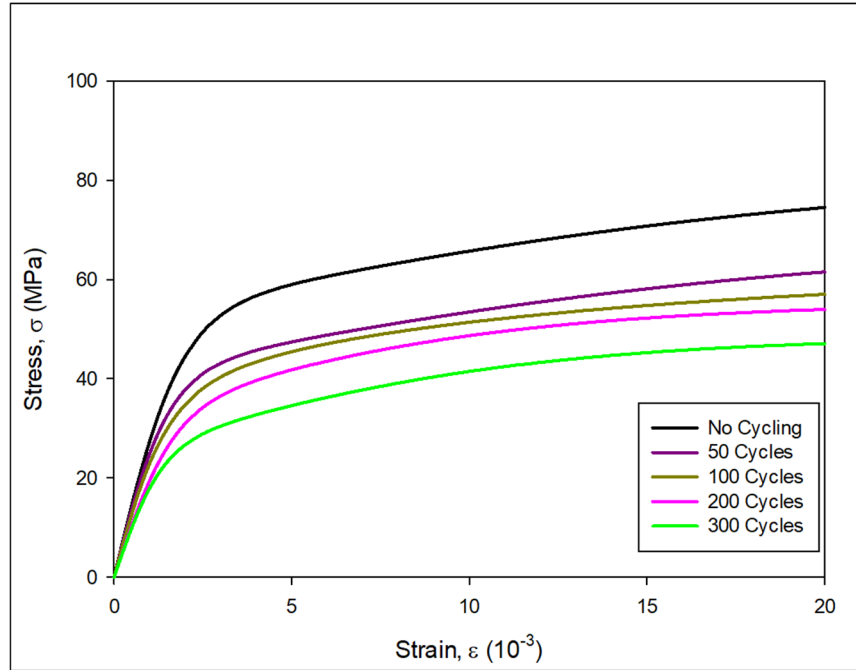
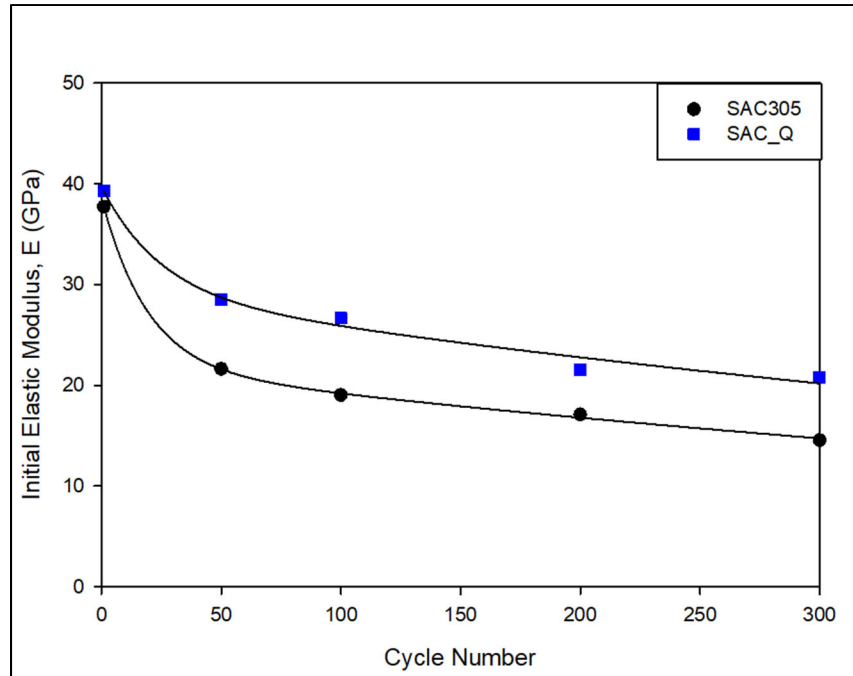


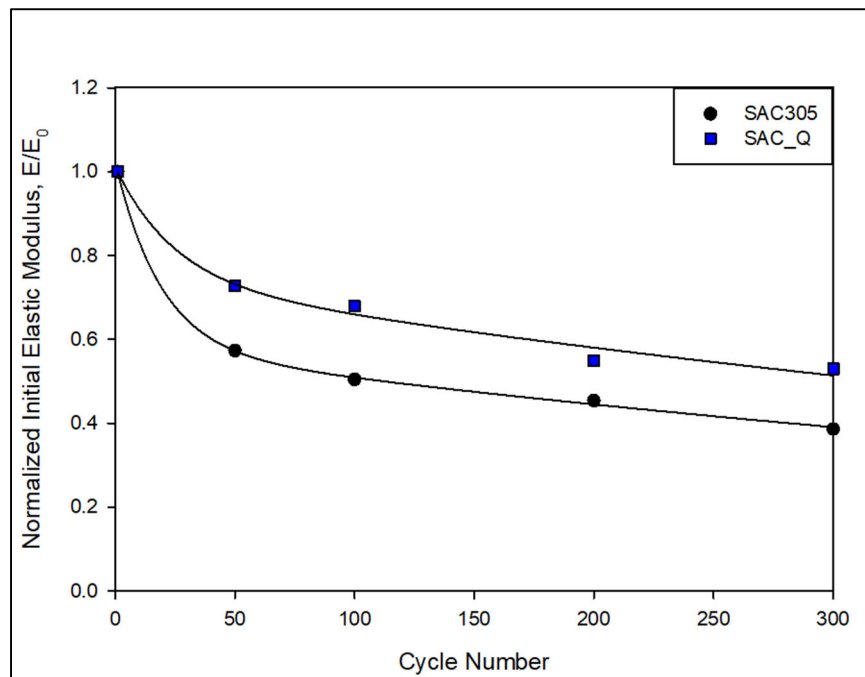
Figure 5.9 Evolution of tensile curves of SAC_Q cycled at T = 100 °C

Table 5.4 Reduction of Tensile Material Properties with Isothermal Mechanical Cycling (100 °C)

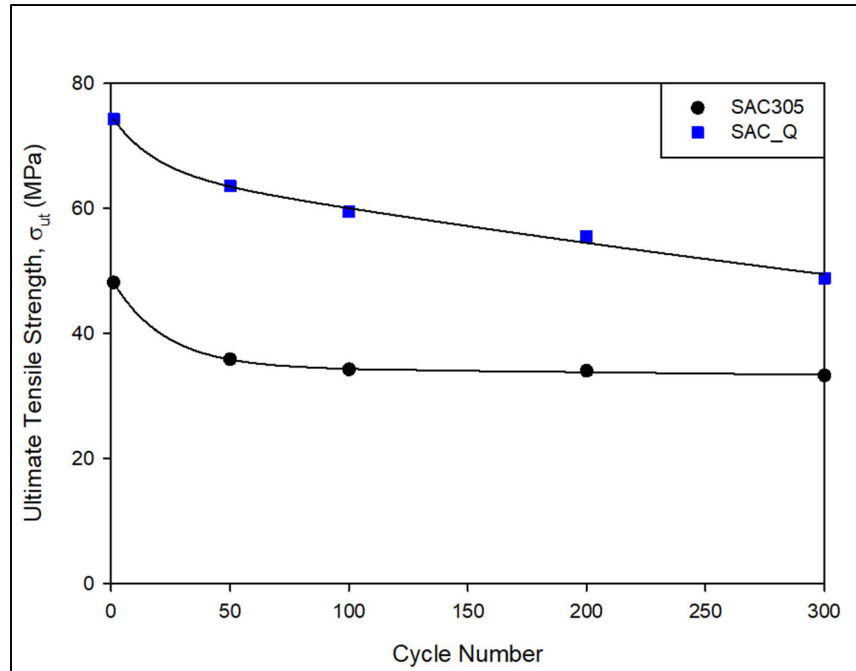
Number of Cycles	Effective Elastic Modulus (GPa)		Ultimate Tensile Strength (MPa)		Yield Stress (MPa)	
	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C	SAC305 Cycled at 100 °C	SAC_Q Cycled at 100 °C
0	37.72	39.26	48.19	74.31	33.09	49.63
50	21.63	28.54	35.89	63.60	25.64	40.06
100	19.02	26.67	34.24	59.48	24.88	35.39
200	17.11	21.59	34.02	55.62	24.00	32.15
300	14.55	20.80	33.30	48.91	22.85	30.00



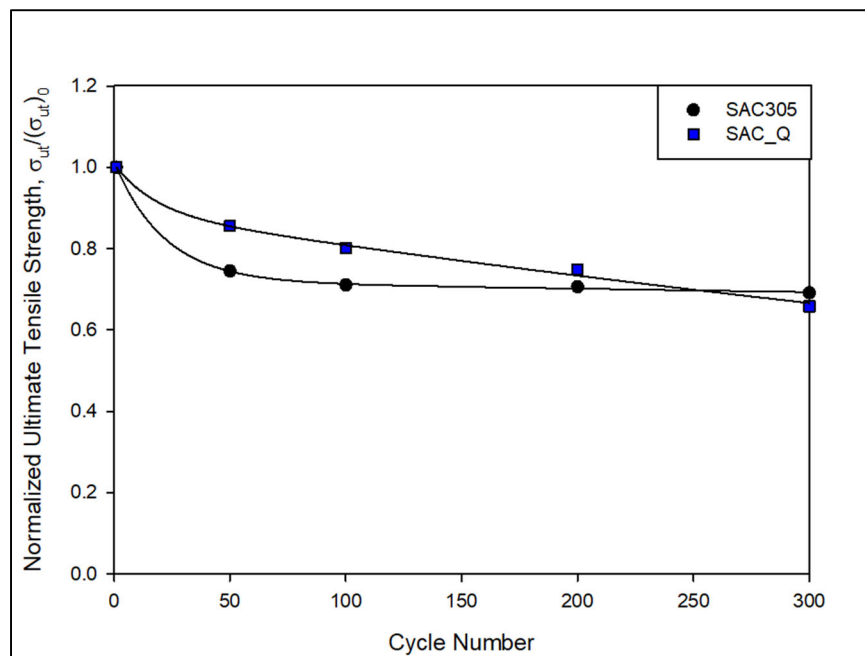
a) Reduction of Initial Elastic Modulus with Cycling



b) Reduction of Normalized Initial Elastic Modulus with Cycling



c) Reduction of Ultimate Tensile Strength with Cycling



d) Reduction of Normalized Ultimate Tensile Strength with Cycling

Figure 5.10 Reduction of Tensile Properties with Mechanical Cycling at 100 °C

From the curves in Fig. 5.10, we observe that the decline in Elastic modulus with mechanical cycling is smaller for SAC_Q than in SAC305, but both the alloys demonstrated a common trend in the reduction of Elastic modulus with mechanical cycling at elevated temperature. The UTS obtained for pristine SAC_Q samples was a lot higher than that observed for SAC305. However, the SAC_Q samples experienced a gradual reduction in the UTS up to 300th cycle, while the reduction in UTS for the SAC305 samples became quite steady after the initial large decline.

5.4 Evolution of Creep Behavior with Mechanical Cycling

The creep curves of pre-cycled SAC305 specimens cycled at $T = 100\text{ }^{\circ}\text{C}$ is presented in Fig 5.11. The corresponding creep curves obtained for pre-cycled SAC305 specimens cycled at $T = 25\text{ }^{\circ}\text{C}$ was presented in Fig 4.11. The experimental data were fit using the well-known Burger's model. The secondary creep strain rate, which is the steady state slope of the creep curve, was then evaluated from this fit and the numerical values have been presented in Table 5.5. The first noticeable difference between the results for the two temperatures is the increase in the initial strain of the creep curves for prior cycling at $T = 100\text{ }^{\circ}\text{C}$. The initial creep strain for SAC305 samples after 50 mechanical cycles at $T = 100\text{ }^{\circ}\text{C}$ was nearly 7 times the initial creep strain of pristine samples. The secondary creep strain rate for both the temperatures increased drastically with the number of cycles prior to the creep test. After 600 prior mechanical cycles, the secondary creep rate of SAC305 samples cycled at $25\text{ }^{\circ}\text{C}$ increased by about 9X (900%), while the secondary creep rate of SAC305 samples cycled at $100\text{ }^{\circ}\text{C}$ increased by about 60X (6000%). The graphical representation of the $\dot{\epsilon}$ of the secondary creep strain rate in SAC305 samples pre-cycled at

both the temperatures is presented in Figure 5.12. Figure 5.13 consists of a set of curves which show the evolution in creep curves with number of cycles for both the cycling temperatures. The blue and red curves represent the creep curves obtained when SAC305 samples were cycled at 25 °C and 100 °C, respectively, while the black curve is the creep curve obtained for pristine SAC305 samples.

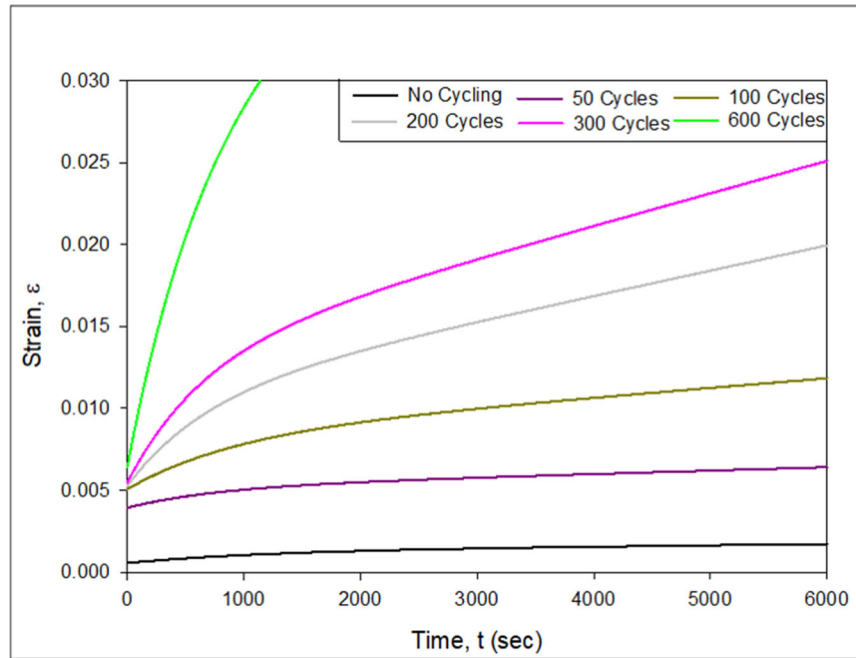
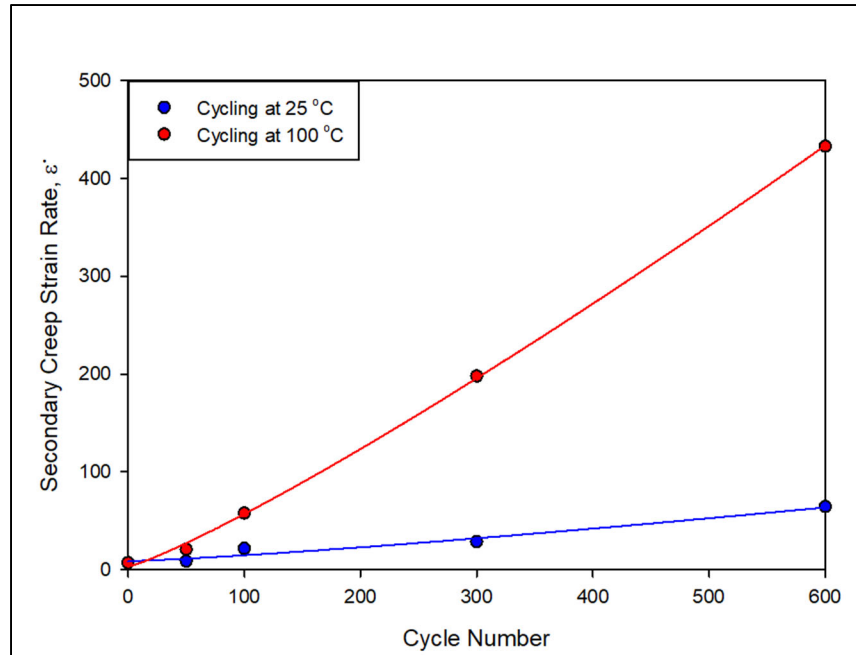


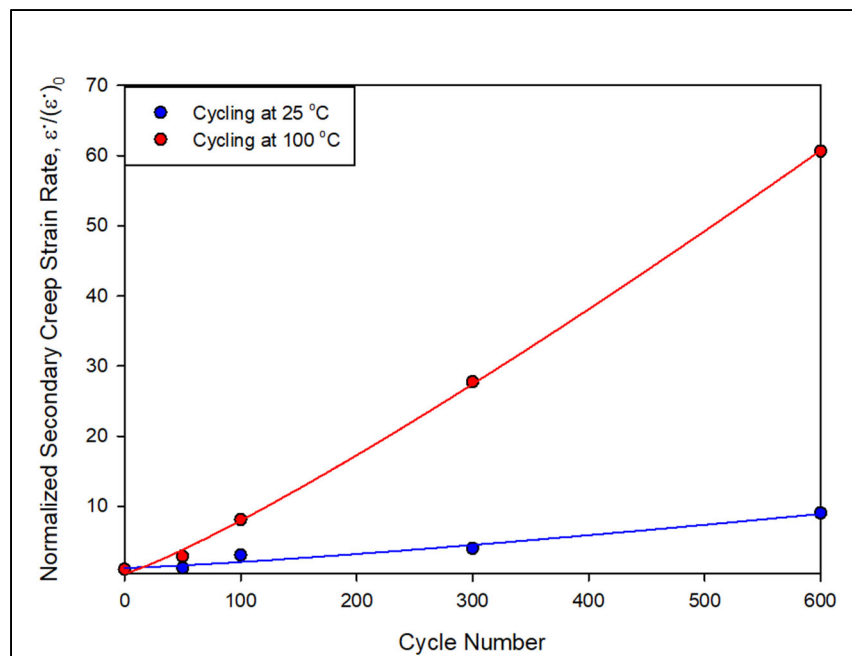
Figure 5.11 Evolution of creep curves of SAC305 cycled at $T = 100\text{ }^{\circ}\text{C}$

Table 5.5 Evolution of Creep Properties with Isothermal Mechanical Cycling (SAC305)

Number of Cycles	Secondary Creep Strain Rate ($\times 10^{-8}\text{ sec}^{-1}$)	
	SAC305 cycled at 25 °C	SAC305 cycled at 100 °C
0	7.14	7.14
50	8.61	20.6
100	21.6	57.8
300	28.5	198
600	64.5	433



a) Reduction of Secondary Creep Strain Rate with Cycling



b) Reduction of Normalized Secondary Creep Strain Rate with Cycling

Figure 5.12 Reduction of Creep Properties of SAC305 with Mechanical Cycling

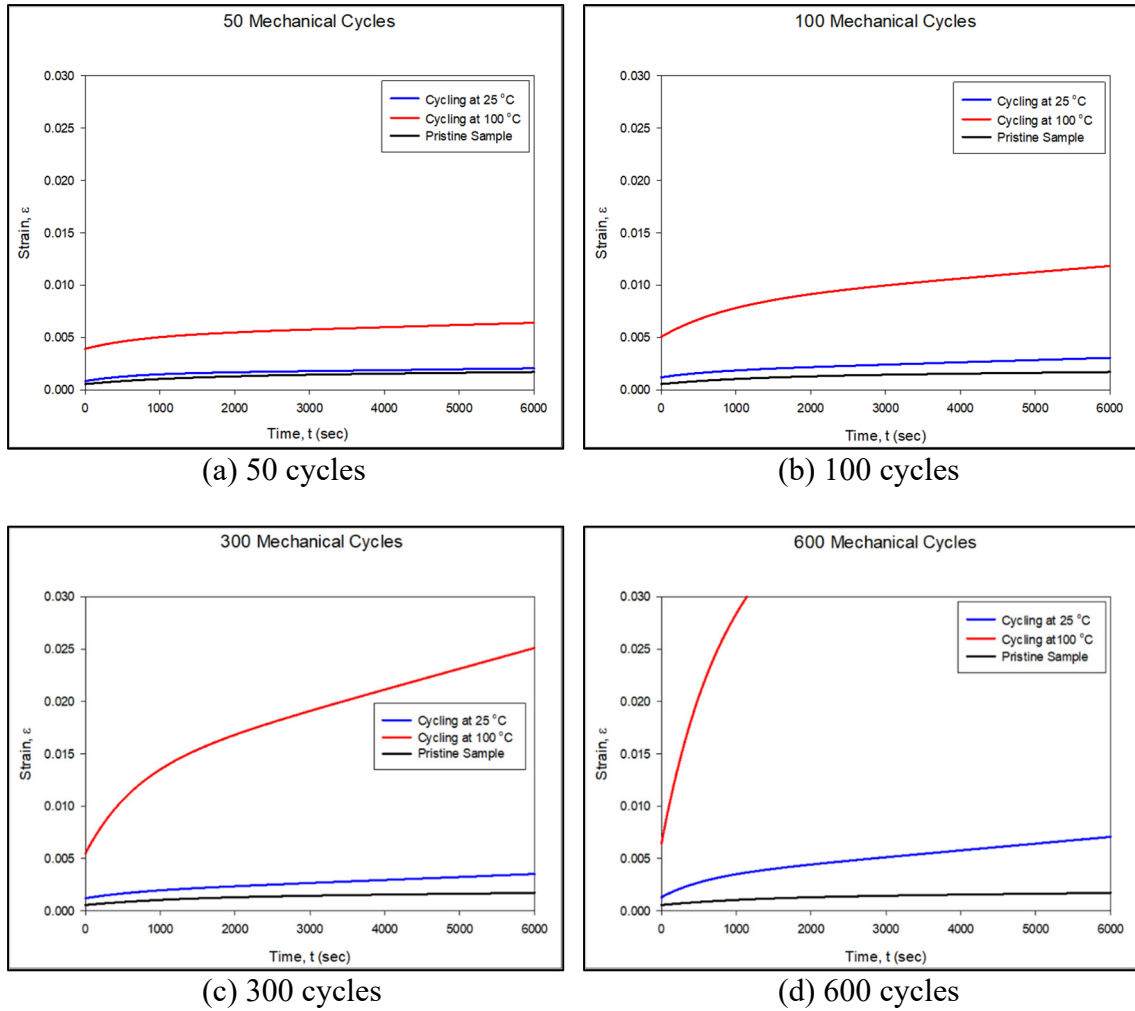


Figure 5.13 Creep Curve Evolution of SAC305 with Number of cycles

The creep curves of 100 °C pre-cycled SAC_Q specimens have been presented in Fig 5.14. The initial creep strain for SAC_Q after 50 mechanical cycles at 100 °C was also obtained to be nearly 7 times the initial creep strain of pristine samples, as observed with SAC305. The secondary creep strain rate for 100 °C pre-cycled samples for both alloys have been presented in Table 5.6. The secondary creep strain rate for SAC_Q also rose sharply with an increase in duration of pre-cycles. After 300 mechanical cycles at 100 °C, the secondary creep rate of SAC305 samples increased by about 27X (2700%), while in SAC_Q samples, it increased by about 23X (2300%). These values were significantly

higher than the creep rate values for the same duration of cycling when these alloys were cycled at room temperature. SAC305 showed a much harsher decrease in properties to mechanical cycling when compared with SAC_Q, especially when the samples were cycled for longer durations. This also illustrates the stiffness introduced in the lead free alloys when doped with bismuth. At elevated temperatures, this stiffness is reduced in SAC_Q as seen in the numeric results of the secondary creep rate. The evolution of the secondary creep strain rate of both alloys pre-cycled at 100 °C is presented in Figure 5.15.

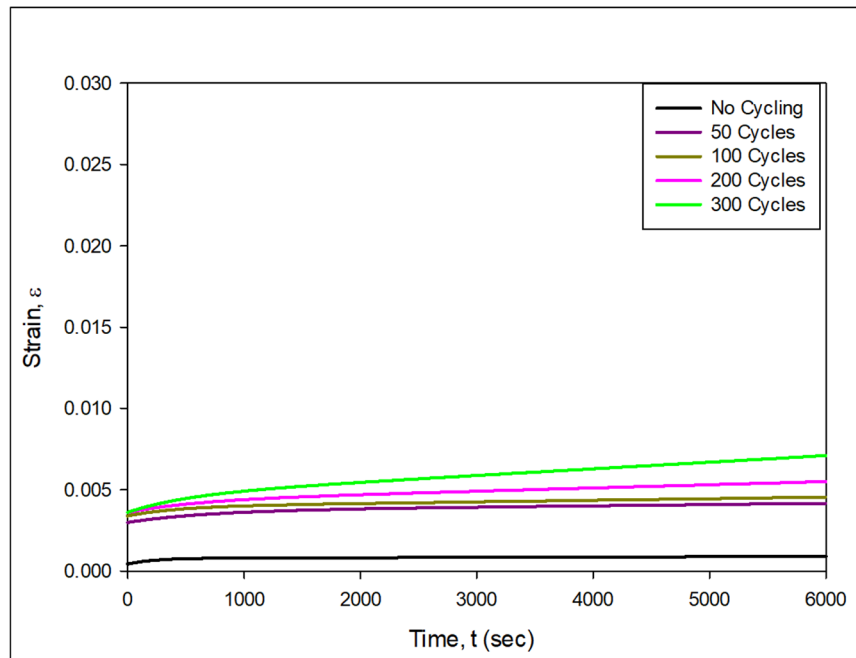
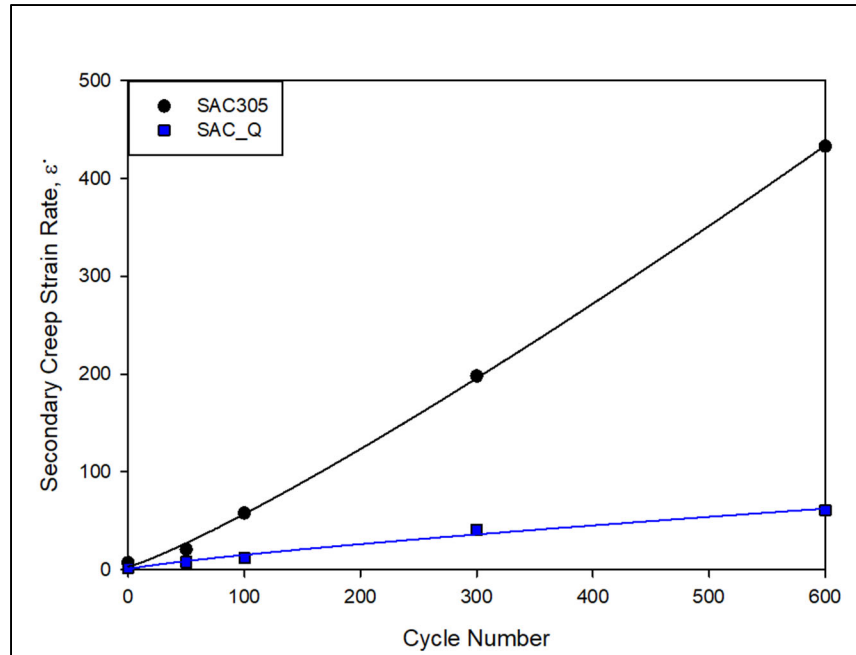


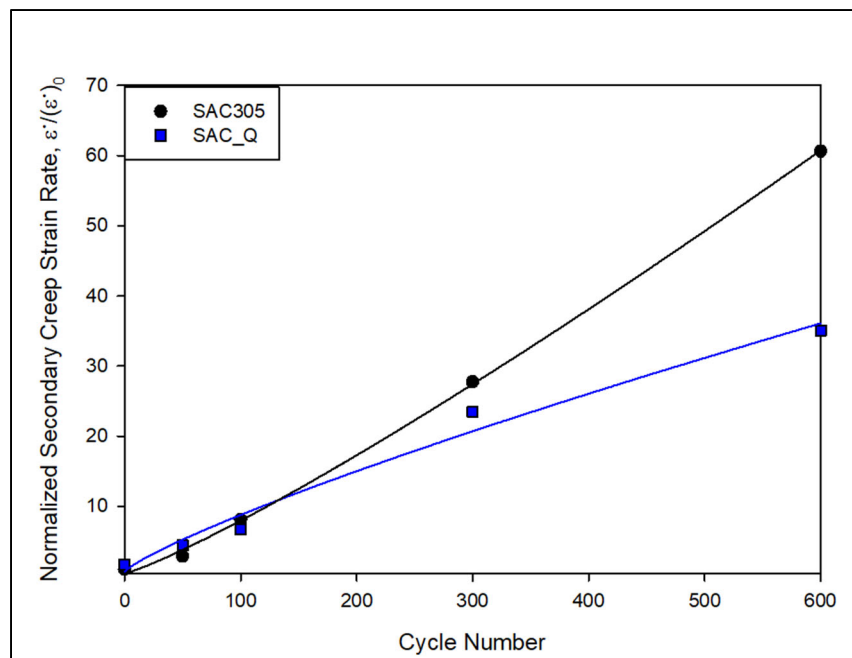
Figure 5.14 Evolution of creep curves of SAC_Q cycled at T = 100 °C

Table 5.6 Evolution of Creep Properties with Isothermal Mechanical Cycling at 100 °C

Number of Cycles	Secondary Creep Strain Rate ($\times 10^{-8} \text{ sec}^{-1}$)	
	SAC305 cycled at 100 °C	SAC_Q cycled at 100 °C
0	7.14	1.73
50	20.6	7.73
100	57.8	11.58
200	154	19.70
300	198	40.66



a) Reduction of Secondary Creep Strain Rate with Cycling



b) Reduction of Normalized Secondary Creep Strain Rate with Cycling

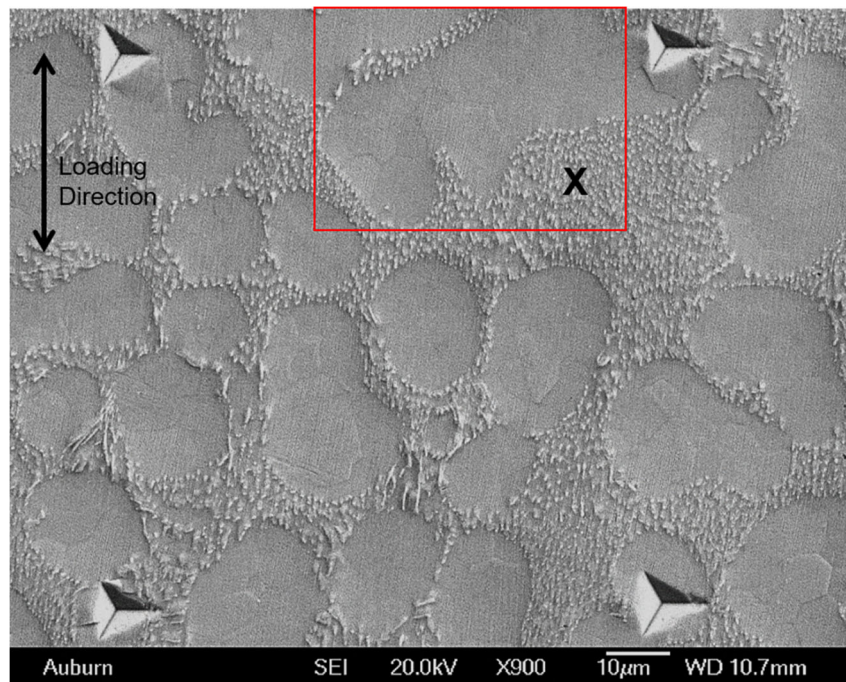
Figure 5.15 Reduction of Creep Properties with Mechanical Cycling at 100 °C

5.5 Evolution of Microstructure with Elevated Temperature Mechanical Cycling

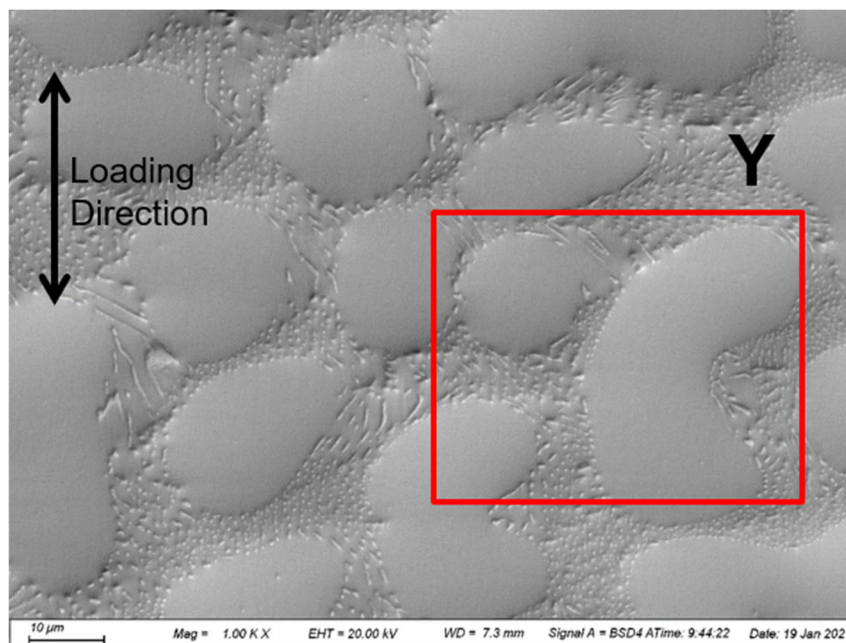
In this section, the observed changes in the microstructure due to room and elevated temperature mechanical cycling will be reviewed. Polished uniaxial rectangular cross-sectioned samples were prepared as discussed earlier. The samples were cycled at a strain range of $\epsilon = \pm 0.01$ at a temperature of 100 °C for durations of 0, 25, 50, 75, and 100 cycles. Further cycles up to failure could not be studied due to the development of a non-flat topography introduced from the mechanical cycling. For this study, we have focused on the changes in the surface microstructure of the alloys. This was due to the fact that the early stages of fatigue typically originate from the surface [132]. The development of the slip lines and plastic deformation near the surface aids in the nucleation of cracks from these regions. Typical non-cycled microstructures of SAC305 are presented in Fig 5.16. The initial microstructures consisted of various sizes of β -Sn dendrites surrounded by eutectic regions containing intermetallics of Cu_6Sn_5 and Ag_3Sn .

Regions X and Y on the micrographs in Fig. 5.16, were tracked in detail and will be discussed here and have been presented in Figs. 5.17 and 5.18, respectively. Small microcracks began to form in the SAC305 specimens just after 25 cycles for both the cases. These microcracks formed along the sub-grain boundaries of the β -Sn dendrites for samples cycled at 25 °C. However, the microcracks formed along the interface of the β -Sn dendrites and the eutectic regions for the samples cycled at 100 °C. Slip bands were observed just after this short period of cycling at $T = 100$ °C, suggesting the weakening of the sample during elevated temperature cycling. This wasn't the case with room temperature cycling of SAC305, where the slip lines were observed a lot later in the cyclic progression. This suggests that the plastic deformation occurs more readily at elevated

temperatures. The eutectic region, however, was unaffected and no cracks nucleated from the initial phases of the cycling from these regions for both cycling temperatures.



(a)



(b)

Figure 5.16 Typical non-cycled SAC305 microstructures

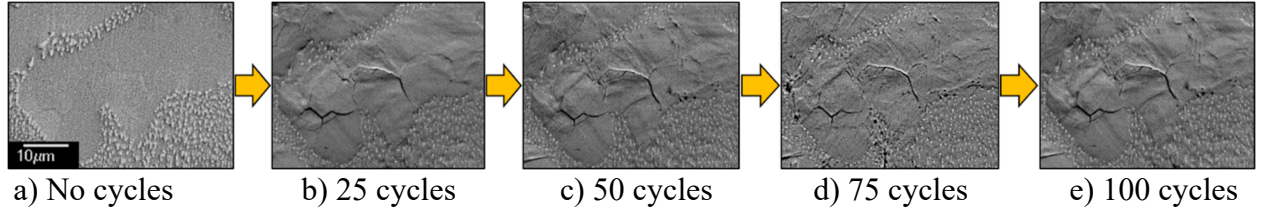


Figure 5.17 Evolution of SAC305 Microstructure with Mechanical Cycling at $T = 25\text{ }^{\circ}\text{C}$ (Region X)

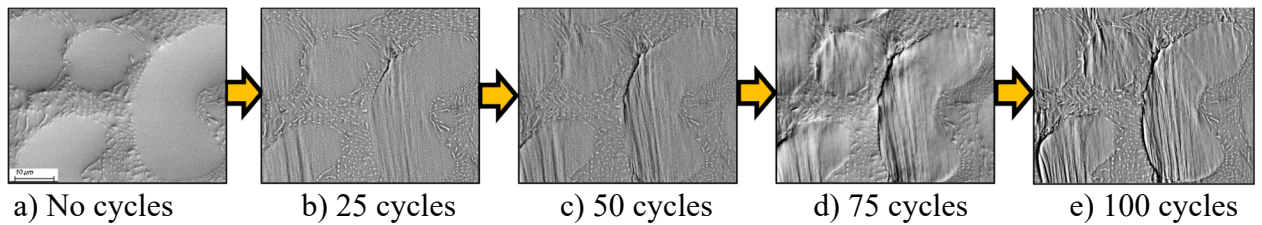


Figure 5.18 Evolution of SAC305 Microstructure with Mechanical Cycling at $T = 100\text{ }^{\circ}\text{C}$ (Region Y)

As the duration of cycling increased, the developed cracks grew in length. The cracks propagated along the sub-grain boundaries for samples cycled at room temperature, while the cracks propagated in the direction of the slip lines for samples cycled at elevated temperature. The slip lines also started to become more prominent during this period for the samples cycled at elevated temperature. The intrusions and extrusions associated with these slip lines became much significant with higher cycling numbers. By closely observing the series of micrographs in Figs. 5.17 and 5.18, one can deduce that the bulk of the damage accumulated due to elevated temperature cycling is in the form of these slip bands and the remaining fraction is in the form of the microcracks. For the room temperature cycling, most of the damage in the initial stages was seen in the form of microcracks and its progression. These microcracks also seemed to propagate in a direction along the slip lines. We can thus conclude that the formation of the slip bands

with microcracks indicates a higher level of damage in the sample and this subsequently reduces the associated mechanical behavior of SAC305 more harshly than with room temperature cycling. No cracks were seen to nucleate in the hard eutectic phases for both temperatures of cycling; rather cracks were observed in these phases only during the crack propagation stage. This suggests that weakening of the β -Sn dendrites is the primary reason for the crack initiation.

A typical non-cycled microstructure of SAC_Q is presented in Fig 5.19. The initial microstructure of SAC_Q looks similar to that of SAC305, with eutectic regions containing intermetallics of Cu_6Sn_5 and Ag_3Sn encapsulating various sizes of β -Sn dendrites. White Bismuth rich regions are additionally visible in different regions of the SAC_Q microstructure (shown in Fig. 5.19). Bismuth helps to enhance the strength of the Tin matrix by Solid Solution Strengthening (SSS) phenomenon. Region Z on the micrographs of Fig 5.18 was tracked in detail after 0, 25, 50, 75, and 100 cycles and will be discussed here.

Small microcracks began to form along the interface of the β -Sn dendrites and the eutectic region just after 25 cycles for the SAC305 samples. Slip bands were observed just after this short period of cycling suggesting the weakening of the sample at elevated temperatures. This wasn't the case with room temperature cycling of SAC305, where the slip lines were observed lot later in the cyclic progression. This suggests that the plastic deformation occurs more readily at elevated temperatures. The eutectic region, however, was unaffected and no cracks nucleated from the initial phases of the cycling from these regions as was similarly observed during room temperature cycling. As the duration of cycling increased, these cracks grew in length. The slip lines also started to become more

prominent during this period. The intrusions and extrusions associated with these slip lines became much significant with higher number of cycles.

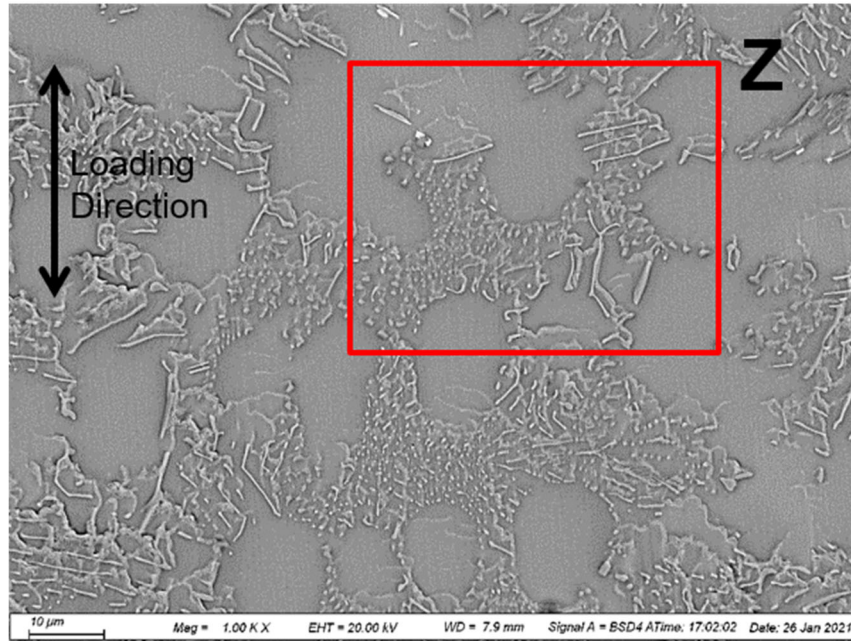


Figure 5.19 Typical non-cycled SAC_Q microstructures

The changes in the region Z of the SAC_Q microstructure with mechanical cycling at 100 °C has been presented in Fig. 5.20. As with the SAC305 specimens cycled at elevated temperature, bulk of the damage accumulated due to elevated temperature cycling is in the form of slip bands and the remaining fraction is in the form of the microcracks. Unlike the SAC305 samples, slip lines began appearing after the 50th cycle and microcracks were observed after the 75th cycle. The severity of the developed slip lines was lot less than that observed after just 10 cycles in SAC305. The microcracks were also smaller in size and quantity than that observed in the SAC305 samples. Additionally, it was observed that the SAC_Q samples exhibited a trend similar to that observed when SAC_Q specimens were subjected to room temperature. The dissolved bismuth in the SAC alloy, thus, has a

similar effect on the microstructure even at elevated temperatures. Unlike that observed in aging conditions [134], Bi does not diffuse into the tin solution when the SAC_Q samples were subjected to elevated temperature mechanical cycling. For both alloys, the microcracks then propagate in a direction along these slip bands. This formation and propagation can be attributed to the decrease in the mechanical properties observed after cycling at 100 °C. No cracks were seen to nucleate in the hard eutectic phases for both alloys, rather cracks were observed in these phases only during the crack propagation stage. This suggests that weakening of the β -Sn dendrites is the primary reason for the crack initiation.

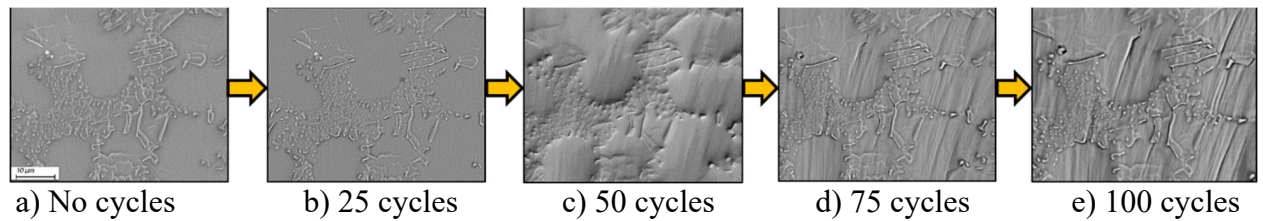


Figure 5.20 Evolution of SAC_Q Microstructure with Mechanical Cycling at T = 100 °C (Region Z)

5.6 Summary and Discussion

In this chapter, the effects of elevated temperature mechanical cycling on the damage accumulation in lead free alloys were investigated. SAC305 and SAC_Q uniaxial cylindrical samples were prepared and subjected to constant strain cyclic tests at a test temperature of 100 °C. The hysteresis loops at various intervals were recorded and the different cyclic properties (peak stress, plastic strain range, and plastic strain energy density) were extracted from these hysteresis loops. This allowed us to evaluate the

reduction of the peak stress and hysteresis loop area with cycling. The SAC305 samples cycled at room temperature demonstrated a higher peak stress and loop area for the same strain range, and these values reduced less than that observed for SAC305 samples cycled at 100 °C. This suggests larger weakening of the alloy during elevated temperature cycling. When comparing both the alloys, the SAC+Bi alloy demonstrated a higher peak stress and loop area for the same strain range and these values decreased less than that observed in SAC305. This suggests the better cyclic performance of Bi doped alloys at elevated temperatures.

To gain a better knowledge of the damage accumulation accompanying the elevated temperature mechanical cycling, pristine SAC305 and SAC_Q samples were mechanically cycled for durations of 0, 50, 100, 300 and 600 cycles at temperatures of $T = 100\text{ }^{\circ}\text{C}$. These prior cycled specimens were then either subjected to room temperature tensile or creep tests to quantify the damage accumulated in the samples due to the cyclic tests. The SAC305 samples cycled at 100 °C proved to have a large damage accumulation with the same number of cycles as deduced from the results when compared to the room temperature cycled SAC305 samples, while SAC_Q exhibited a superior performance in the tensile and creep properties when cycled for the same durations at 100 °C. However, the SAC_Q samples could not be cycled up to 600 cycles at 100 °C due to the larger plastic work dissipated per cycle.

An SEM microstructural study was conducted alongside to view the changes in the microstructure and correlate them to the changes that occur with cycling. Polished rectangular cross-sectioned samples were indented to mark regions of interest which were tracked after each cycling duration. Slip bands and microcracks were evident in the

SAC305 samples cycled at 100 °C after only 25 cycles. These features were observed in the SAC_Q samples after the 75th cycle. The microcracks, for SAC305 (100 °C cycling) were observed to occur on the interface of the eutectic and β -Sn regions while they occurred on the sub-grain boundaries of the β -Sn dendrites for the SAC305 (25 °C cycling) and SAC_Q (100 °C cycling), with slip lines being visible towards the latter cycles. With increased durations of cycling, the slip lines became more visible while the microcracks grew in length and the slip line damage was the major visual damage accumulation mechanism for both the alloys cycled at 100 °C. The cracks then propagated in a direction along the slip lines as cycling progressed. No cracks were observed to nucleate in the eutectic phases and cracks were seen there only during the propagation stage.

CHAPTER 6

EFFECT OF STRAIN RANGE ON THE CYCLIC AND CONSTITUTIVE PROPERTIES OF LEAD FREE SOLDER ALLOYS

6.1 Introduction

In chapter 4, the accumulation of damage in several solder materials (SAC305, SAC+Bi, and SAC+Bi-Ni-Sb) during mechanical cycling at room temperature was investigated. Circular cross-sectioned solder specimens were first reflowed, and these samples were then mechanically cycled for various durations using a Micro-Mechanical tester. Cyclic stress-strain, monotonic stress-strain, and creep tests were then conducted on the prior cycled samples. The cyclic stress-strain curves obtained were studied to observe the change of hysteresis loop properties (peak stress, hysteresis loop area, and plastic strain range) with mechanical cycling. The monotonic stress-strain and creep test data were plotted, and several mechanical properties were characterized for various levels of cycling. Using the data from these tests, we have been able to characterize and quantify the cycling induced damage through the observed reductions of several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) with the amount of prior cycling. All of the mechanical cyclic testing in our prior work and the work of others has been performed at room temperature ($T = 25\text{ }^{\circ}\text{C}$) and were conducted on a single strain range, and thus could not be easily extended to thermal cycling applications, where the maximum strain experienced by the solder joint would depend on the cyclic temperature range and the different materials used in the electronic package.

In this chapter, we have extended our prior study to examine mechanical cycling at different levels of total strain range. The three solder alloys previously tested were now cycled up to the 20th cycle for various magnitudes of total strain range. Curves of initial plastic work accumulated vs total strain range was then achieved for all the three alloys. This would allow one to estimate the initial plastic work accumulated for any particular total strain range for SAC305, SAC_Q or Innolot.

The following portion of this chapter then goes to investigate the damage accumulation of SAC305 samples cycled at 4 different levels of initial plastic work accumulated ($\Delta W \approx 0.25, 0.5, 0.75, 1 \text{ MJ/m}^3$). Using the chart obtained, SAC305 samples were cycled at these initial plastic work dissipated up to 600 cycles. The hysteresis loops obtained were then studied and compared. Uniaxial tensile tests at room temperature were also conducted on specimens that had been previously mechanically cycled for various durations (e.g. 0, 50, 100, 200, 300, 600 cycles) at room temperature for the 4 different strain ranges. This allowed us to study the evolution of the constitutive behavior of the solder alloys that occurred during the room temperature mechanical cycling due to the fatigue damage that builds up in the specimens. In particular, the changes in the elastic modulus, yield strength, and ultimate strength, were evaluated for the different strain ranges. These experiments were then repeated for SAC305 samples cycled at an elevated temperature of 100 °C.

These changes in the properties that occur during mechanical cycling would then allow us to estimate and quantify the damage accumulated due to mechanical cycling at any particular strain range. These properties could then be incorporated into a constitutive

model (such as Anand Model) that could then provide a method which would allow the modelling of the damage accumulation within SAC305 lead free alloys.

6.2 Evolution of Cyclic Stress-Strain Curves

In this section, the evolution of the cyclic stress-strain curves with strain range will be presented. Uniaxial SAC305, SAC_Q, and Innolot samples were subjected to mechanical cycling with strain ranges of $\varepsilon = \pm 0.0005, \pm 0.0015, \pm 0.0025, \pm 0.0035, \pm 0.0045, \pm 0.006$, and ± 0.01 . The samples were tested at room temperature and a strain rate of $\dot{\varepsilon} = 0.002 \text{ s}^{-1}$. The initial hysteresis cyclic curves obtained have been presented in Figs. 6.1 – 6.3. As computed in the previous sections, each of the hysteresis curves were fit from the raw data using eqns. 3.5 and 3.6. At least 5 samples were tested for each condition in all the three alloys. A common trend was observed for the cycling of all three alloys. For the smallest strain range (Total Strain Range, $\varepsilon = 0.001$), plastic deformation was little. With an increase in applied total strain range, the plastic deformation and the plastic strain range (distance between the two strain axes intercepts) increased in magnitude as is evident from the curves. The peak stress also increased as the strain range increased but reaches a saturated value when the largest strain range (Total Strain Range, $\varepsilon = 0.02$) is applied. The maximum stress at each strain range for each alloy was then fit using the four parameter hyperbolic tangent model (eqn. 3.2) to obtain the red colored cyclic stress-strain curve shown in Figs. 6.1 – 6.3. These cyclic stress-strain curves (presented in Figure 6.4) were found to be lower than their corresponding monotonic tensile curve counterpart and have been. This phenomenon occurs if the material cyclically softens [132]. As observed in Chapter 4, all the three alloys cyclically soften when cycled at a constant strain range.

Thus, the cyclic stress-strain curve obtained agrees with the stated theory. Fig 6.5 compares the evolution of the hysteresis curve with strain range among the three alloys.

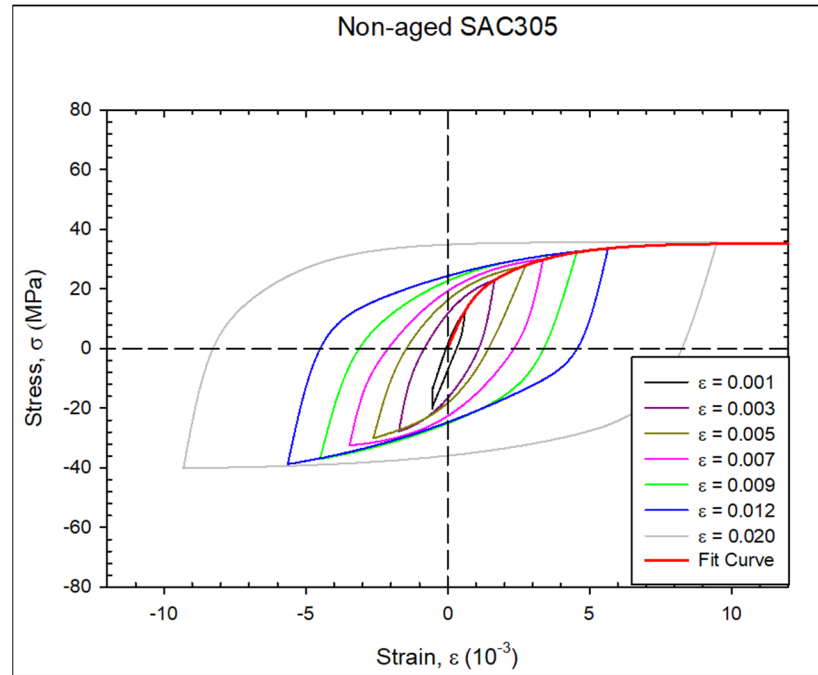


Figure 6.1 Evolution of hysteresis loop with strain range (SAC305 cycled at $T = 25\text{ }^{\circ}\text{C}$)

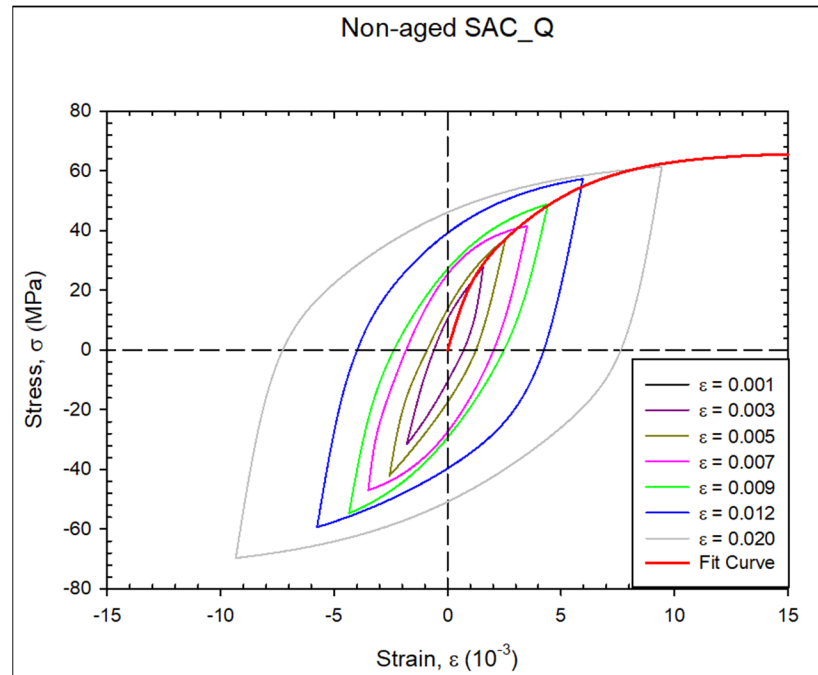


Figure 6.2 Evolution of hysteresis loop with strain range (SAC_Q cycled at $T = 25\text{ }^{\circ}\text{C}$)

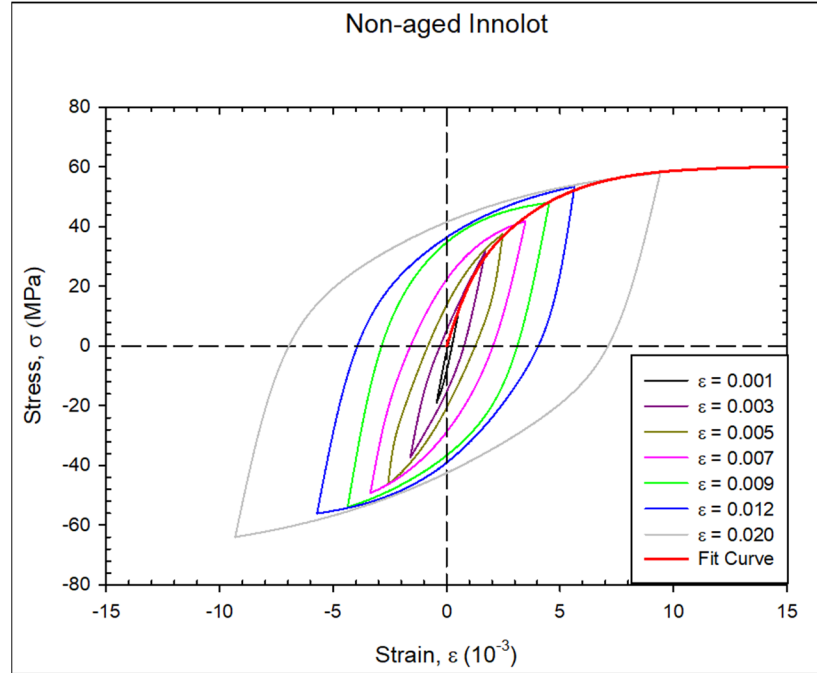


Figure 6.3 Evolution of hysteresis loop with strain range (Innolot cycled at $T = 25\text{ }^{\circ}\text{C}$)

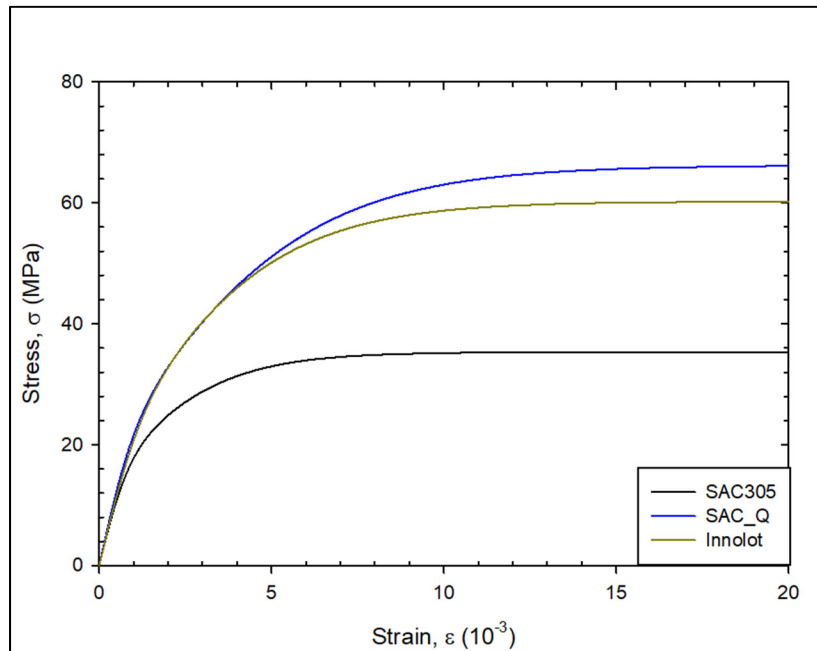
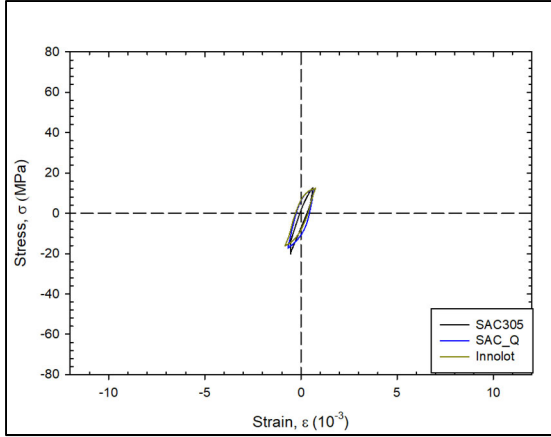
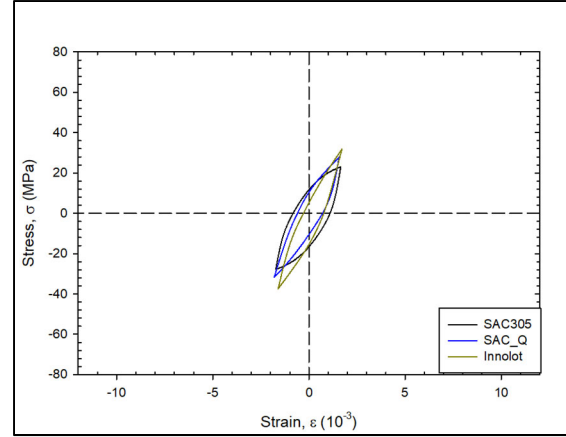


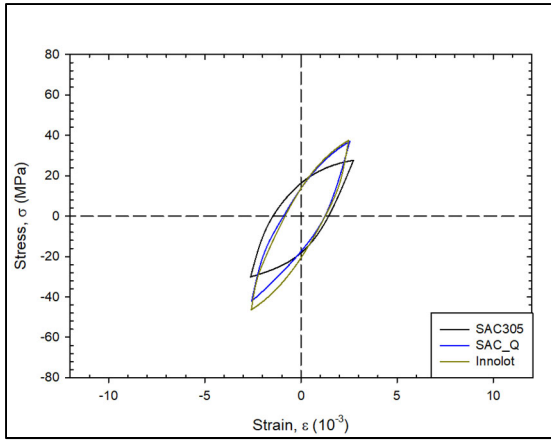
Figure 6.4 Fit Cyclic Stress-Strain Curve Obtained for all three alloys



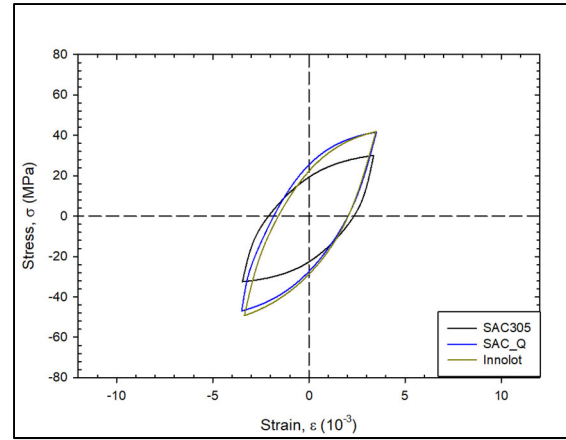
(a) Total Strain Range, $\varepsilon = 0.001$



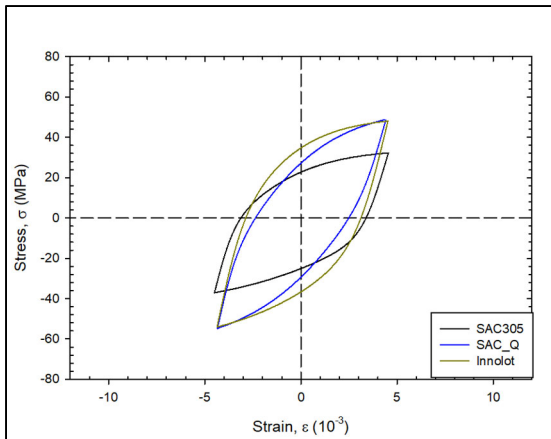
(b) Total Strain Range, $\varepsilon = 0.003$



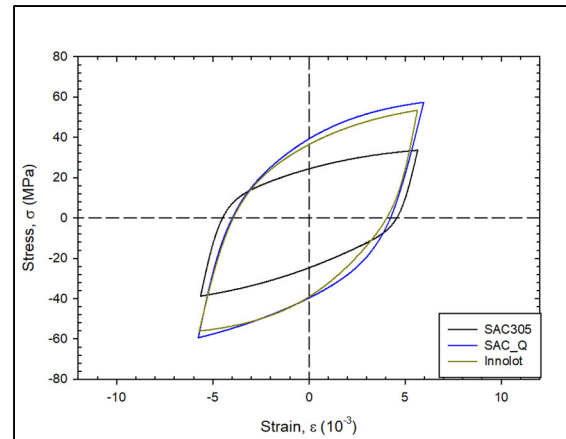
(c) Total Strain Range, $\varepsilon = 0.005$



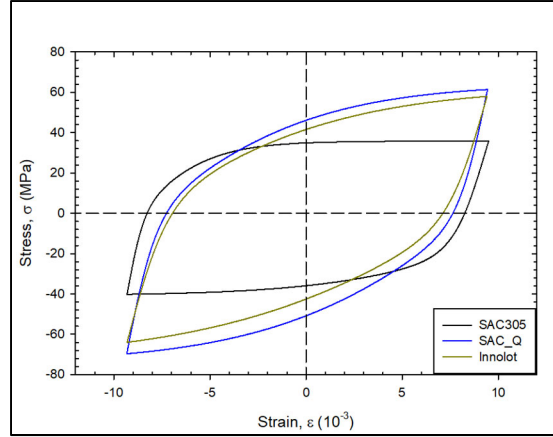
(d) Total Strain Range, $\varepsilon = 0.007$



(e) Total Strain Range, $\varepsilon = 0.009$



(f) Total Strain Range, $\varepsilon = 0.012$



(g) Total Strain Range, $\varepsilon = 0.020$

Figure 6.5 Initial Hysteresis Curves Obtained at Different Strain Ranges

Eqn. 3.7 was then used to evaluate the hysteresis loop area for each curve. Plots for initial plastic work dissipated vs total applied strain range were then generated for all three alloys (Fig 6.6). These plots would thus estimate the strain range needed to cycle an alloy at a strain rate of $\dot{\varepsilon} = 0.002 \text{ s}^{-1}$ to obtain a certain value of initial plastic work accumulated.

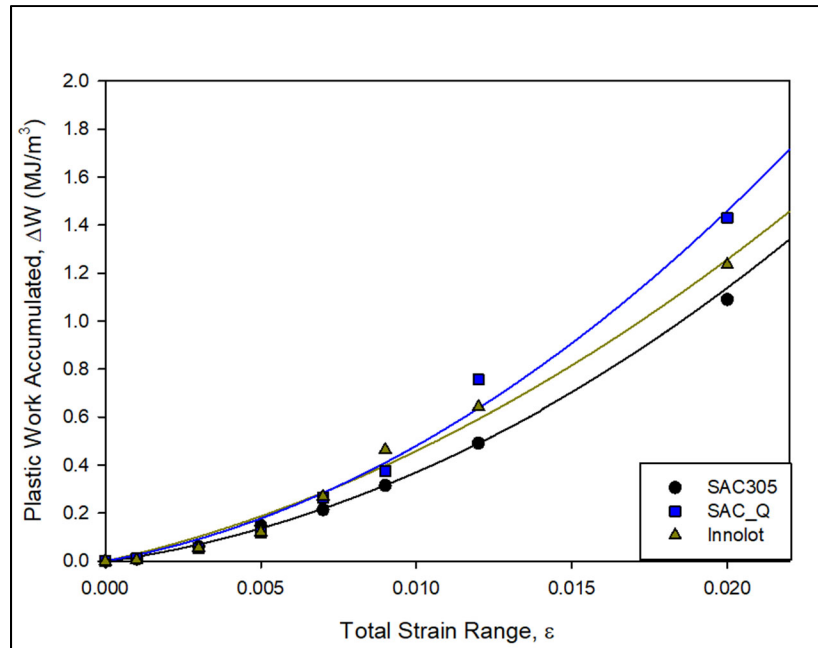


Figure 6.6 Plots of Plastic Work Accumulated vs Total Strain Range

6.3 Evolution of Cyclic Behavior of SAC305 with Mechanical Cycling at 4 Different Initial Plastic Work Magnitudes

From this section onwards, the objective will be to determine the evolution in various properties about by mechanical cycling. In contrast to the results presented in Chapter 4, the changes quantified in the following sections will focus on particular values of initial plastic work accumulated ($\Delta W \approx 0.25, 0.5, 0.75, 1 \text{ MJ/m}^3$). Cylindrical uniaxial samples of SAC305 were then cycled at specific strain ranges (obtained from plots such as in Fig 6.6) such that the aforementioned initial plastic work accumulated values are achieved. Results from cycling at both room temperature and elevated temperature will be presented. Future studies (not covered in this dissertation) would be to compare the changes in cyclic and constitutive properties with other doped lead free alloys if they are cycled at the same initial plastic work dissipated, rather than cycling at the same strain range or the same ratio of fatigue duration of cycling (as covered in Chapter 4 of this dissertation).

6.3.1 Room Temperature (25 °C) Cycling

Figure 6.7 displays the plot of initial plastic work accumulated vs the applied total strain range for SAC305 samples cycled at room temperature and a strain rate of $\dot{\epsilon} = 0.002 \text{ s}^{-1}$. Dashed lines shown in Figure 6.7 indicate the applied strain ranges picked such that the initial plastic work dissipated within the sample, ΔW , had values of approximately 0.25, 0.5, 0.75, 1 MJ/m^3 . Once these approximate strain ranges were obtained, cylindrical SAC305 samples were subjected to mechanical cycling of these particular strain range values. The constant strain mechanical cycling was conducted for up to 600 cycles at room

temperature. The raw data was then fit with eqns. 3.5 and 3.6 to obtain fit curves for particular durations of cycle. The evolution of the hysteresis loops with mechanical cycling for the ΔW values has been presented in Figure 6.8. From these curves, we see that as the applied strain range is increased, a higher peak stress is obtained. Regardless of the applied strain range, the evolution of the hysteresis loops exhibited a similar trend. A cyclic softening phenomenon is observed throughout the cycling progress with the peak stress reducing consistently with an increased duration of cycles. However, it was seen that for $\Delta W \approx 0.25$ and 0.5 , the largest decrease in peak stress (and consequently, the loop area) occurred during the latter stages of the cycling, while for $\Delta W \approx 0.75$ and 1 , the largest decrease in peak stress was observed during the initial stages of the mechanical cycling. The plastic strain range was seen to be fairly constant for all the conditions. Figure 6.9 consists of a set of curves which show the evolution in the hysteresis loop with number of cycles for all four strain ranges. All the cyclic data have been compiled in Tables 6.1 – 6.3 and the evolution of these properties have been displayed in Fig 6.10.

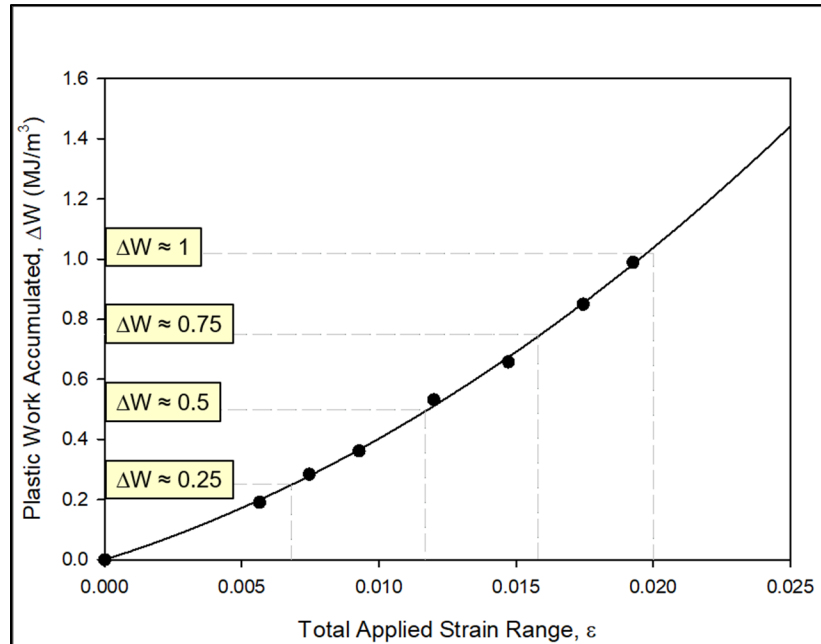
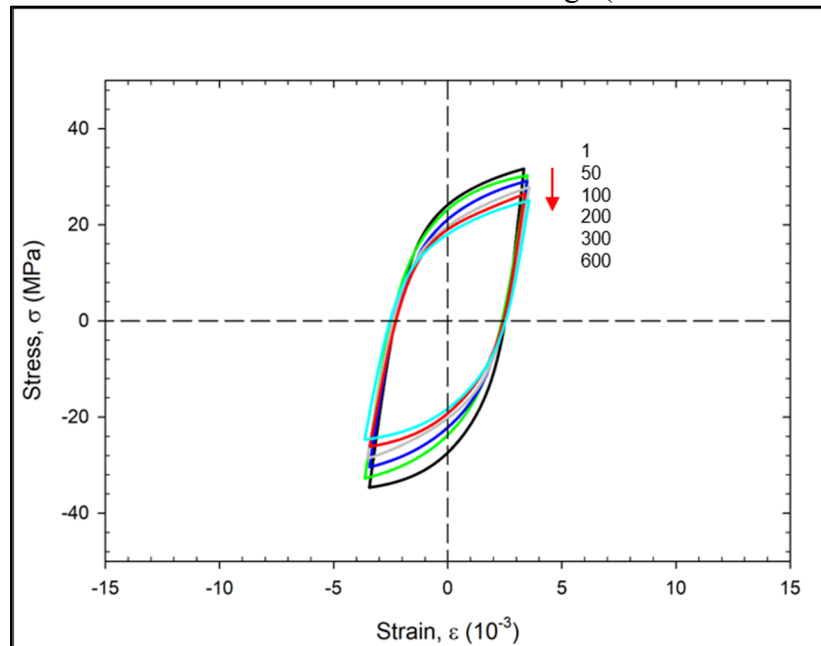
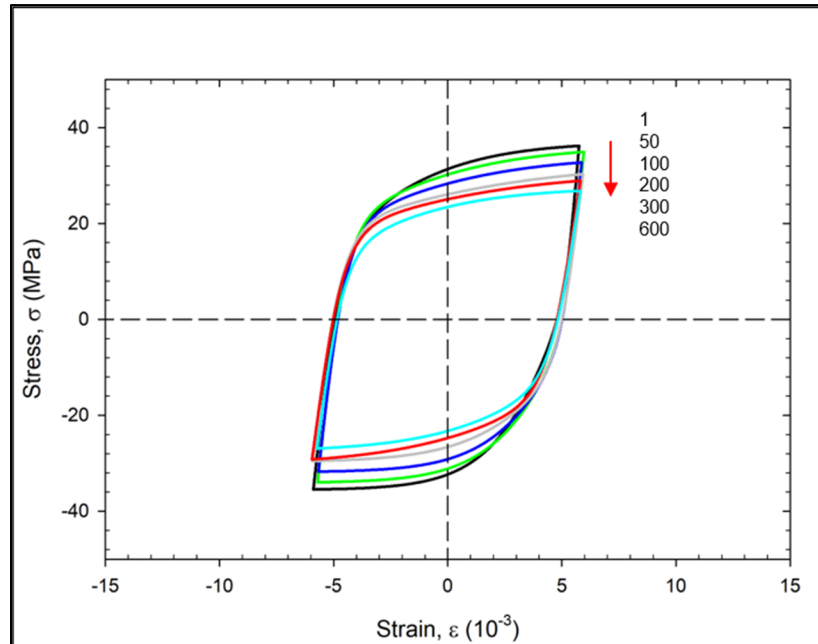


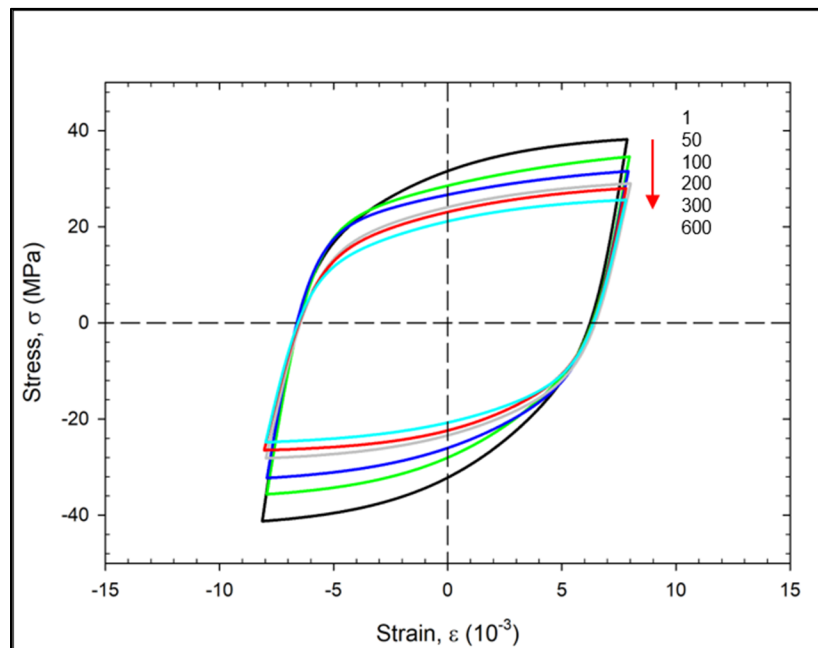
Figure 6.7 Initial Plastic Work vs Total Strain Range (SAC305 25 °C Cycling)



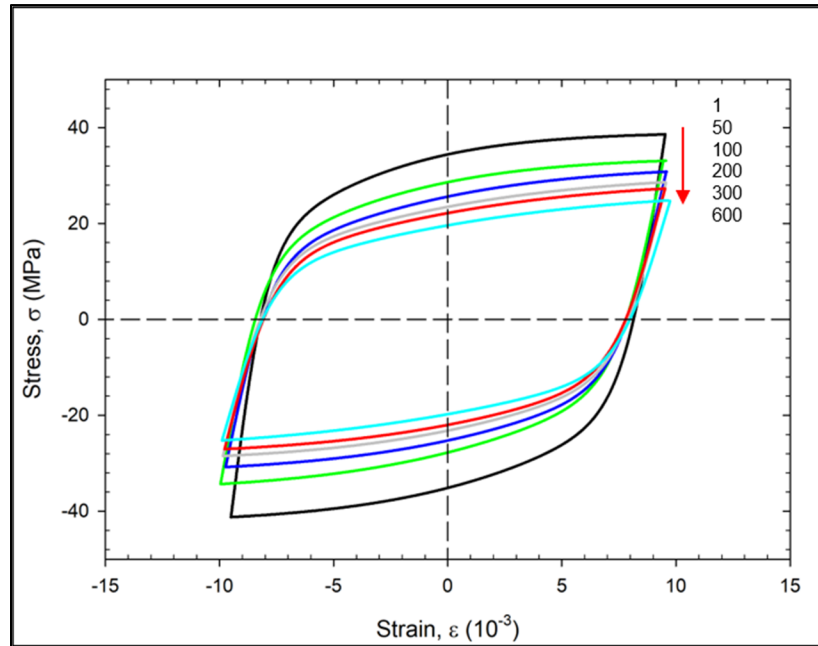
a) $\Delta W \approx 0.25 \text{ MJ/m}^3$



b) $\Delta W \approx 0.5 \text{ MJ/m}^3$

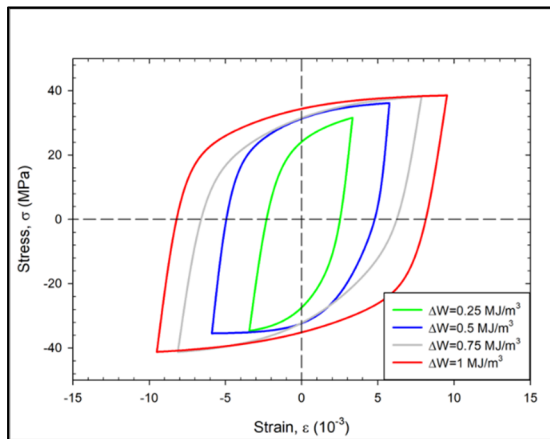


c) $\Delta W \approx 0.75 \text{ MJ/m}^3$

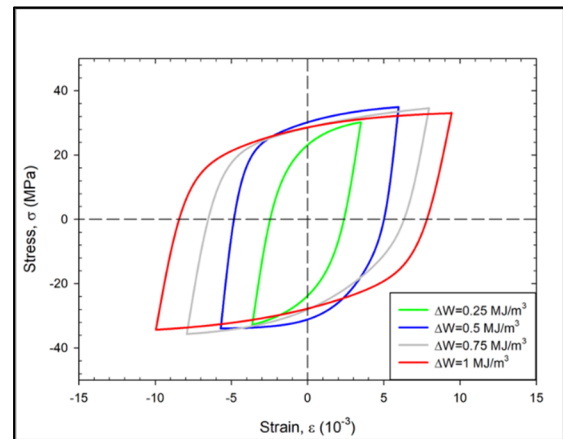


a) $\Delta W \approx 1 \text{ MJ/m}^3$

Figure 6.8 Evolution of Hysteresis Loop with Mechanical Cycling
(SAC305 cycled at 25 °C)



(a) 1 cycle



(b) 50 cycles

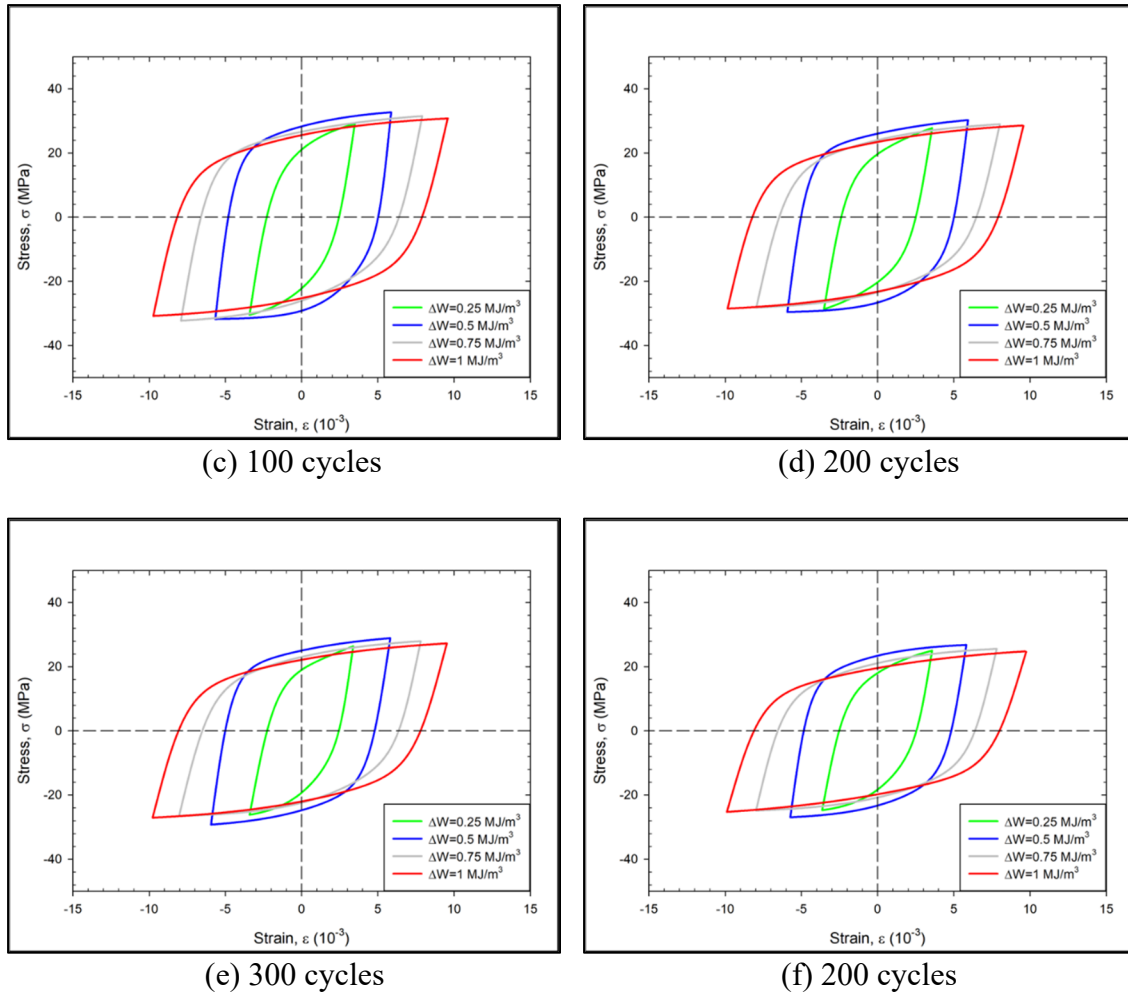


Figure 6.9 Hysteresis Loop Evolution of SAC305 with Number of cycles
(Cycled at 25 °C)

Table 6.1 Evolution of Hysteresis Loop Area with Mechanical Cycling at 25 °C
(SAC305)

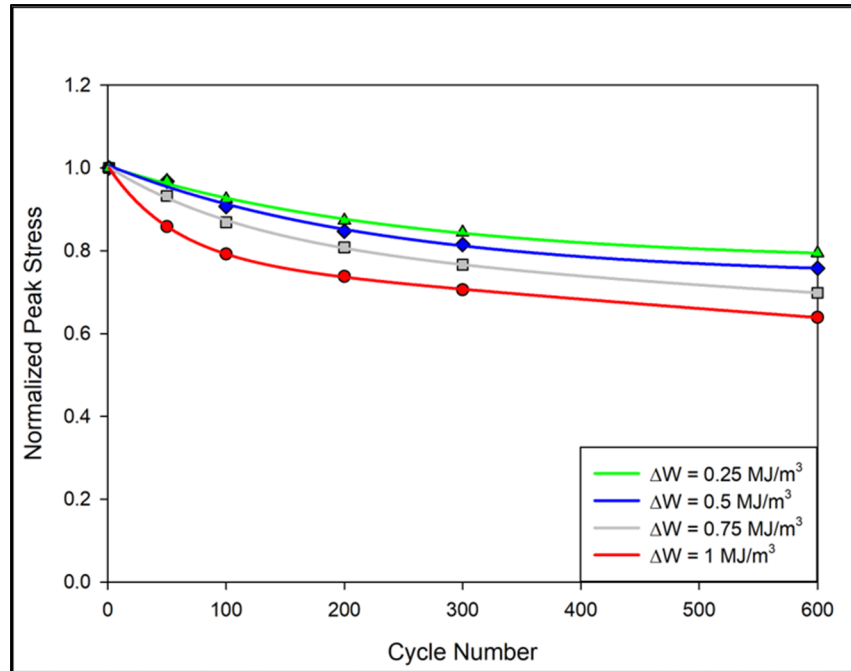
Number of Cycles	Hysteresis Loop Area, ΔW (MJ/m ³)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	0.253	0.563	0.797	1.161
50	0.238	0.527	0.711	0.883
100	0.228	0.498	0.669	0.804
200	0.216	0.462	0.622	0.743
300	0.209	0.443	0.588	0.709
600	0.195	0.407	0.521	0.617

Table 6.2 Evolution of Peak Stress with Mechanical Cycling at 25 °C (SAC305)

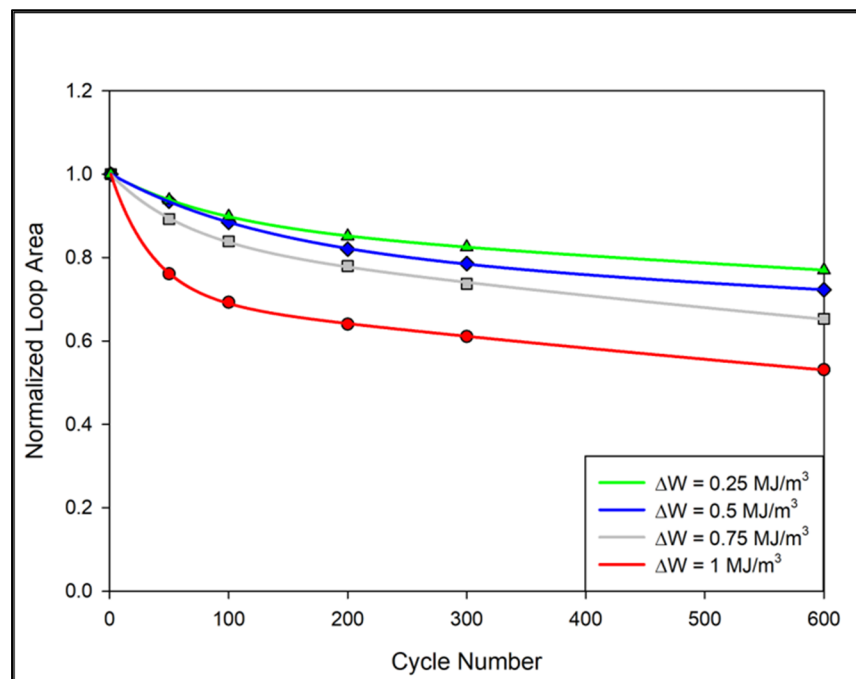
Number of Cycles	Peak Stress, $\sigma_{\max}$ (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	31.63	36.20	38.16	38.65
50	30.24	34.94	34.59	33.18
100	29.10	32.74	31.56	30.62
200	27.82	30.31	29.00	28.52
300	26.44	28.92	27.99	27.28
600	25.04	26.82	25.60	24.71

Table 6.3 Evolution of Plastic Strain Range with Mechanical Cycling at 25 °C (SAC305)

Number of Cycles	Plastic Strain Range, ε_p (%)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	0.49	0.95	1.32	1.65
50	0.49	0.95	1.31	1.63
100	0.49	0.95	1.32	1.61
200	0.50	0.94	1.31	1.62
300	0.50	0.94	1.32	1.59
600	0.50	0.94	1.31	1.59



a) Reduction of Normalized Peak Stress with Cycling



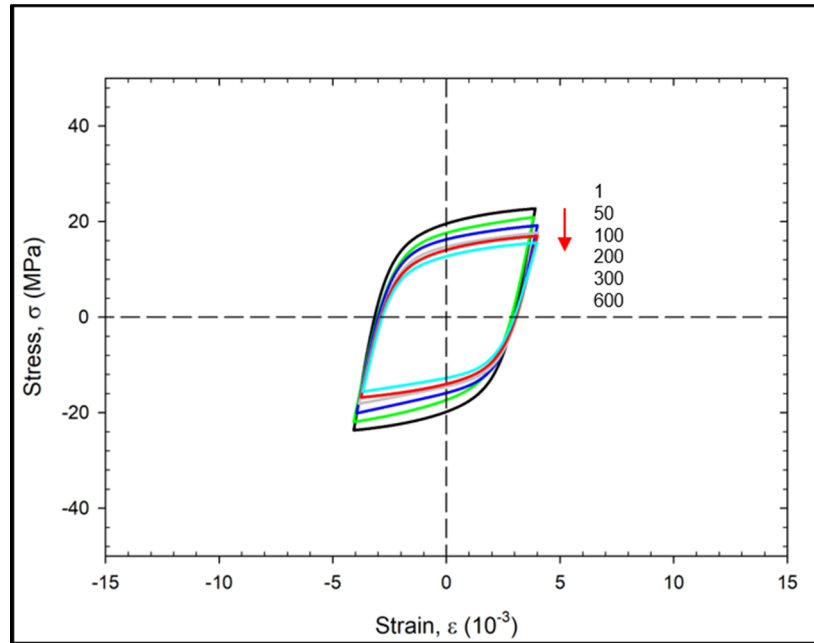
b) Reduction of Normalized Loop Area with Cycling

Figure 6.10 Reduction of Cyclic Properties of SAC305 with Mechanical Cycling (25 °C)

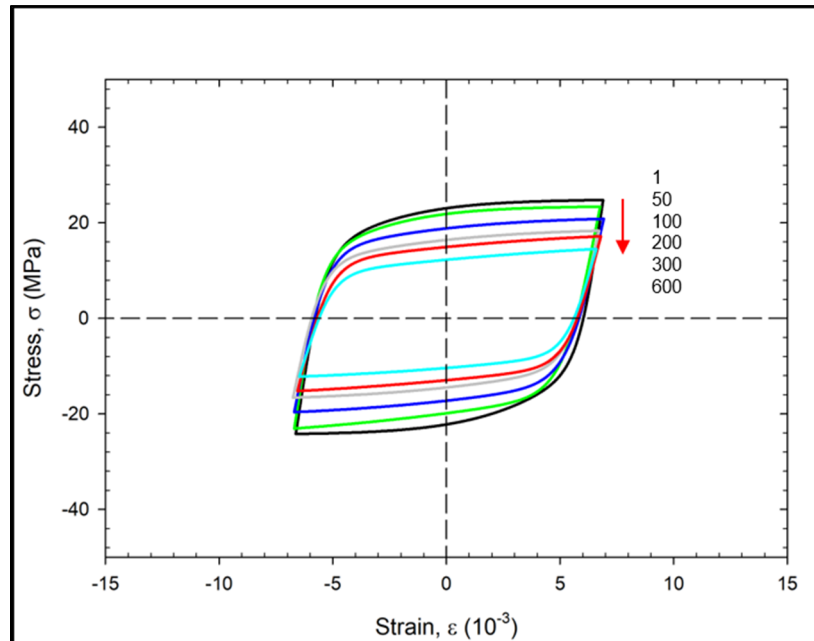
6.3.2 Elevated Temperature (100 °C) Cycling

Similar to the previous section, SAC305 samples were also cycled at a temperature of 100 °C and a strain rate of $\dot{\epsilon} = 0.002 \text{ s}^{-1}$. Strain ranges were picked such that the initial plastic work dissipated within the sample, ΔW , had values of approximately 0.25, 0.5, 0.75, 1 MJ/m³. Once these approximate strain ranges were obtained, cylindrical uniaxial SAC305 samples were subjected to mechanical cycling of these particular strain range values. These strain ranges had a higher magnitude than those obtained at room temperature cycling. This indicates that to generate the same hysteresis loop area, SAC305 samples would need to be cycled at a larger displacement when cycled at an elevated temperature. The constant strain mechanical cycling was conducted for up to 600 cycles at this elevated temperature. The evolution of the hysteresis loops with mechanical cycling for each of the selected ΔW values has been presented in Figure 6.11. For each value of ΔW , the hysteresis loops exhibit the same trend as observed with cycling at 25 °C. As the strain range increased, the peak stress increased and the decline in peak stress also increased with number of cycles. In addition to this, for the same ΔW , the hysteresis loops at 100 °C were much wider (indicating an increased plastic strain range) and had a lower peak stress when compared to its room temperature counterpart. Once again, the plastic strain range remained fairly constant with a consistent decline in peak stress and loop area as the cycling progressed. Figure 6.12 consists of a set of curves which show the evolution in the hysteresis loop with number of cycles for all four strain ranges. All the cyclic data have been compiled in Tables 6.4 – 6.6 and the graphical representation of the evolution of these properties have been displayed in Fig 6.13. From the curves in Fig. 6.13, we can

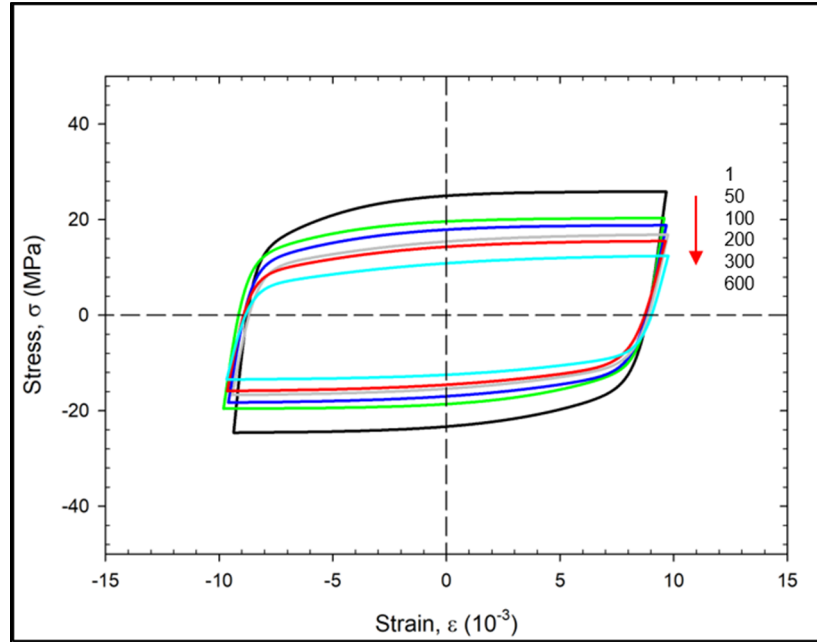
see that the decrease in peak stress and loop area with isothermal mechanical cycling are much harsher at elevated temperatures than at room temperature.



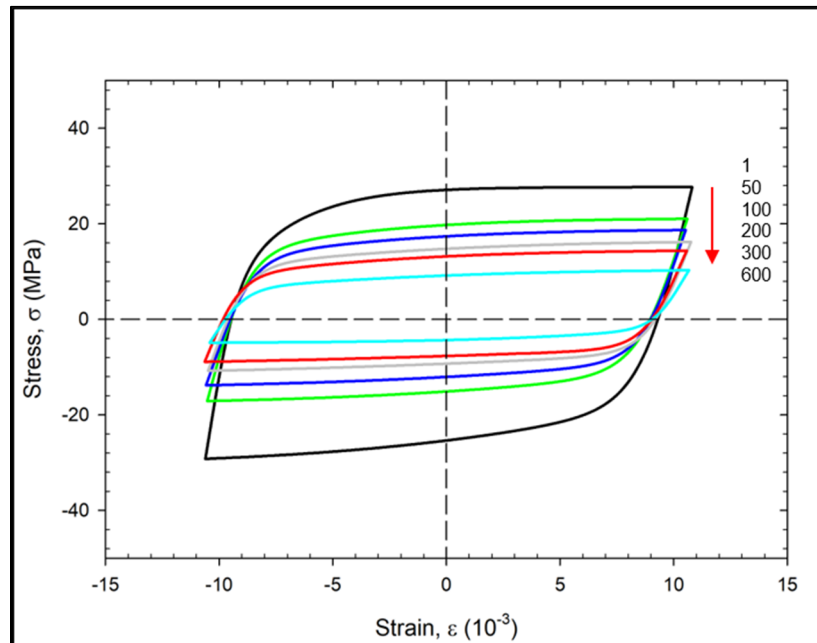
a) $\Delta W \approx 0.25 \text{ MJ/m}^3$



b) $\Delta W \approx 0.5 \text{ MJ/m}^3$

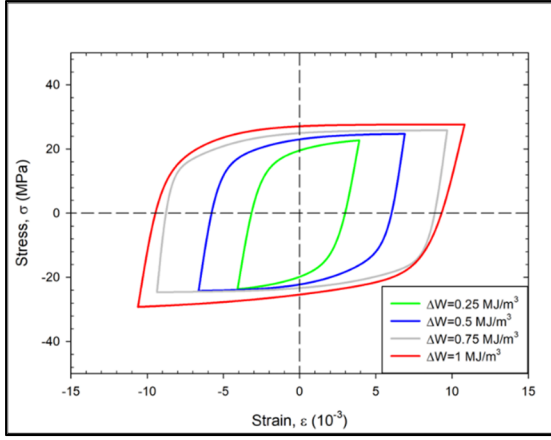


c) $\Delta W \approx 0.75 \text{ MJ/m}^3$

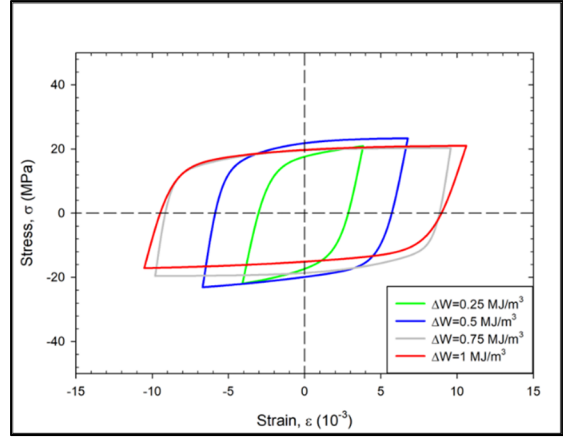


d) $\Delta W \approx 1 \text{ MJ/m}^3$

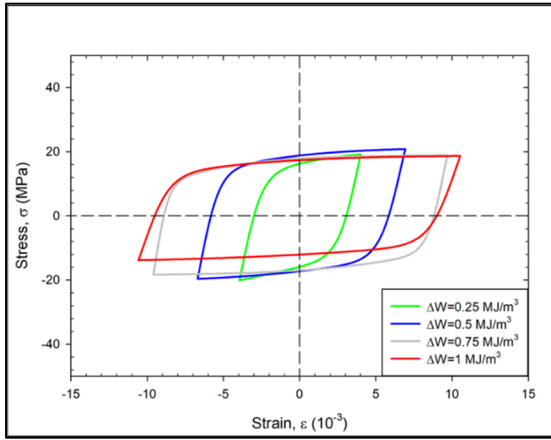
Figure 6.11 Evolution of Hysteresis Loop with Mechanical Cycling (SAC305 cycled at 100 °C)



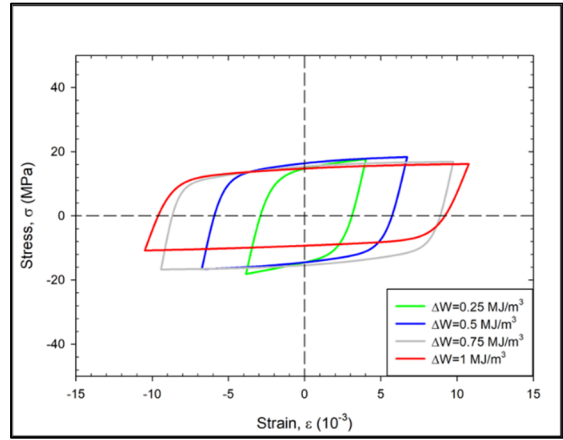
(a) 1 cycle



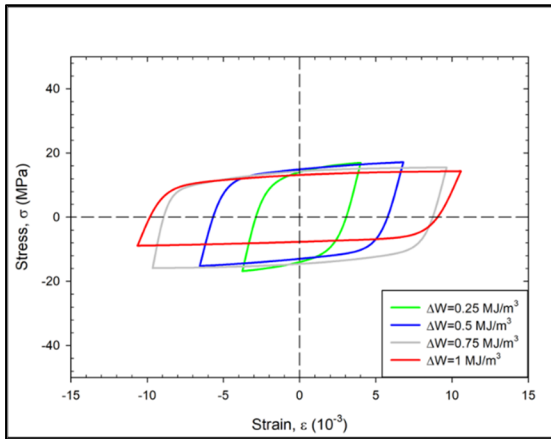
(b) 50 cycles



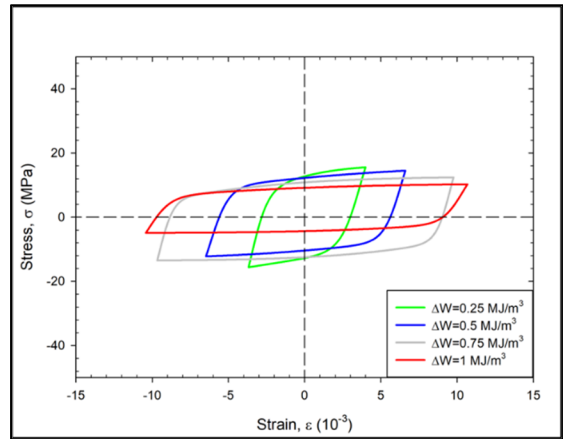
(c) 100 cycles



(d) 200 cycles



(e) 300 cycles



(f) 200 cycles

Figure 6.12 Hysteresis Loop Evolution of SAC305 with Number of cycles
(Cycled at 100 °C)

Table 6.4 Evolution of Hysteresis Loop Area with Mechanical Cycling at 100 °C (SAC305)

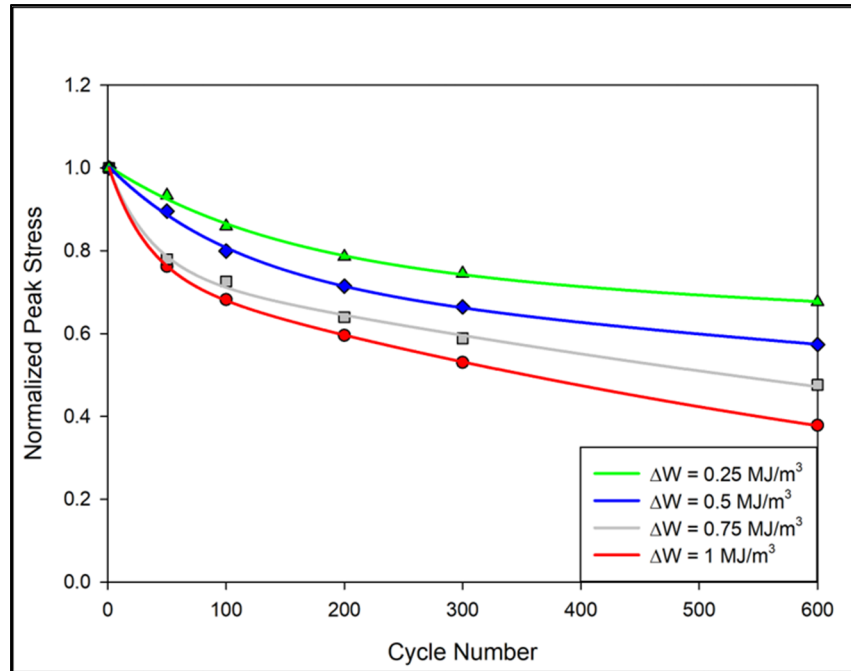
Number of Cycles	Hysteresis Loop Area, ΔW (MJ/m ³)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	0.24	0.56	0.81	0.98
50	0.20	0.46	0.64	0.65
100	0.19	0.42	0.59	0.55
200	0.17	0.38	0.53	0.47
300	0.16	0.36	0.49	0.42
600	0.15	0.30	0.39	0.29

Table 6.5 Evolution of Peak Stress with Mechanical Cycling at 100 °C (SAC305)

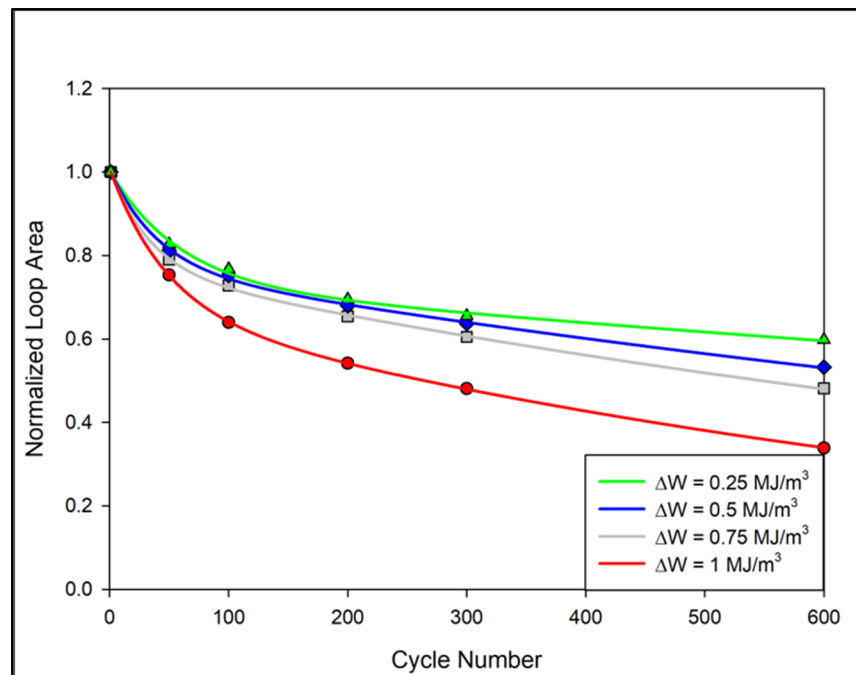
Number of Cycles	Peak Stress, $\sigma_{\max}$ (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	22.71	24.77	25.94	27.44
50	21.20	22.18	20.22	20.91
100	19.52	19.80	18.83	18.71
200	17.85	17.70	16.59	16.35
300	16.93	16.46	15.27	14.56
600	15.37	14.21	12.35	10.38

Table 6.6 Evolution of Plastic Strain Range with Mechanical Cycling at 100 °C (SAC305)

Number of Cycles	Plastic Strain Range, ε_p (%)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
1	0.59	1.17	1.78	1.88
50	0.57	1.15	1.78	1.87
100	0.57	1.15	1.78	1.88
200	0.57	1.14	1.76	1.88
300	0.58	1.15	1.76	1.89
600	0.58	1.16	1.76	1.88



a) Reduction of Normalized Peak Stress with Cycling



b) Reduction of Normalized Loop Area with Cycling

Figure 6.13 Reduction of Cyclic Properties of SAC305 with Mechanical Cycling (100 °C)

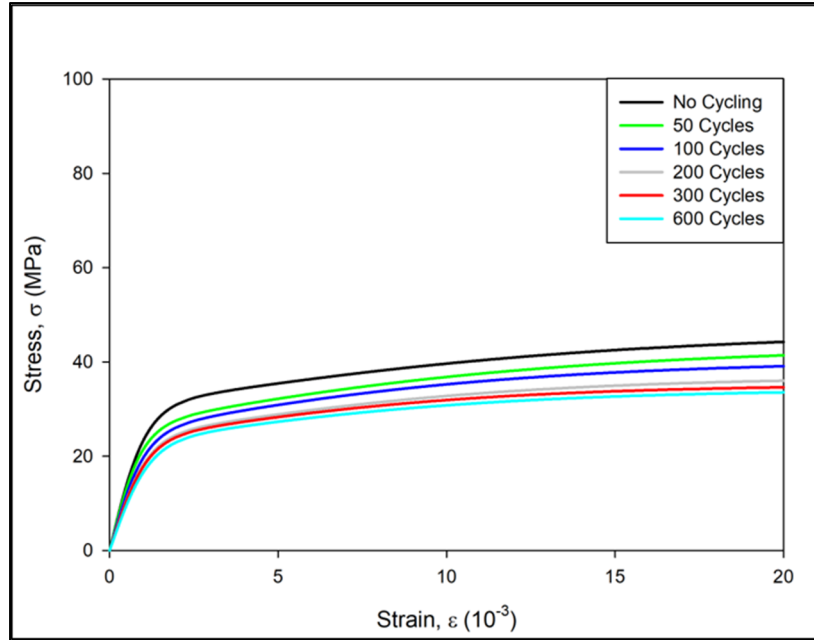
6.4 Evolution of Stress-Strain Behavior with Mechanical Cycling

This section focuses on quantifying the damage accumulation in the lead free alloys when they are mechanical cycled at different strain ranges for both the specified cycling temperatures. This comprised of initially subjecting the uniaxial circular cross-section samples to prior mechanical cycles of durations above the average fatigue failure life of the alloys (e.g. 0, 50, 100, 200, 300, and 600 cycles). Once the specimens were cycled up to either of the aforementioned cycle number at 100 °C, they were stored in a freezer at -40 °C to prevent any aging effects. These samples were then subjected to monotonic tensile tests at 25 °C. The results obtained for the two cycling temperatures will now be assessed individually.

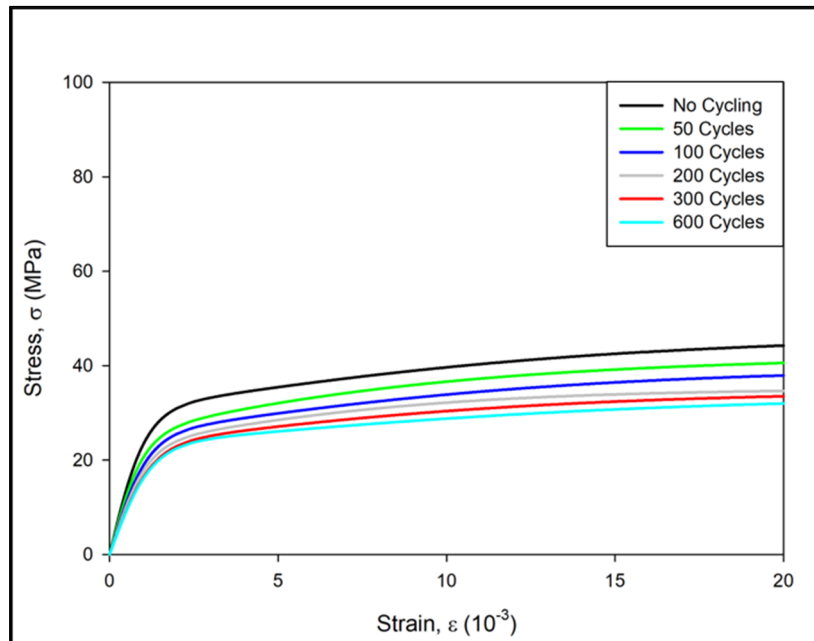
6.4.1 Room Temperature (25 °C) Cycling

Figure 6.14 displays the measured evolution of the tensile stress-strain curves obtained for 25 °C pre-cycled SAC305 samples at the four strain ranges. The same strain ranges used to obtain an initial plastic work of ($\Delta W \approx 0.25, 0.5, 0.75, 1 \text{ MJ/m}^3$) were used to obtain the pre-cycled samples. Similar monotonic tensile stress-strain curves obtained for 25 °C pre-cycled SAC305 samples was presented in Figure 4.5. The stress-strain data from five experimental tests were fit with a four parameter hyperbolic tangent model to generate an average stress-strain curve for each set of prior cycling conditions. Each of the monotonic stress-strain curves represents the tensile curve obtained after pre-cycling samples up to that specific number of cycles. This was done for all the initial plastic work mentioned previously. All four curves show a similar trend in the evolution of the curves. Cycling the samples for 50 cycles was sufficient to drastically reduce the Elastic modulus (initial slope) and the ultimate tensile strength (UTS, peak stress); the effect being least

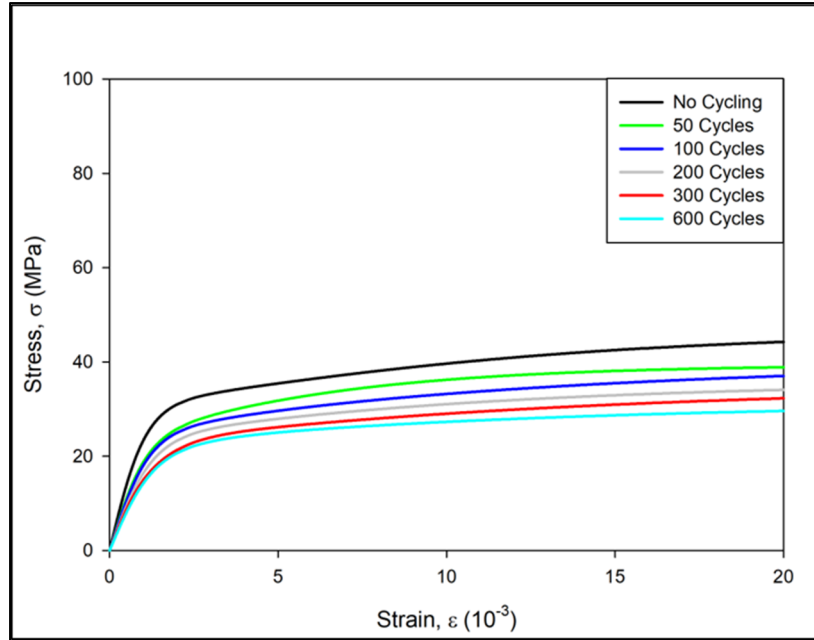
prominent when the samples were cycled at $\Delta W \approx 0.25 \text{ MJ/m}^3$ while the most drastic effect was observed when the samples were cycled at $\Delta W \approx 1 \text{ MJ/m}^3$. Figure 6.15 consists of a set of curves which show the evolution in tensile curves with number of cycles for all the four different strain ranges. The black curve represents the stress-strain curve obtained when pristine SAC305 samples were subjected to monotonic tensile test at 25 °C. The green, blue, gray, and red curves represent the tensile curves obtained when the pre-cycled samples had an initial hysteresis loop area, ΔW , of 0.25, 0.5, 0.75, and 1 MJ/m^3 , respectively. Table 6.7 – 6.9 summarizes the calculated values of the effective elastic modulus (E), Ultimate Tensile Strength (UTS), and yield stress (YS) for SAC305 specimens pre-cycled at the four values of initial plastic work dissipated, respectively. The values suggest that significant damage is accumulated within the samples when they are cycled at different strain ranges. This accumulated damage continues to increase in magnitude as the duration of cycles increased as is evident from the changes in the values of the Elastic modulus, UTS and the yield stress. It was observed that cycling SAC305 at 25 °C for 600 cycles with an initial plastic work of $\Delta W \approx 0.25 \text{ MJ/m}^3$ brought about a reduction of about 36%, 29%, and 23% in Elastic modulus, UTS, and Yield Stress, respectively. Similarly, cycling the SAC305 specimens at 25 °C for 600 cycles with an initial plastic work of $\Delta W \approx 1 \text{ MJ/m}^3$ resulted in a reduction of 51%, 41%, and 31% in Elastic modulus, UTS, and Yield Stress, respectively. The reductions in normalized Elastic modulus, UTS, and yield stress have been graphically presented in Figure 6.16.



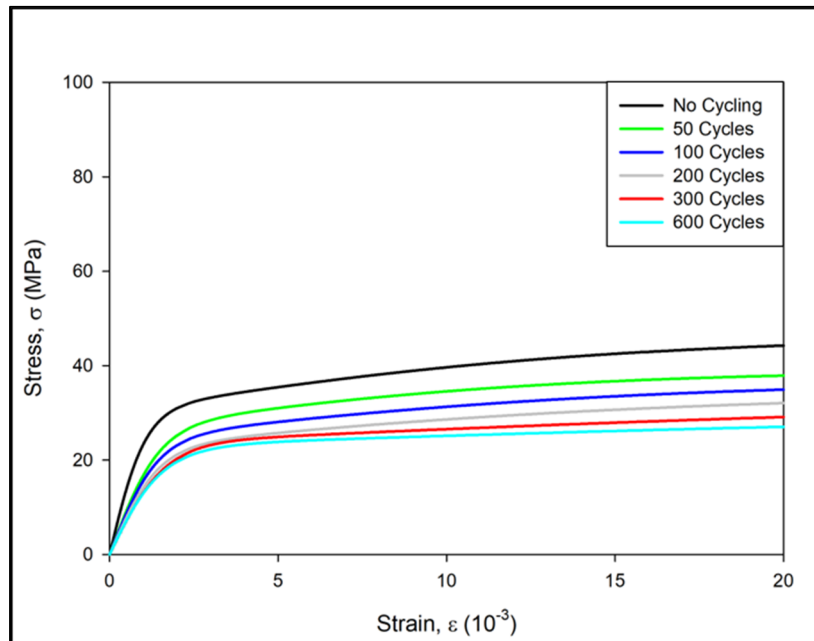
a) $\Delta W \approx 0.25 \text{ MJ/m}^3$



b) $\Delta W \approx 0.5 \text{ MJ/m}^3$



c) $\Delta W \approx 0.75 \text{ MJ/m}^3$



d) $\Delta W \approx 1 \text{ MJ/m}^3$

Figure 6.14 Evolution of Tensile Stress-Strain Curve with Mechanical Cycling (SAC305 cycled at 25 °C)

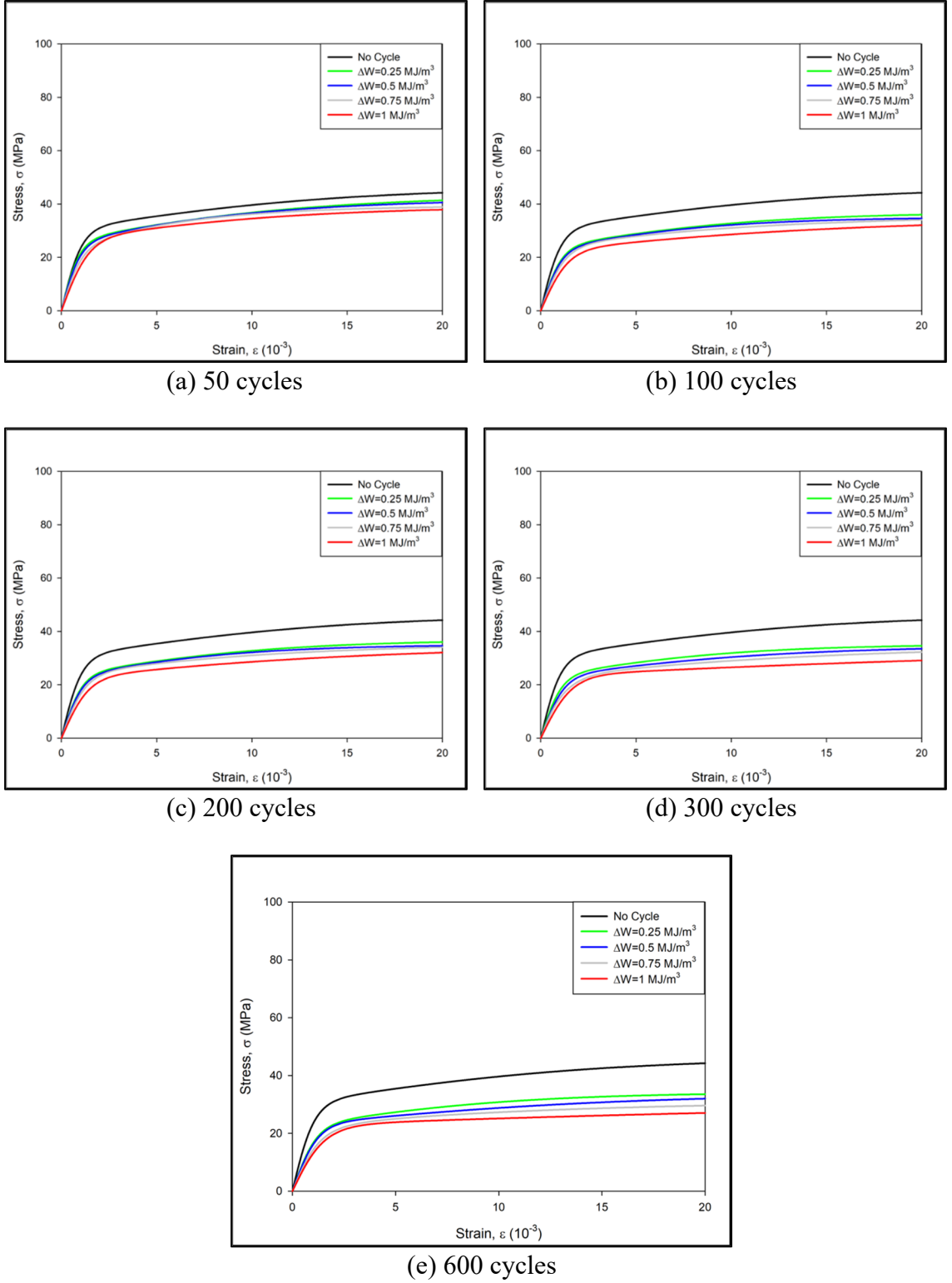


Figure 6.15 Tensile Curve Evolution of 25 °C Pre-cycled SAC305 with Number of cycles

Table 6.7 Evolution of Initial Elastic Modulus with Mechanical Cycling at 25 °C (SAC305)

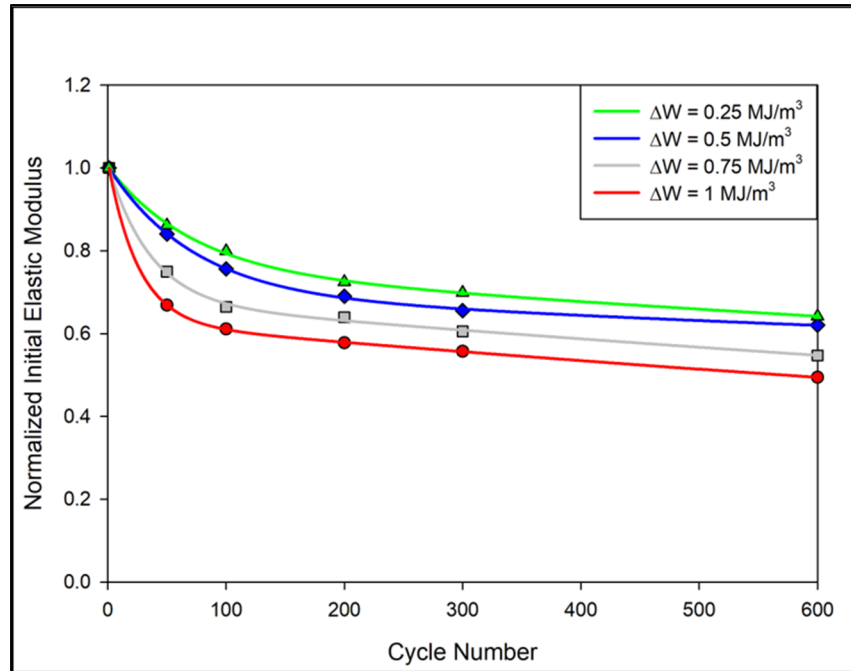
Number of Cycles	Initial Elastic Modulus, E (GPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	37.72	37.72	37.72	37.72
50	32.49	31.70	28.28	25.23
100	30.15	28.52	25.07	23.05
200	27.34	26.04	24.12	21.80
300	26.36	24.74	22.85	21.02
600	24.22	23.41	20.65	18.64

Table 6.8 Evolution of UTS with Mechanical Cycling at 25 °C (SAC305)

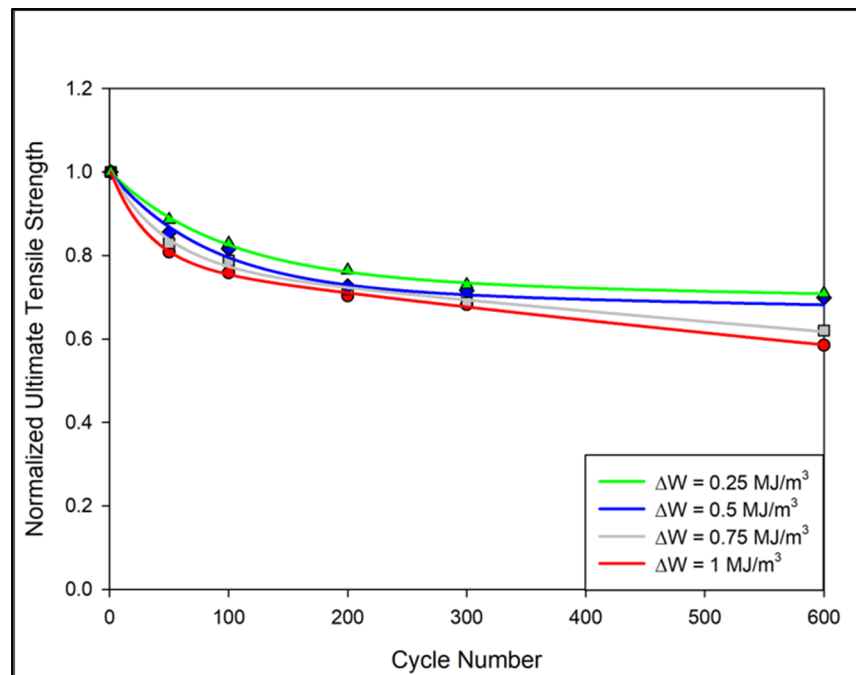
Number of Cycles	Ultimate Tensile Strength, σ_{uts} (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	48.19	48.19	48.19	48.19
50	42.68	41.20	39.98	38.95
100	39.94	39.33	37.96	36.55
200	36.86	35.02	34.68	33.89
300	35.16	34.50	33.14	32.88
600	34.18	33.69	29.90	28.20

Table 6.9 Evolution of Yield Stress with Mechanical Cycling at 25 °C (SAC305)

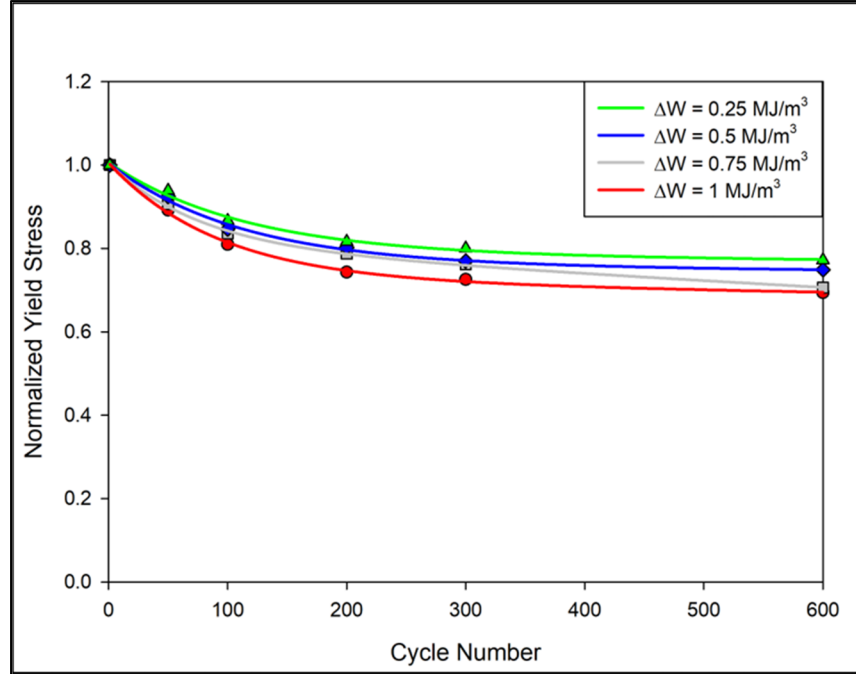
Number of Cycles	Yield Stress, σ_{ys} (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	33.09	33.09	33.09	33.09
50	31.07	30.59	29.98	29.52
100	28.67	28.02	27.66	26.79
200	27.02	26.54	26.05	24.59
300	26.48	25.47	25.21	24.01
600	25.56	24.78	23.36	22.97



a) Reduction of Normalized Initial Elastic Modulus with Cycling



b) Reduction of Normalized Ultimate Tensile Strength with Cycling



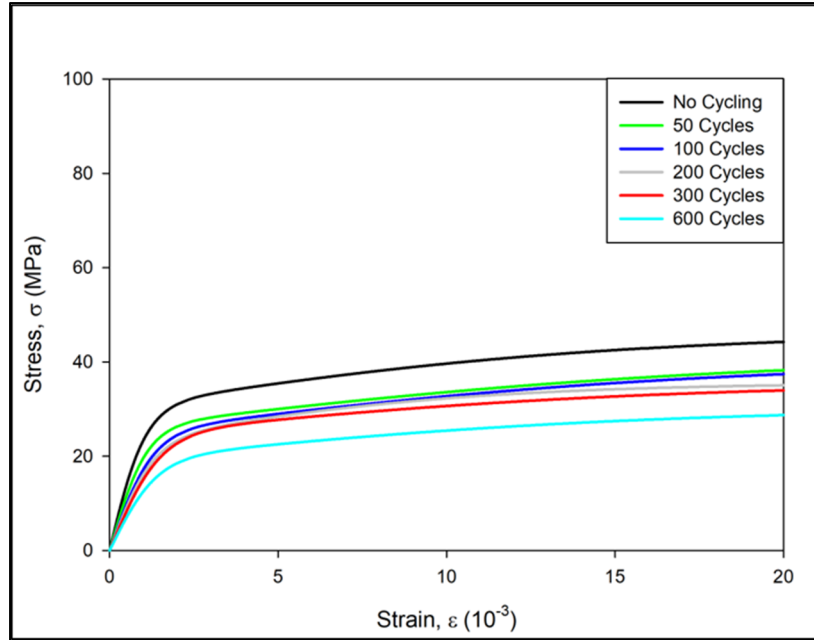
c) Reduction of Normalized Yield Stress with Cycling

Figure 6.16 Evolution of tensile curve properties of 25 °C pre-cycled SAC305 samples

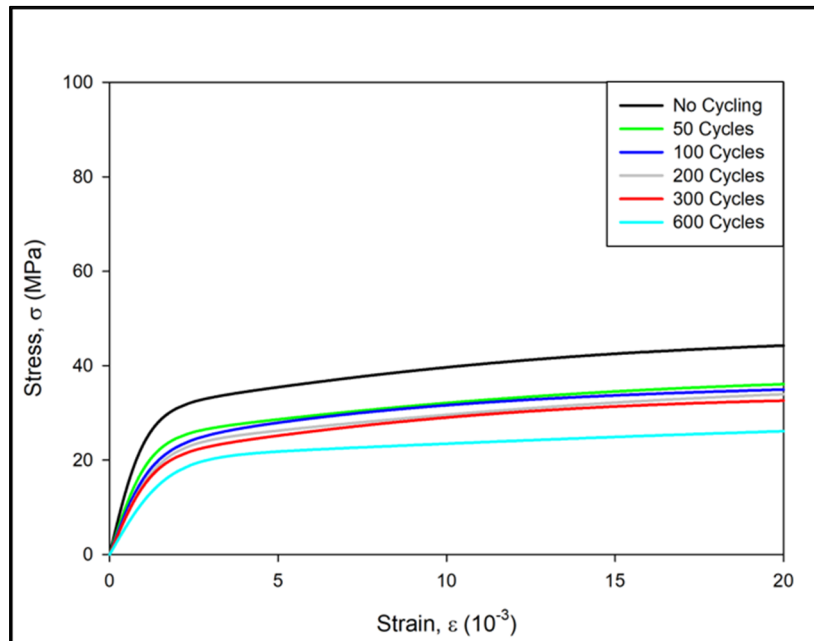
6.4.2 Elevated Temperature (100 °C) Cycling

Figure 6.17 displays the measured evolution of the tensile stress-strain curves obtained for 100 °C pre-cycled SAC305 samples at the four strain ranges. The same strain ranges used to obtain an initial plastic work of ($\Delta W \approx 0.25, 0.5, 0.75, 1 \text{ MJ/m}^3$) at 100 °C were used to obtain the pre-cycled samples. Similar monotonic tensile stress-strain curves obtained for 100 °C pre-cycled SAC305 samples was presented in Figure 5.6. The stress-strain data from five experimental tests were fit with a four parameter hyperbolic tangent model to generate an average stress-strain curve for each set of prior cycling conditions. Each of the monotonic stress-strain curves represents the tensile curve obtained after pre-cycling samples up to that specific number of cycles. This was done for all the initial plastic work mentioned previously. All four curves show a similar trend in the evolution

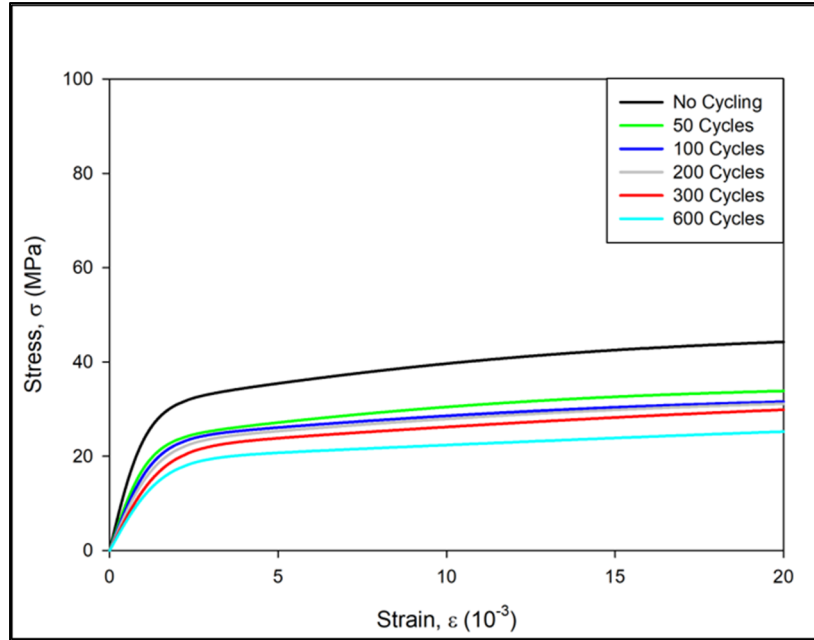
of the curves. Cycling the samples for 50 cycles was sufficient to drastically reduce the Elastic modulus (initial slope) and the ultimate tensile strength (UTS, peak stress); the effect being least prominent when the samples were cycled at $\Delta W \approx 0.25 \text{ MJ/m}^3$ while the most drastic effect was observed when the samples were cycled at $\Delta W \approx 1 \text{ MJ/m}^3$. Figure 6.18 consists of a set of curves which show the evolution in tensile curves with number of cycles for all the four different strain ranges. The black curve represents the stress-strain curve obtained when pristine SAC305 samples were subjected to monotonic tensile test at 25 °C. The green, blue, gray, and red curves represent the tensile curves obtained when the pre-cycled samples had an initial hysteresis loop area, ΔW , of 0.25, 0.5, 0.75, and 1 MJ/m^3 , respectively. Table 6.10 – 6.12 summarizes the calculated values of the effective elastic modulus (E), Ultimate Tensile Strength (UTS), and yield stress (YS) for SAC305 specimens pre-cycled at the four values of initial plastic work dissipated, respectively. The values suggest that significant damage is accumulated within the samples when they are cycled at different strain ranges. This accumulated damage continues to increase in magnitude as the duration of cycles increased as is evident from the changes in the values of the Elastic modulus, UTS and the yield stress. It was observed that cycling SAC305 at 100 °C for 600 cycles with an initial plastic work of $\Delta W \approx 0.25 \text{ MJ/m}^3$ brought about a reduction of about 62%, 35%, and 36% in Elastic modulus, UTS, and Yield Stress, respectively. Similarly, cycling the SAC305 specimens at 100 °C for 600 cycles with an initial plastic work of $\Delta W \approx 1 \text{ MJ/m}^3$ resulted in a reduction of 83%, 58%, and 57% in Elastic modulus, UTS, and Yield Stress, respectively. The changes in the normalized Elastic modulus, UTS, and yield stress have been graphically presented in Figure 6.19.



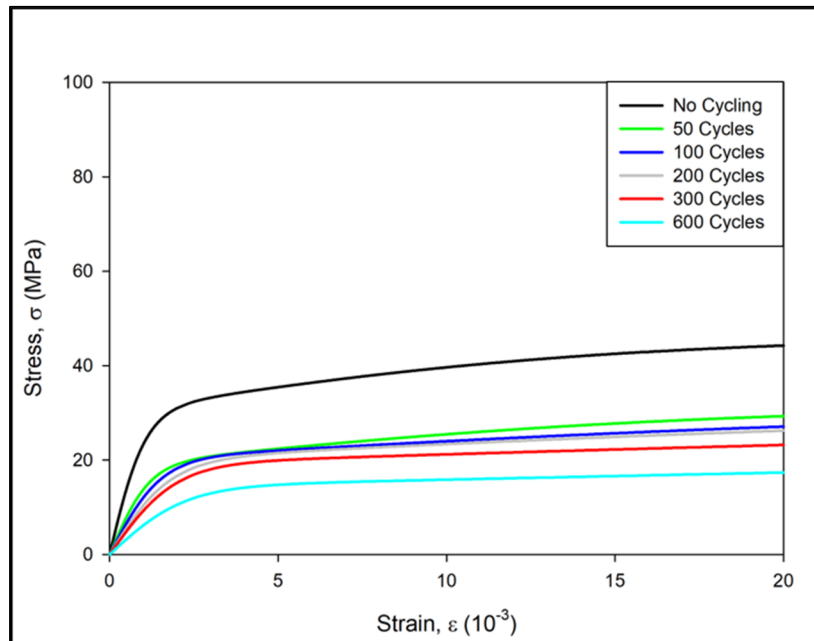
a) $\Delta W \approx 0.25 \text{ MJ/m}^3$



b) $\Delta W \approx 0.5 \text{ MJ/m}^3$

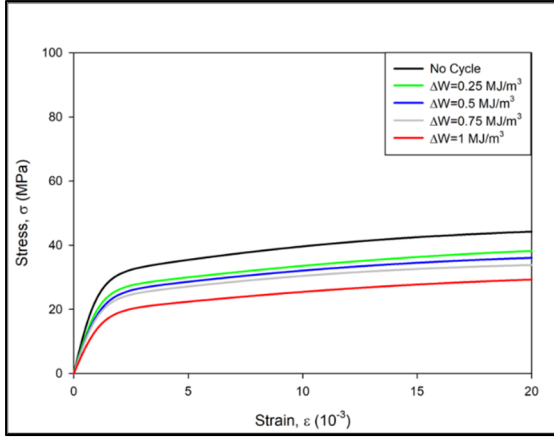


c) $\Delta W \approx 0.75 \text{ MJ/m}^3$

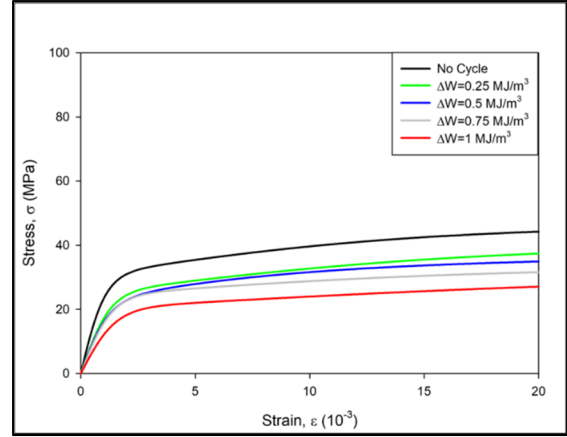


d) $\Delta W \approx 1 \text{ MJ/m}^3$

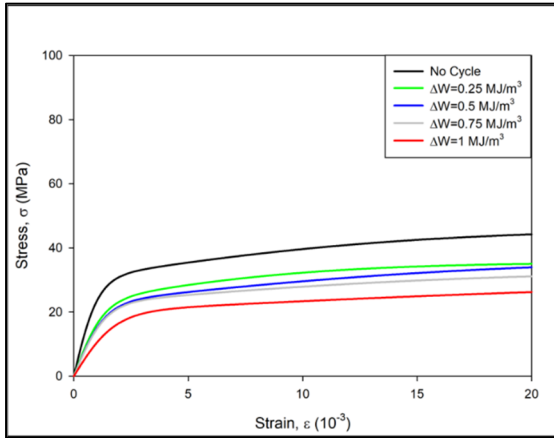
Figure 6.17 Evolution of Tensile Stress-Strain Curve with Mechanical Cycling (SAC305 cycled at 100 °C)



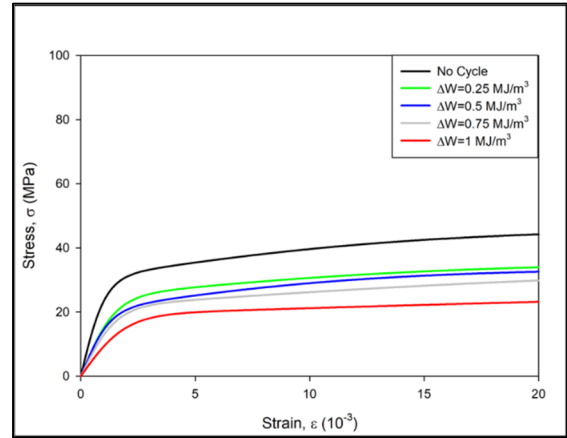
(a) 50 cycles



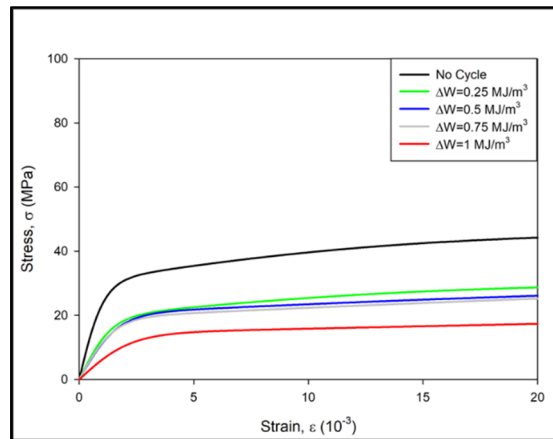
(b) 100 cycles



(c) 200 cycles



(d) 300 cycles



(e) 600 cycles

Figure 6.18 Tensile Curve Evolution of 100 °C Pre-cycled SAC305 with Number of cycles

Table 6.10 Evolution of Initial Elastic Modulus with Mechanical Cycling at 100 °C (SAC305)

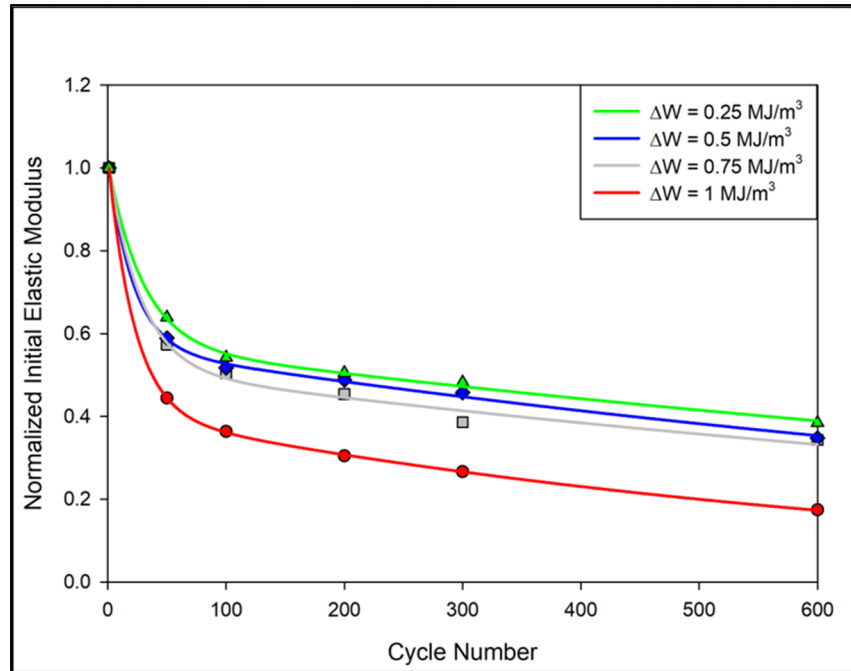
Number of Cycles	Initial Elastic Modulus, E (GPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	37.72	37.72	37.72	37.72
50	24.11	22.22	21.63	16.75
100	20.46	19.52	19.02	13.72
200	19.05	18.41	17.11	11.49
300	18.19	17.24	14.55	10.05
600	14.51	13.12	12.95	6.57

Table 6.11 Evolution of UTS with Mechanical Cycling at 100 °C (SAC305)

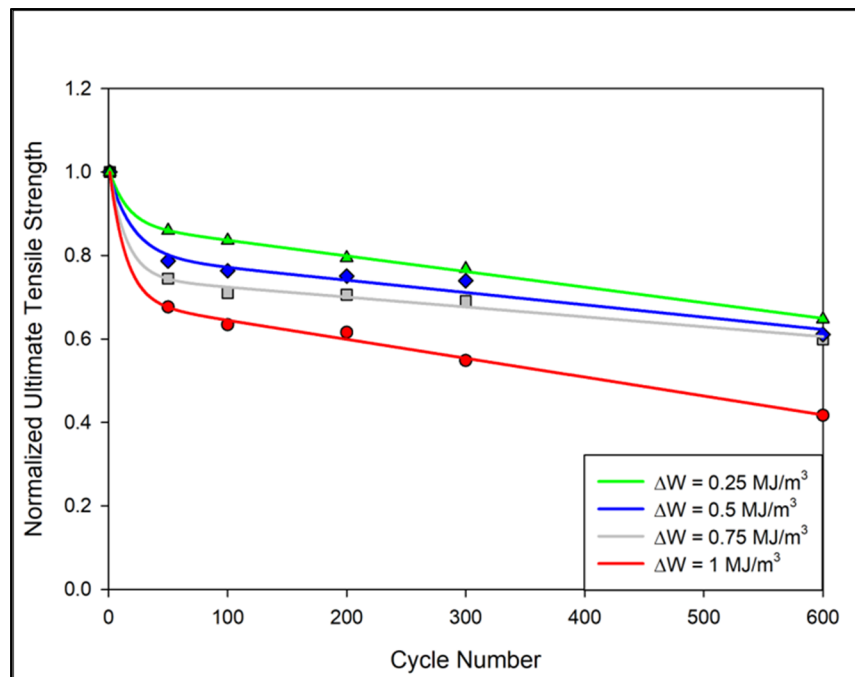
Number of Cycles	Ultimate Tensile Strength, σ_{uts} (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	48.19	48.19	48.19	48.19
50	41.45	37.93	35.89	32.61
100	40.3	36.78	34.24	30.56
200	38.29	36.18	34.02	29.69
300	37.01	35.64	33.30	26.43
600	31.22	29.43	28.87	20.09

Table 6.12 Evolution of Yield Stress with Mechanical Cycling at 100 °C (SAC305)

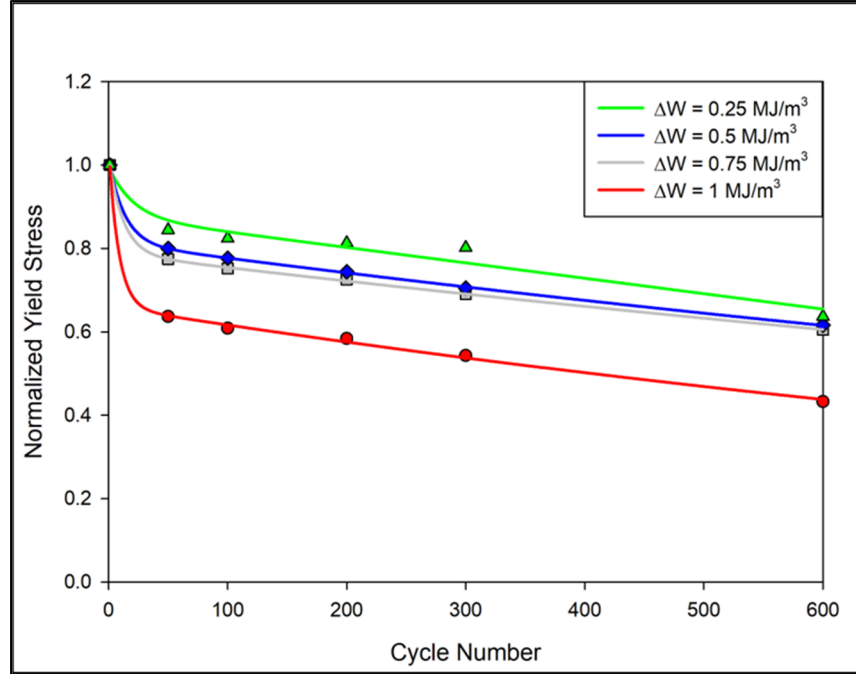
Number of Cycles	Yield Stress, σ_{ys} (MPa)			
	$\Delta W \approx 0.25$ MJ/m ³	$\Delta W \approx 0.5$ MJ/m ³	$\Delta W \approx 0.75$ MJ/m ³	$\Delta W \approx 1$ MJ/m ³
0	33.09	33.09	33.09	33.09
50	27.94	26.47	25.64	21.07
100	27.27	25.7	24.88	20.14
200	26.9	24.62	24.00	19.33
300	26.53	23.33	22.85	17.98
600	21.07	20.39	20.02	14.31



a) Reduction of Normalized Initial Elastic Modulus with Cycling



b) Reduction of Normalized Ultimate Tensile Strength with Cycling



c) Reduction of Normalized Yield Stress with Cycling

Figure 6.19 Evolution of tensile curve properties of 100 °C pre-cycled SAC305 samples

6.5 Summary and Discussion

In this chapter, the effects of different strain ranged mechanical cycling on the damage accumulation in lead free alloys were investigated. Uniaxial cylindrical SAC305, SAC_Q and Innolot samples were prepared and subjected to constant strain cyclic tests at 7 different strain ranges at a test temperature of 25 °C. Initial hysteresis loops for each cyclic condition of each alloy were recorded and plotted. These initial hysteresis loop data were then used to construct charts of initial plastic work accumulated vs applied cyclic strain range. Such charts would allow the estimation of the initial plastic work dissipated at any given strain range for the particular strain rate used.

The SAC305 chart was then used to estimate the strain ranges to obtain an initial plastic work, ΔW , of approximately 0.25, 0.5, 0.75, and 1 MJ/m³. Uniaxial cylindrical

SAC305 samples were then cycled at these 4 strain ranges at room temperature for 600 cycles. Similarly, strain range levels needed to obtain an initial plastic work, ΔW , of approximately 0.25, 0.5, 0.75, and 1 MJ/m³ at a cycling temperature of 100 °C were also evaluated. SAC305 samples were then also cycled at these 4 strain levels at 100 °C for 600 cycles. The hysteresis loops obtained for all the conditions were recorded and the different cyclic properties (peak stress, plastic strain range, and plastic strain energy density) were extracted from these hysteresis loops. This allowed us to evaluate the evolution of the peak stress and hysteresis loop area with cycling. It also allowed the comparison in the difference in evolution of cyclic properties when SAC305 was cycled at the same initial plastic work accumulated at the two cycling temperatures. The cyclic properties also reduced with number of cycles at higher applied strain ranges. It was observed that the elevated temperature cycling brought about a much larger reduction in the cyclic properties despite cycling at the same initial plastic work accumulated level. This suggests that the decrease in properties brought about by elevated temperature cycling is much harsher than the decrease in properties brought about by cycling at a larger strain.

To gain a better knowledge of the damage accumulation accompanying the isothermal mechanical cycling, pristine SAC305 samples were mechanically cycled for durations of 0, 50, 100, 200, 300 and 600 cycles at temperatures of $T = 25$ and 100 °C. These prior cycled specimens were cycled at strain ranges previously mentioned to obtain specific values of initial plastic work accumulated. These pre-cycled samples were then subjected to room temperature monotonic tensile tests to quantify the damage accumulated in the samples due to the cyclic tests. The trends observed in the reduction of the Elastic modulus, UTS, and yield stress were similar to that observed with the changes in hysteresis

loop area and peak stress reduction. It was once again observed that SAC305 samples cycled at 100 °C proved to have a larger damage accumulation with the same number of cycles and same initial plastic work when compared with its room temperature counterpart.

Overall, all these data can be incorporated in a constitutive model to better model the viscoplastic nature of lead free alloys in electronic packages. All most all the models used to estimate solder life fatigue assumes the plastic work accumulated within the lead free solder joint to be constant. This is not the case, and with the usage of this data, one should be able to better estimate the changes of various properties of lead free joints when subjected to cyclic loading.

CHAPTER 7

AGING EFFECTS ON THE CYCLIC PROPERTIES OF SAC305 LEAD FREE SOLDER

7.1 Introduction

Fatigue of solder joints exposed to thermal cycling is a common electronic packaging failure mode. Stresses and strains often result in electronic assemblies exposed to a temperature changing environment due to mismatches in the coefficients of thermal expansion (CTE) of the soldered components and the printed circuit board (PCB). Therefore, cyclic temperature changes can lead to alternating stresses and strains within the solder joints. Such exposures can occur in products that experience power cycling or during accelerated life thermal cycling tests used for qualification. Cyclic loading typically leads to micro cracks forming within the solder material, followed by macro cracks and eventually to fatigue failure. Energy dissipation occurs during cyclic loading due to yielding and occurrence of viscoplastic deformations, and can be quantified using the area of the cyclic stress-strain curve hysteresis loops, which is equal to the strain energy density dissipated per cycle. While effects of aging have been studied in some detail on the solder constitutive behavior such as stress-strain and creep, there have been few prior studies on the effects of aging on solder cyclic stress-strain behavior of lead free solders.

In this chapter, the accumulation of damage in SAC305 lead free solder during mechanical cycling at room temperature under various aging conditions was investigated. Circular cross-sectioned solder specimens were initially prepared and reflowed. These samples were then subjected to various aging conditions of 0 days, 1 day, 2 days, 5 days, 10 days and 30 days at an aging temperature of 125 °C in an environmental chamber. After

the aging period, the samples were mechanically cycled at room temperature at various strain ranges using a Micro-Mechanical tester. After aging, the fabricated samples were subjected to strain controlled cyclic stress-strain loading (constant positive and negative strain limits) at room temperature (25 °C). In this chapter, all specimens were tested at a constant strain rate of 0.002 sec⁻¹. The nominal diameter of the uniaxial specimens in this work was 1.2 mm. Seven strain limits were applied on the pre-aged samples, and the gage length was chosen as 5 mm. In addition, at least 5 tests were conducted for each solder alloy and aging condition. The cyclic stress-strain curves obtained were studied to observe the evolution of hysteresis loop properties (peak stress, hysteresis loop area, and plastic strain range) with mechanical cycling. The values obtained were then compared between the various aging conditions to understand the effect of aging duration on the cyclic properties of SAC305 lead free solder. Curves of initial plastic work accumulated vs applied strain range were also developed which would allow the estimation of the initial hysteresis loop area for any given strain range at a particular aging condition and strain rate. The mechanical behavior of a solder test specimen is closely tied to its microstructure. Therefore, to better understand aging induced evolution of cyclic stress-strain behavior, the effects of aging on solder microstructure of a fixed region have been studied using SEM.

7.2 Evolution of Cyclic Stress-Strain Curves with Aging

In this section, the evolution of the cyclic stress-strain curves with strain range will be presented. Uniaxial cylindrical SAC305 samples were subjected to mechanical cycling with strain ranges of $\epsilon = \pm 0.0025, \pm 0.0035, \pm 0.0045, \pm 0.006, \pm 0.0075, \pm 0.009, \text{ and } \pm 0.01$. The samples were tested at room temperature and a strain rate of $\dot{\epsilon} = 0.002 \text{ s}^{-1}$. A 5mm

gage length and a testing temperature of 25 °C was utilized for all the experiments. Samples were aged at an extreme temperature of 125 °C for 0, 1, 2, 5, 10, and 30 days. Aged samples were then stored in a freezer at -40 °C to prevent any further changes in constitutive properties and microstructure. The initial hysteresis cyclic curves obtained have been presented in Figs. 7.1 – 7.6. Each of the figures corresponds to one of the aforementioned aging durations. As computed in the previous sections, each of the hysteresis curves were fit from the raw data using eqns. 3.5 and 3.6. At least 5 samples were tested for each aging condition. The maximum stress at each strain range for each alloy was then fit using the four parameter hyperbolic tangent model (eqn. 3.2) to obtain the pink colored cyclic stress-strain curve shown in Figs. 7.1 – 7.6.

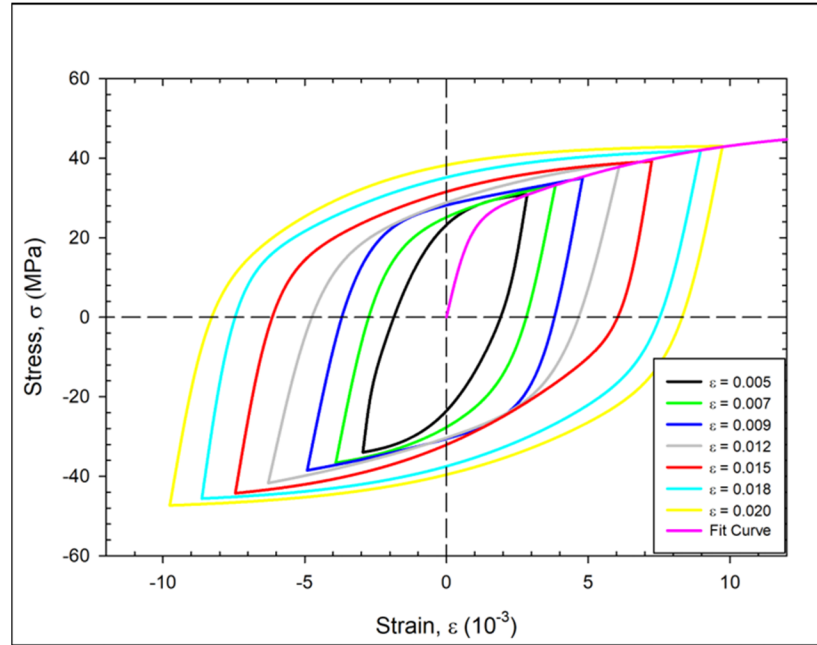


Figure 7.1 Evolution of hysteresis loop with strain range (SAC305 0 days aging)

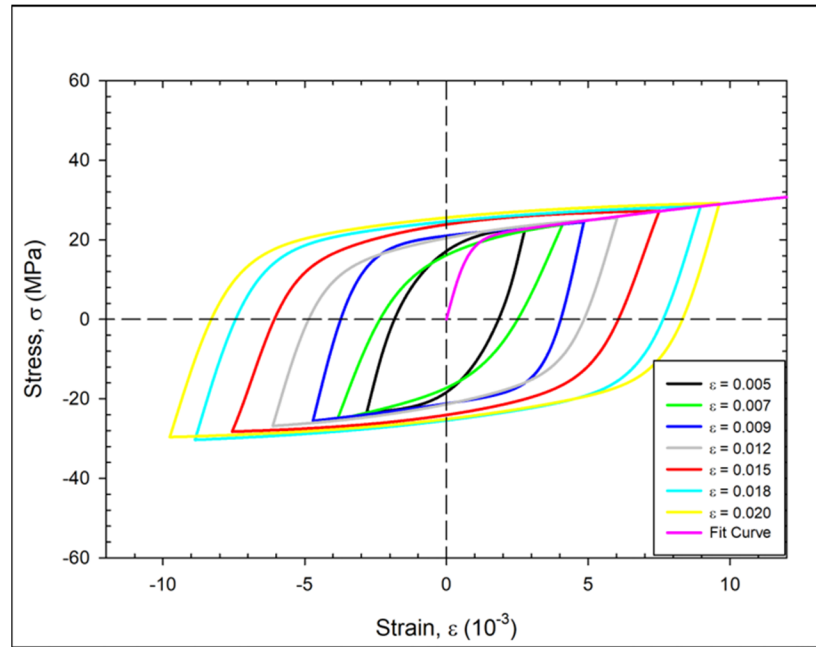


Figure 7.2 Evolution of hysteresis loop with strain range (SAC305 1 day aging)

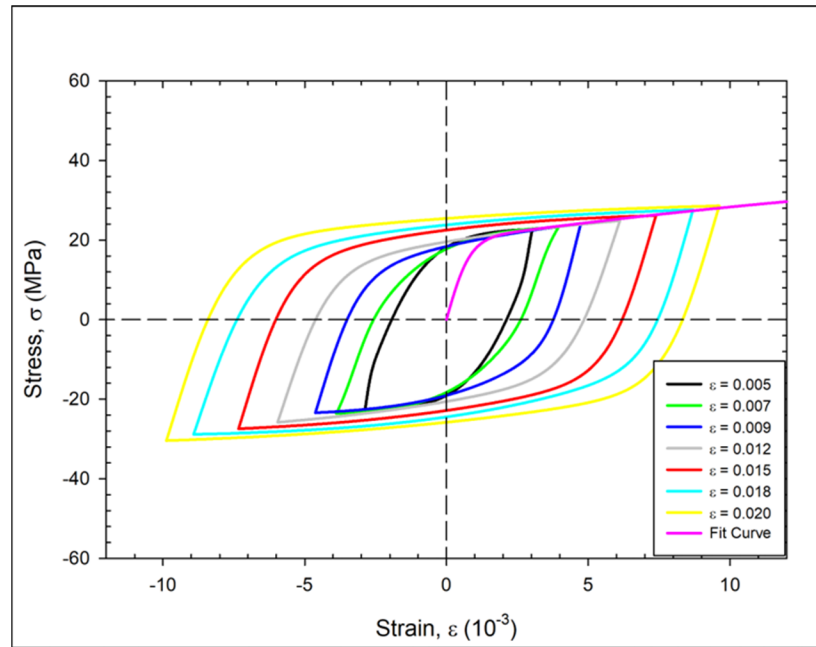


Figure 7.3 Evolution of hysteresis loop with strain range (SAC305 2 days aging)

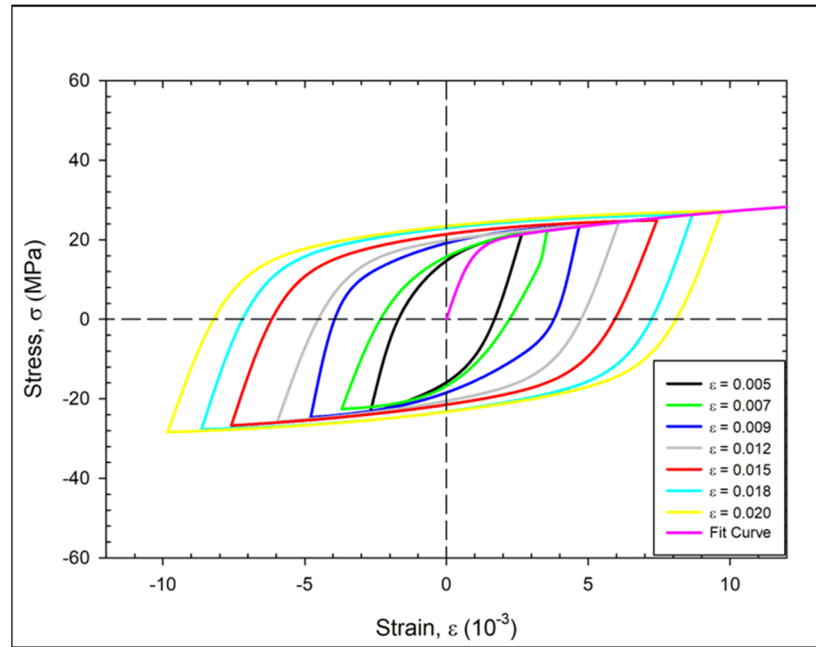


Figure 7.4 Evolution of hysteresis loop with strain range (SAC305 5 days aging)

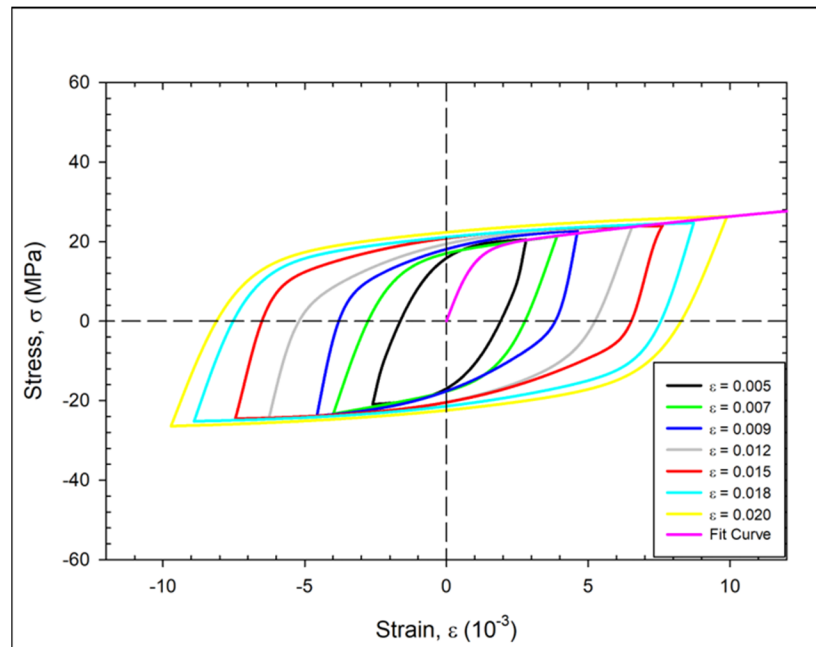


Figure 7.5 Evolution of hysteresis loop with strain range (SAC305 10 days aging)

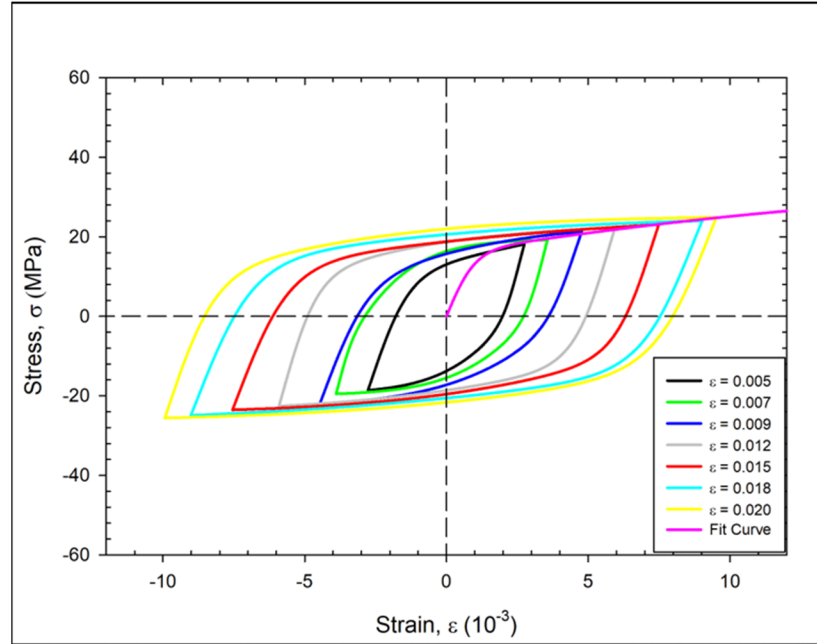


Figure 7.6 Evolution of hysteresis loop with strain range (SAC305 30 days aging)

Morrow hypothesized that on the microscopic level, cyclic stress is mostly affiliated to the ability to resist the movements while the cyclic plastic strain is associated with the dislocation movements. From the processed cyclic stress-strain curves, the hysteresis loop areas were computed using eqn. 3.7. It was observed that the hysteresis loop area increased as the applied strain range was increased. This increase in hysteresis loop area with various applied strain ranges at any particular aging duration at has been depicted in Figure 7.7. Such plots of initial plastic work accumulated vs the total applied strain range were also generated and presented in the previous chapter. This would allow the estimation of initial plastic work accumulated for any given strain range and aging duration ($\dot{\epsilon} = 0.002 \text{ s}^{-1}$). In doing so, one will be able to cycle SAC305 samples at different strain ranges and aging durations to achieve the same initial plastic work dissipated. Thus, a comparison between differences in cyclic properties (with the same initial strain energy stored) at various aging durations can be conducted.

For any particular strain range, the hysteresis loop area also decreased with an increased aging duration. The changes in hysteresis loop area vs aging duration (at fixed strain ranges) has been presented in Fig. 7.8. By analyzing Fig. 7.8, we can deduce that most of the reduction in initial hysteresis loop area at any given applied strain range, occurs within the first 5 days of the aging period. Beyond 5 days, the decrease in initial hysteresis loop area starts to flatten, indicating the stabilization of the initial hysteresis loop area with aging. The decrease of the hysteresis loop area with increased aging suggests that energy dissipation was mitigated with aging.

Peak stresses and plastic strain ranges were also calculated. The peak stress is defined as the maximum stress during the cycle after reaching a stable condition. A common trend was observed for all the aging conditions tested. For the smallest strain range (Total Strain Range, $\varepsilon = 0.005$), plastic deformation was little. With an increase in applied total strain range, the plastic deformation and the plastic strain range (distance between the two strain axes intercepts) increased in magnitude as is evident from the curves. The increment of plastic strain range with the increased aging times means more dislocation movements occurred during the test.

The peak stress also increased as the strain range increased but reaches a saturated value when the largest strain range (Total Strain Range, $\varepsilon = 0.02$) is applied. The variation in peak stress with aging duration at different applied strain levels has been presented in Fig 7.9. This evolution of peak stress with aging duration (at certain applied strain range levels) is shown in Figure 7.10. We observe that the sharpest decrease in peak stress at any applied strain level occurs within the first 2 days of aging. After 2 days of aging, the reduction in peak stress with aging duration decreases at a fairly constant rate. The

decrease of the peak stress with aging indicates the specimen was losing the ability to resist the movements of dislocations.

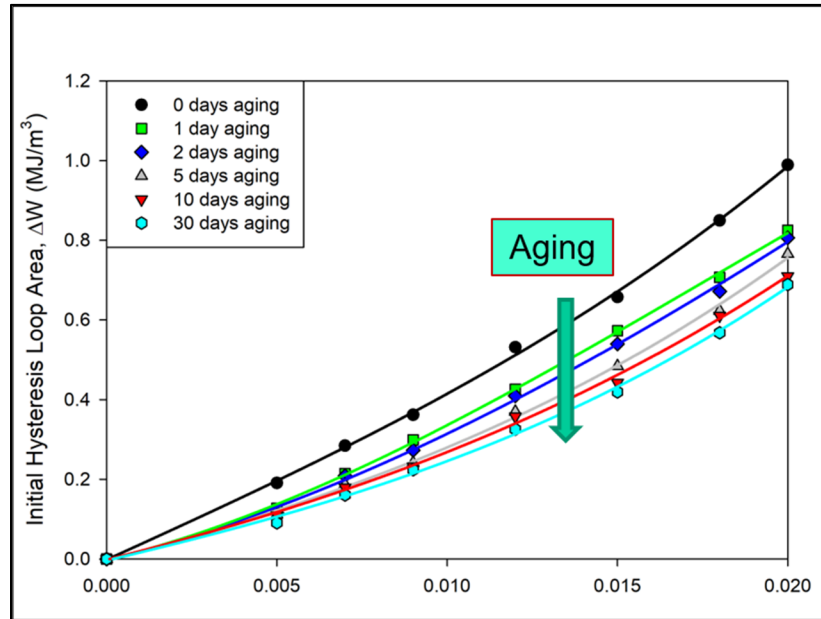


Figure 7.7 Plots of Plastic Work Accumulated vs Total Strain Range

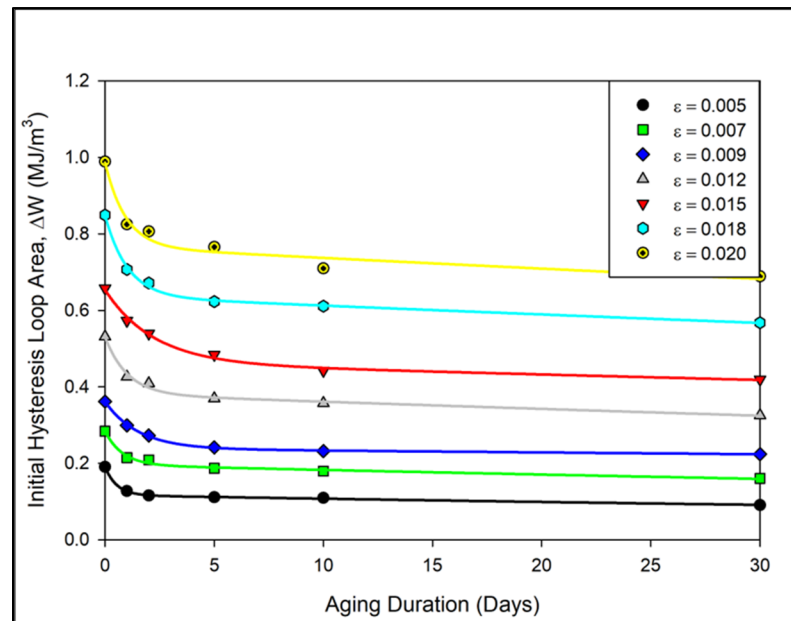


Figure 7.8 Plots of Plastic Work Accumulated vs Aging Duration

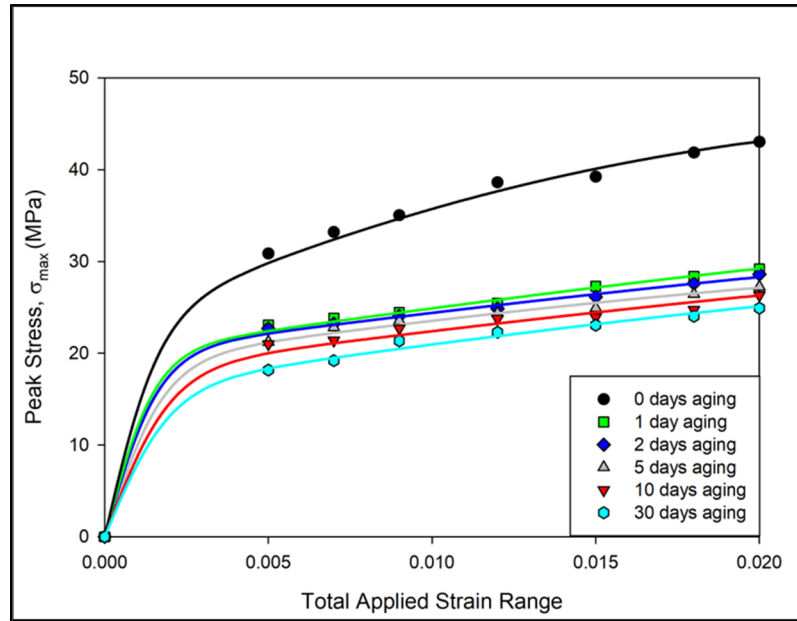


Figure 7.9 Plots of Peak Stress vs Total Strain Range

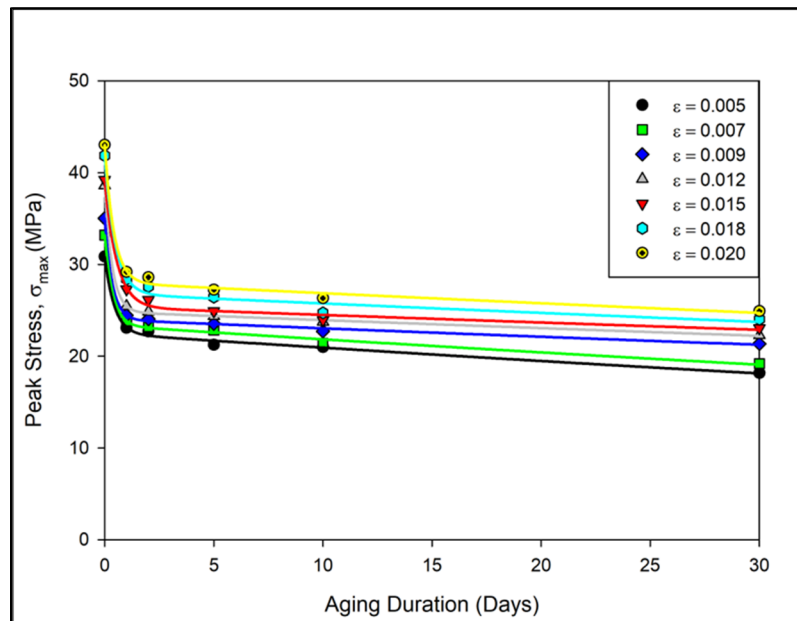
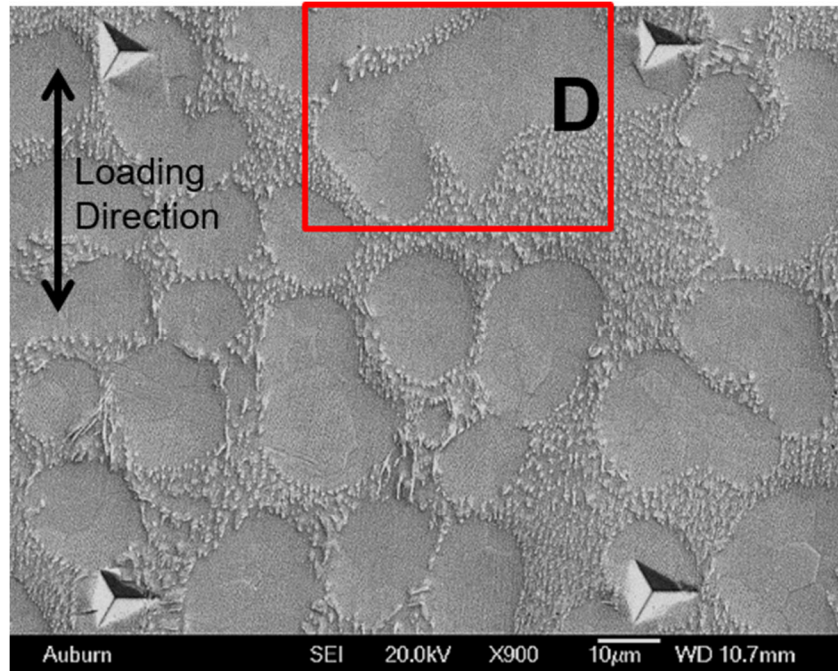


Figure 7.10 Plots of Peak Stress vs Aging Duration

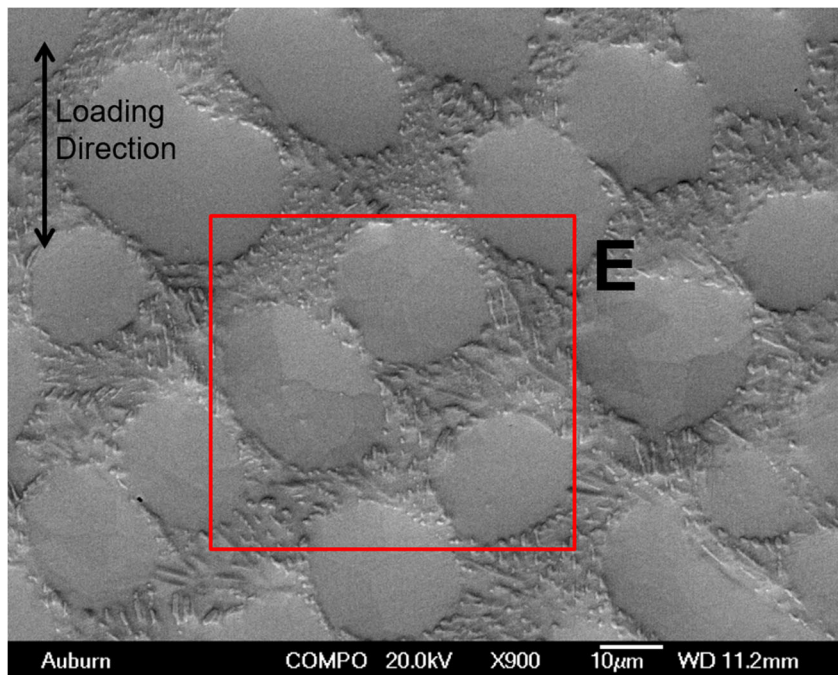
7.3 Evolution of Microstructure with Cycling and Aging

To observe microstructural evolution due to mechanical cycling, special rectangular cross-section uniaxial test specimens were prepared and polished. After visually inspecting a sample, it was temporarily attached to an epoxy preform/carrier using double sided tape, so that a 30 x 3 mm flat surface of the solder sample was exposed. The exposed surface was then polished using standard procedures until the microstructures were clearly visible under an optical microscope. To locate and track the selected fixed regions, regions of interest were demarcated using four nanoindentation marks placed at their corners using a Hysitron TI 950 nanoindentation system.

After polishing and applying the nanoindentation marks, the specimens were carefully removed from the temporary carriers and viewed under the SEM. Two regions of interest for each aging condition will be presented in this section (Figures 7.11 and 7.12). The samples were then aged at a temperature of 125 °C for 0 or 10 days. After the aging period, the same regions of interest were tracked under the SEM and corresponding images were taken. The nanoindentation marks facilitated in relocating the fixed regions of interest. The microtester was used to mechanically cycle these aged specimens at room temperature, and a total strain range of $\epsilon = \pm 0.005$ was used. A gage length of 5 mm was again used for the tests, as well as a strain rate of $= 0.002 \text{ sec}^{-1}$. After various durations of cycling (0, 25, 50, 75, 100, 200, 300 cycles), the specimens were removed from the testing machine and SEM observations were made of the microstructures in the fixed regions of interest. As seen in the figures, β -Sn dendrites are surrounded by eutectic regions of IMC particles. The brittle IMC particles were mostly Ag_3Sn and Cu_6Sn_5 and provided pinning points that helped mitigate and formed barriers against dislocation movements.



(a)



(b)

Figure 7.11 Typical non-cycled SAC305 microstructures

The regions E and F marked with a red box in the micrographs in Figure 7.11 were magnified and closely observed for changes in microstructure during the mechanical cycling. The corresponding micrographs depicting the cyclic induced damage in regions E and F are shown in Figures 7.12 and 7.13, respectively. The SAC305 sample microstructure presented in Fig 7.11) b) was then aged for 10 days at a temperature of 125 °C. Microstructural observations were then recorded with the SEM after both the aged and non-aged specimens were cycled for 0, 10, 25, 50, 100, 200, and 300 loading cycles. From the initial micrographs for non-aged region C (Fig 7.12), we can see the formation of microcracks originating in the β -Sn phase especially along the subgrain boundaries. These cracks grew in length as the number of cycles increased, propagated along the maximum tensile stress plane and in the process weakening the β -Sn dendrites. The cracks began to then coalesce and subsequently created larger transgranular cracks. Another observation was that not all cracks resulting from this fatigue induced damage nucleated from slip bands. The observation of peaks and valleys formed in the dendrite structure provides evidence of the creation of extrusions and intrusions. The formation of microvoids was evident after the 100 cycle loading period. No such changes (in size or distribution) or development of microcracks were noticed in the intermetallic or eutectic region. One can thus infer that this region does not play a major part in the development of the microcracks.

During the aging before cycling, the most obvious changes were the coarsening of IMC particles (e.g. differences between Figure 7.13 (a) and Figure 7.13 (b). In particular, the number of IMC particles decreased, while the average size of the particles and average particle separation distance increased significantly with aging. A common observation is

that larger particles are produced at the expense of smaller particles. This IMC coarsening phenomenon is also known as Ostwald Ripening.

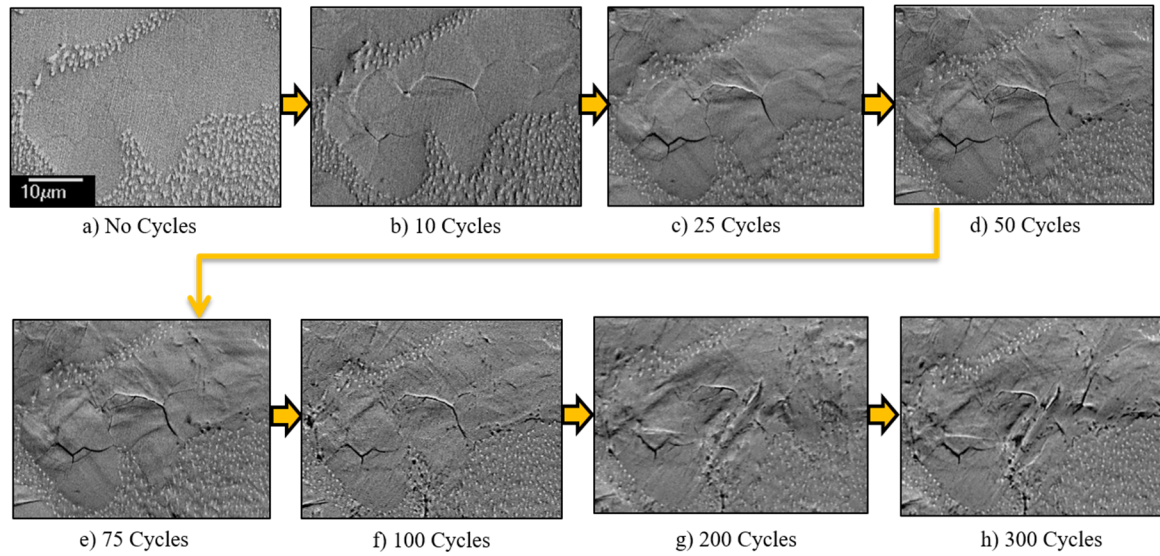


Figure 7.12 Microstructural Evolution of SAC305 with Mechanical Cycling at $T = 25\text{ }^{\circ}\text{C}$ (Region D)

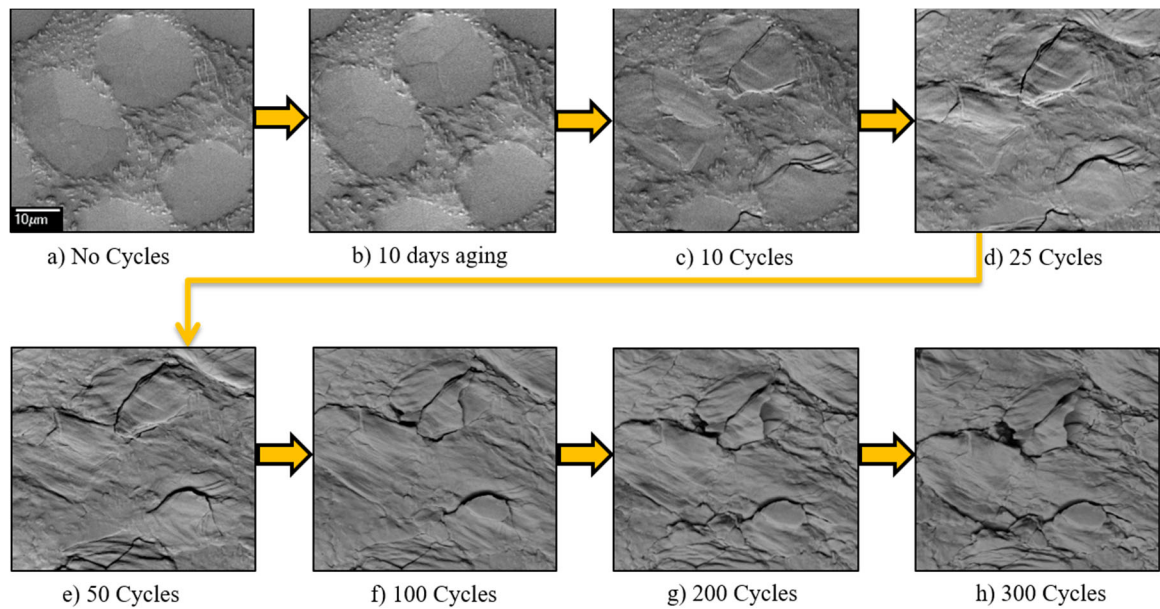


Figure 7.13 Aging Induced Microstructural Evolution of SAC305 with Mechanical Cycling at $T = 25\text{ }^{\circ}\text{C}$ (Region E)

According to the Gibbs-Thompson effect, an increase in particle size is accompanied by a decrease of the solute concentration in the matrix surrounding the particle. This leads to a concentration gradient, and solute atoms near smaller particles will diffuse towards the larger particles where the reductions in solute concentration has occurred. In addition, atoms from the smaller particles will go back into solution. The overall effects are shrinkage of smaller particles, and growth of larger particles. Since Ostwald Ripening is a diffusion-based process, use of higher aging temperatures will increase the rate of coarsening significantly.

In case of aged samples, it was observed that the cycling induced damage consisted primarily of small intergranular cracks forming within dendrites just after the 10th cycles. These cracks continued to grow as the cycling continued, resulting in a weakening of the dendrite structure. In addition, the IMC region became weaker due to the coarsening and coalescing of IMCs during the prior aging. Hence, there are some tiny cracks visible in the IMC region even after 25 cycles in case of region E (Figure 7.13 (d)). These cracks eventually helped the transgranular cracks develop more quickly. In comparison to the non-aged SAC305 samples, the IMC region was less effective pinning and blocking the intergranular cracks. Therefore, it has been found that aged SAC305 samples exhibited more damage compared to the non-aged samples after the same cycling duration. Isothermal aging leads to the degradation of the microstructure of SAC305, which increase the rate of damage accumulation and eventually decreases the fatigue life.

The reduction of the strength of SAC305 after aging can be attributed to 2 major facts. First, aging causes coarsening of the Ag_3Sn and Cu_6Sn_5 intermetallic compounds and hence reduces their ability to block dislocation movements. Second, the β -Sn phase

also coarsens/grows with aging, and hence reduces the strength of the alloy. As a result, a significant difference was evident in the cyclic stress-strain behavior, before and after aging.

Typically, IMC particles tend to become more round (spherical) with aging. Atoms on the surface of a particle have higher energy than the atoms inside a particle due to unsatisfied bonds in the surface atoms. A cylindrical or elongated particle has a higher fraction of surface atoms (or higher surface to volume ratio) compared to a spherical (round) particle with the same volume. As a result, the free energy per atom of an elongated particle or several small particle is higher than a big round particle with equal volume. This free energy difference is the driving force that causes the transition of IMC particles from smaller to a bigger size. This IMC coarsening phenomenon is also known as Ostwald Ripening. According to the Gibbs-Thompson effect [135-137], an increase in particle size is accompanied by a decrease of the solute concentration in the matrix surrounding the particle. This leads to a concentration gradient, and solute atoms near smaller particles will diffuse towards the larger particles where the reductions in solute concentration has occurred. In addition, atoms from the smaller particles will go back into solution. The overall effects are shrinkage of smaller particles, and growth of larger particles. A schematic of the variation of solute concentration around a small and a large particles is shown in Figure 7.14. Since Ostwald Ripening is a diffusion-based process, use of higher aging temperatures will increase the rate of coarsening significantly.

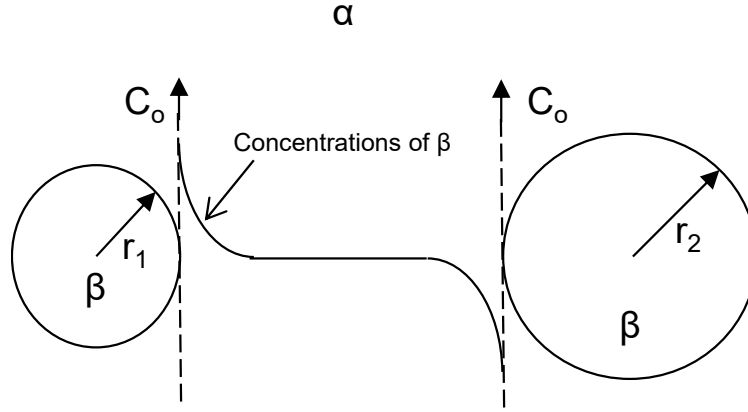


Figure 7.14 Schematic of Solute Concentration in Front of A Small and a Large Particle [135].

The coarsening and coalescing of IMC particles during aging is known to play a critical role in the decrease of solder mechanical properties. IMC particles will pin and block the movement of dislocations. However, aging leads to both a smaller number of larger IMC particles and increased spacing between the IMC particles. The interparticle spacing λ can be calculated from the following equation:

$$\lambda = \frac{4(1-f)r}{3f} \quad (7.1)$$

Where f is the volume fraction of the particles and r is the particle radius. Hence for a fixed volume fraction of the IMC particles, spacing between the particles increases with increasing particle diameter. Orowan proposed a mechanism that when a dislocation crosses incoherent precipitate particles (like the IMC particles in present case), it will bow and leave a loop of stress field around the particle [135, 137, 138]. A schematic of the interaction between a dislocation and IMC particles is shown in Figure 7.12.

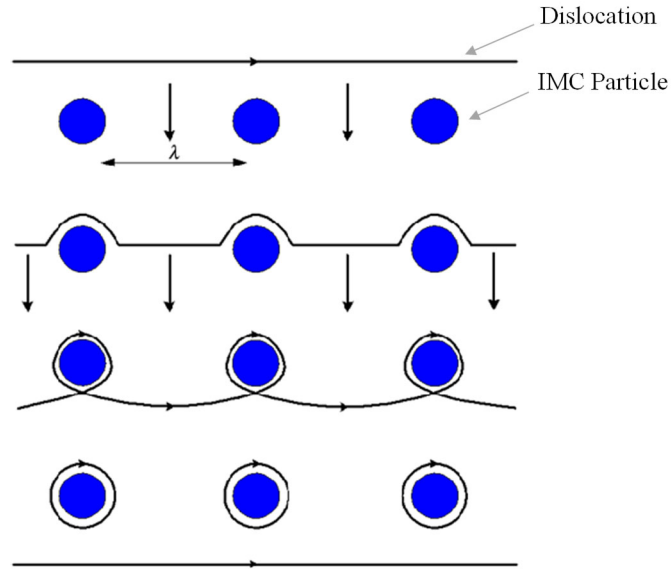


Figure 7.15 Schematic Representation of a Dislocation Passing IMCs (Orowan Looping) [138]

The yield stress and strength of a material depends on the shear stress (also known as the Orowan stress) that is required for a dislocation to pass through the particles. This shear stress will decrease as the inter-particle spacing increases during aging as shown in equation 7.2 [135, 137, 139]

$$\tau_o = \frac{Gb}{\lambda} \quad (7.2)$$

Where τ_o is the shear stress required for a dislocation to pass through the particles, G is the shear modulus, and b is the magnitude of the burgers vector. Therefore, aging leads to the coarsening of the IMC particles which causes to increase the interparticle spacing. Thus, the yield stress and strength of a material decrease after aging due to the increased interparticle spacing.

7.4 Summary and Discussion

In this chapter, an investigation on the effects of aging on the cyclic stress-strain behavior of reflowed SAC305 lead free solders was examined. Using a micro tester machine equipped with a newly designed gripping fixture, uniaxial isothermal cyclic tests were conducted. For preparing the testing specimens, a reflowed (RF) solidification profile was used, which produced microstructures of SAC305 similar to that observed in industrial solder joints. Cylindrical uniaxial test specimens of reflowed SAC305 were formed in circular cross-section glass tubes using a vacuum suction process. Specimens aged at 125 °C for different aging times (0, 1, 2, 5, 10, and 30 days) were subjected to cyclic (tension/compression) mechanical loading at room temperature under strain controlled cycling at different strain levels. After fitting the cyclic stress-strain curves obtained from the tests, the evolution of the cyclic stress-strain behavior with aging has been characterized in terms of peak stress, hysteresis loop, and plastic strain range. It was observed that the hysteresis loop area, plastic strain range, and peak stress of SAC305 decreased while the plastic strain range increased with an increased aging period for any particular strain range. For any particular aging condition, it was observed that the hysteresis loop area, peak stress and the plastic strain range, all increased with an increase in applied strain range.

Plots of initial plastic work accumulated vs applied strain range were then constructed to help facilitate the deduction of initial plastic work at any given aging condition and applied strain range. Such curves would aid in allowing the cycling of different aged SAC305 samples such that the initial plastic work dissipated would remain the same, thus allowing a more comprehensive comparison between the effect of aging on SAC305 lead free solder alloy.

The effects of prior aging on damage accumulation occurring in the microstructure of SAC305 subjected to mechanical cycling was also studied. Uniaxial rectangular cross-sectioned samples were prepared and polished so that the microstructural changes could be tracked after the initial aging, and then subsequently with mechanical cycling. In particular, we examined the microstructural changes that occurred in small fixed regions in the solder samples, rather than using several different regions. Regions of interest near the center of the sample were marked using small indents formed with a nanoindentation system. Samples were then subjected to aging at 125 °C for various durations to produce several different initial microstructures. Scanning electron microscopy (SEM) was used to investigate the aging induced microstructural changes in the regions of interest in the solder sample. After aging, the samples were then subjected to mechanical cycling. After various durations of cycling (e.g. 0, 10, 25, 50, 100, 200, 300 cycles) that were below the fatigue life of the materials, the regions of interest were again examined using SEM. Using the recorded images, the microstructural evolutions in the fixed regions were observed, and the effects of the initial aging on the results were determined. It was found that the number of IMC particles decreased while the average diameter of the particles increases significantly due to the initial aging.

After the cyclic loading was performed on the sample, it was observed that the cycling induced damage consisted primarily of small intergranular cracks forming along the sub-grain boundaries within the β -Sn dendrites. These cracks continued to grow in size as the cycling continued, resulting in a weakening of the dendrite structure, and eventually resulting in the formation of large transgranular cracks. The distribution and size of the intermetallic particles in the inter-dendritic regions were observed to remain essentially

unchanged with the application of the mechanical cyclic load. Relative to the non-aged samples, there were significant differences observed in the rate and intensity of the micro crack growth occurring in the heavily aged samples that began with much coarser microstructures. In particular, there were tiny cracks visible in the eutectic regions even after a small number of loading cycles (e.g. 25-50 cycles). These cracks eventually helped the transgranular cracks develop more quickly. In comparison to the non-aged SAC305 samples, the IMC region was less effective in pinning and blocking the intergranular cracks. Therefore, it was found that aged SAC305 samples exhibited more damage compared to the non-aged samples after the same cycling duration.

CHAPTER 8

EVOLUTION OF THE MECHANICAL PROPERTIES OF INDIVIDUAL LEAD FREE SOLDER BALLS SUBJECTED TO CYCLIC LOADING

8.1 Introduction

Previous studies conducted on the effect of mechanical cycling on the material behavior of lead free solders have been performed on bulk samples subjected to tension and compression. Our goal in this study was to determine the evolution of the mechanical properties of doped lead free solder joints when subjected to mechanical shear cycling. Experiments conducted on actual solder joints would help us gain a better understanding on the real life effects of shear cycling. In this study, we have characterized the mechanical cyclic induced evolution of mechanical properties of individual solder joints. The testing of individual joints is important because there are significant variations of properties between actual joints and conventional bulk samples (e.g. dog-bone, rectangular, or lap shear samples). The test assemblies in this study were (3X3) BGA packages composed of total nine 0.75 mm diameter lead free solder balls which were formulated by reflowing solder spheres soldered onto 0.55 mm diameter Cu pads on FR4 coupons. The solder balls were cycled using an Instron Micromechanical tester along with a newly designed fixture which facilitates the tester to cycle a solder ball individually. The solder balls tested were of different alloys thus also giving an idea of the effect of the addition of various dopants on the mechanical cyclic properties. The balls were cycled for various durations including to failure. Nanoindentation tests were performed on the specimens to study the evolution in mechanical properties (e.g. elastic modulus, hardness and creep strain rate) of the solder balls as a function of duration of cycling. The prime objective of this study was to better

understand the damage accumulation and evolution of a specific solder joint due to mechanical cyclic fatigue loading.

In this chapter, effects of shear mechanical cycling on SAC305 and SAC_Q solder ball joints was investigated. This would help us to understand the effects of fatigue cycling on solder joints in electronic packages and give us an idea of the role played by the effect of added bismuth on the mechanical cycling properties. The evolution of elastic modulus, hardness and creep resistance with various durations of shear mechanical cycling was evaluated with the help of a nanoindentation machine. The change in properties were then compared between the two lead free alloys.

8.2 Experimental Procedure

For this study, SAC305 (96.5Sn-3.0Ag-0.5Cu) and SAC_Q (92.7Sn-3.4Ag-0.5Cu-3.3Bi) lead free solder joints were tested. The joints had a diameter of about 30mils (762 μm). Figure 8.1 shows an example of the test joints used for the experiments. The samples consisted of a 3 x 3 Ball Grid Array (BGA) of the lead free alloys to be tested. The substrates used in the specimens were FR-4 PCBs. Samples which had an Immersion Silver (ImAg) surface finish on the copper pads were chosen as they produced the best shear strength when compared with other surface finishes [143]. Flux was deposited on the pads using a stencil. A reflow profile with a maximum temperature of 245 °C was obtained with the help of a Vitonics-Soltec convection oven. The profile also ensured that the assembly was kept above the liquidus temperature for about 45-60 seconds. The samples were then cleaned, dried and stored in a freezer at -40 °C to hinder any aging effects.

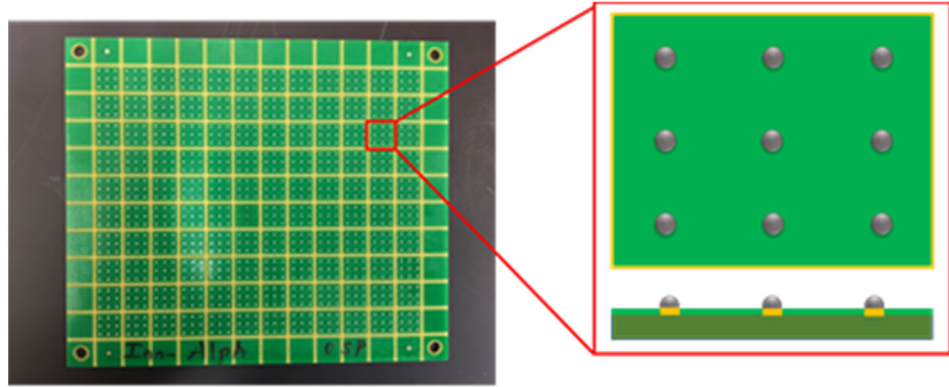


Figure 8.1 3x3 BGA Specimen Sample

Samples were covered with a tape and then confined in epoxy resin. The tape was used to prevent epoxy from entering the space between the individual joints which allows the cycling of each joint separately. The tape from these samples were removed by coarse grinding using a 320 grit SiC paper. Grinding papers up to 1200 grit were then used to reduce the scratches on the samples. The samples were then finally polished a 0.02 μm colloidal silica suspension to flatten the top surface of the solder joints and making the joints suitable for nanoindentation tests. A polished sample encapsulated in epoxy is shown in Figure 2. All the polished joints were viewed under a Polarized Light Microscope to verify that all experiments were conducted on single grain joints.

The Micromechanical Tester shown in Figure 3.20 was used to cycle the individual solder joints. The cyclic mechanical shear loading was conducted with a peak load of 600 gf (equivalent to a stress of about 24MPa). The joints were individually cycled in the shear direction for various durations of 50, 100, 150, 200 and 300 cycles. After every selected cyclic condition, the sample was removed and the evolution in Elastic Modulus, Hardness and Creep Strain Rate of each joint was characterized using the nanoindentation machine (Figure 3.18).

The nanoindentation machine utilized a probe with a Berkovich geometric tip to record the changes in load against indentation displacement. The recorded data was measured in the direction normal to the polished face. At least 5 indents were made on every solder joint for each set of conditions. Through trial and error, it was observed that a peak holding load of 30 mN was ideal for the nanoindentation tests. Using this high load not only prevented creep effects but also ensured that the indents made were deep enough to prevent surface effects while also ensuring these large indents covered all the solder phases present. A schematic depicting all the experimental techniques described above and used for this study has been presented in Figure 8.2.

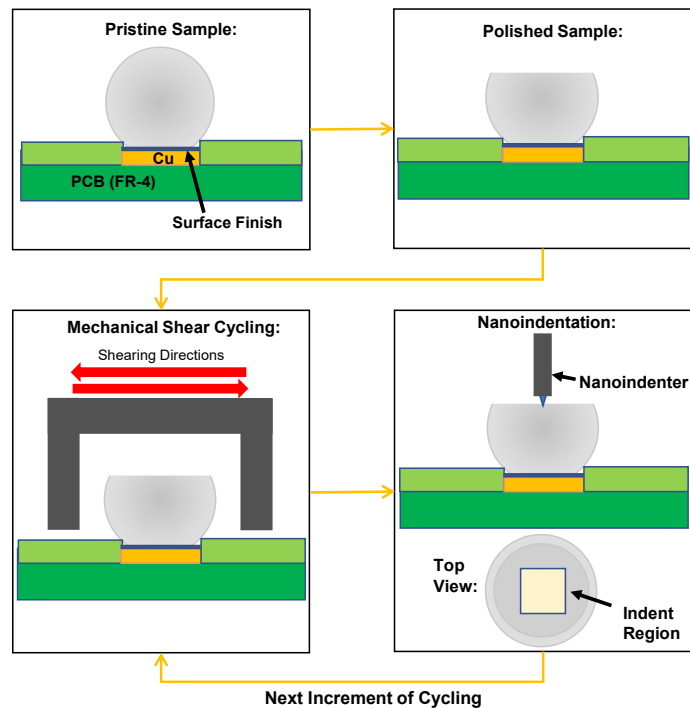


Figure 8.2 Schematic showing the experimental procedure

A trapezoidal loading profile with a peak load of 30 mN held for 30 seconds was used to determine the study the changes in elastic modulus and hardness with mechanical cycling. The hardness was evaluated by dividing the peak load applied by the contact area

of the indenter. Oliver and Pharr proposed a method to obtain the reduced modulus from the slope of the unloading curve of the nanoindentation experiment [129]. As shown in their work, the following equation was then used to evaluate the elastic modulus of the indented material.

$$E = \frac{1 - \nu^2}{\left[\frac{1}{E_r} \right] - \left[\frac{1 - \nu_i^2}{E_i} \right]} \quad (8.1)$$

where E and ν are the Elastic Modulus and Poisson's ratio of the indented material respectively, E_r is the reduced modulus, E_i and ν_i are the Elastic Modulus and Poisson's ratio of the indenter respectively. Oliver and Pharr [129] also demonstrated measurements of elastic moduli of anisotropic materials in their paper. The Hardness was evaluated using Equation 3.12.

8.3 Evolution of Elastic Modulus and Hardness

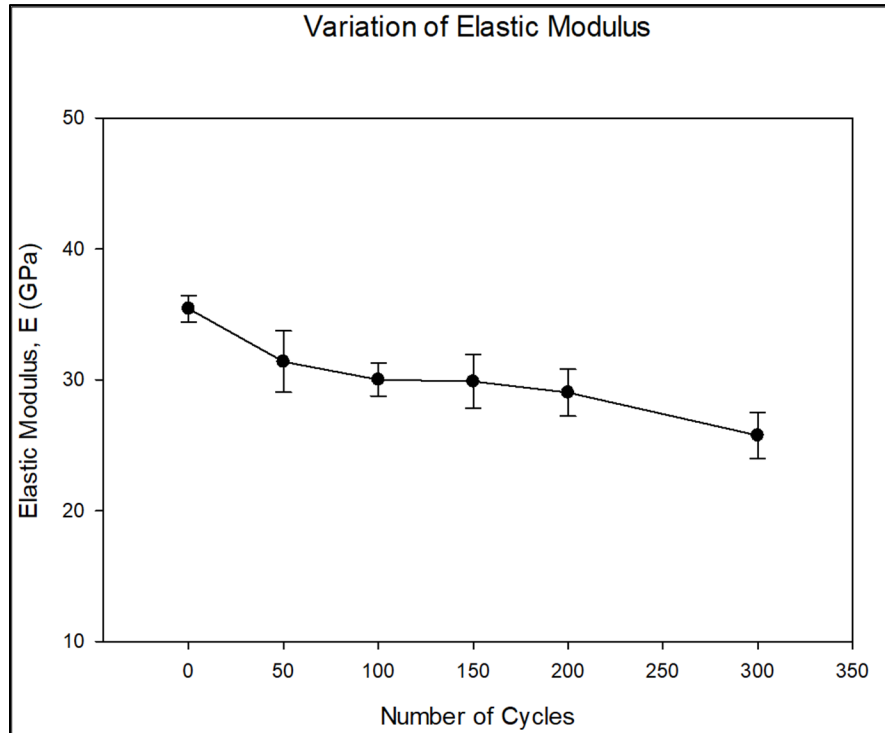
After the solder balls were polished, the elastic modulus and hardness of the solder Balls were obtained using the trapezoidal loading profile. The indents were done in a column to keep track of the indents. Once the initial (0 cycle) indentation tests were performed, the samples were mechanically cycled in shear up to 50 cycles using the Micromechanical tester shown in Figure 3.21 with a peak load of 600 gf. Each Ball was individually cycled. Once the 50 cycles were completed for all of the Balls, the sample was then indented again to measure the changes in the elastic modulus and hardness due to the cycling. This process was then repeated for 100, 150, 200 and 300 cycles. The results of two different solder Balls will be presented here.

It was observed that there was a continuous decline in both elastic modulus and hardness with cycling of the solder balls regardless of the solder alloy. Table 8.1 shows

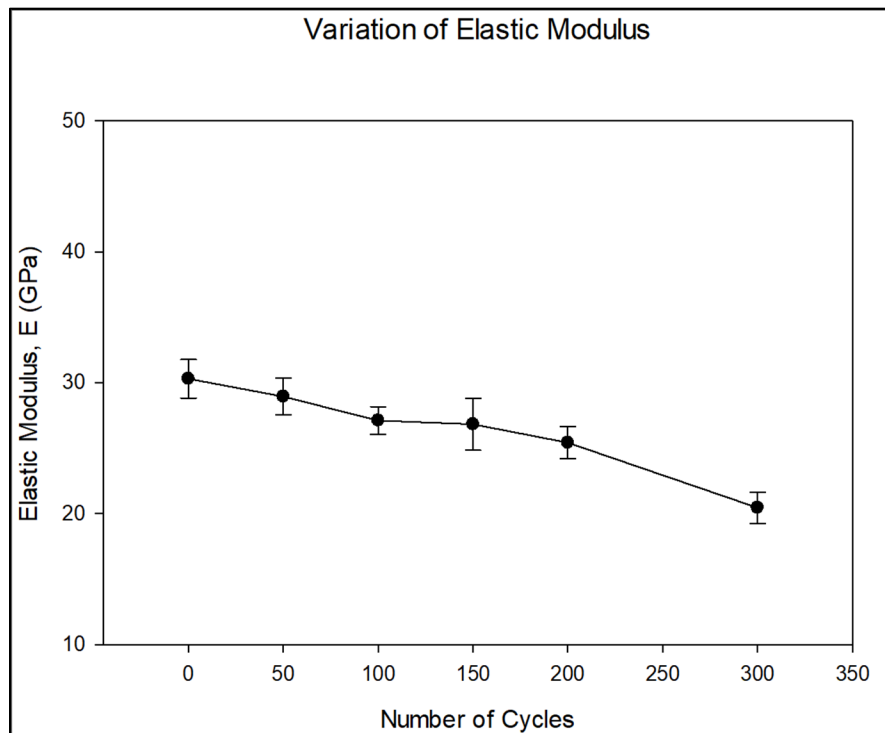
the changes in elastic modulus of two SAC305 Balls (Balls 1 and 2) and two SAC_Q Balls (Balls 3 and 4) with mechanical cycling. Ball 1 exhibited a decline from 35.45 GPa to 25.74 GPa, while Ball 2 exhibited a decline from 30.32 GPa to 20.45 GPa in elastic modulus after 300 cycles. This corresponds to decreases of 27.4% and 32.6%, respectively, for Balls 1 and 2. Figs. 8.3(a) and 8.3(b) illustrate this evolution in elastic modulus with shear mechanical cycling for SAC305 Balls. After 300 cycles, the elastic modulus of Ball 3 exhibited a decline from 43.12 GPa to 24.19 GPa, while Ball 4 exhibited a decline from 38.85 GPa to 24.21 GPa. This corresponds to decreases of 43.90% and 37.68%, respectively, for Balls 3 and 4. Figs. 8.4(a) and 8.4(b) demonstrate the evolution in elastic modulus of SAC_Q Balls with shear mechanical cycling. By comparing Figs. 8.3 and 8.4, we can see that both alloys exhibited the same trend in evolution of elastic modulus with shear mechanical cycling. It was also observed that the decrease in elastic modulus for SAC_Q was greater than that of SAC305 for the same loading condition, although the individual values of the modulus were higher for SAC_Q. This could be due to a higher damage accumulation in SAC_Q than in SAC305.

Table 8.1 Cycling Induced Changes of Elastic Modulus of SAC305 and SAC_Q

Cycling Duration (Cycles)	Elastic Modulus (GPa)			
	SAC305 Balls		SAC_Q Balls	
	Ball 1	Ball 2	Ball 3	Ball 4
0	35.45	30.32	43.12	38.85
50	31.39	28.96	36.64	37.05
100	30.02	27.12	35.63	35.70
150	29.89	26.83	32.82	33.59
200	29.03	25.42	31.16	33.10
300	25.74	20.45	24.19	24.21

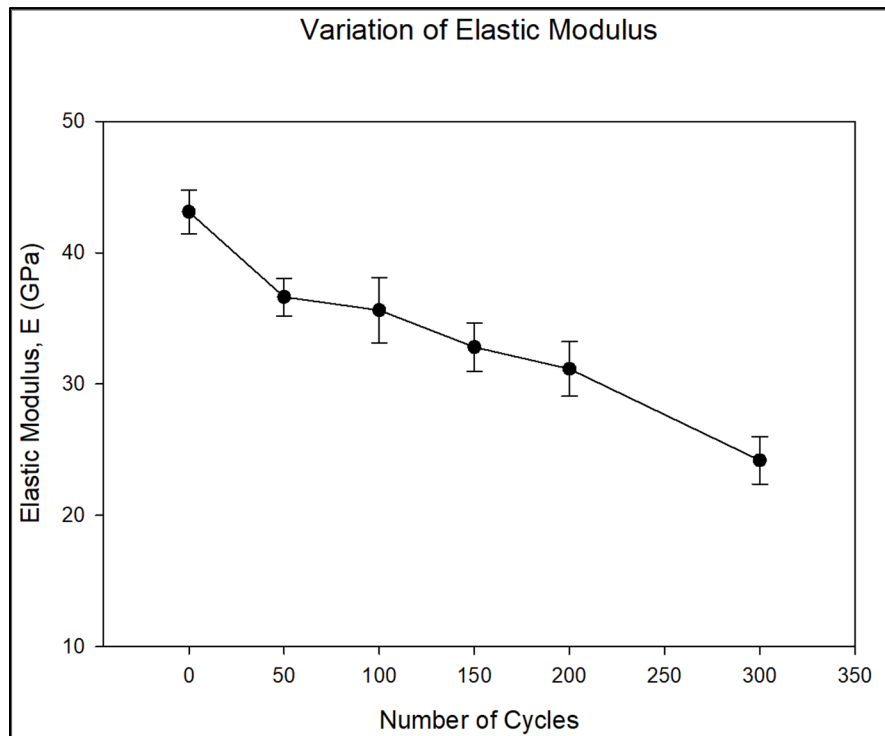


a) Ball 1

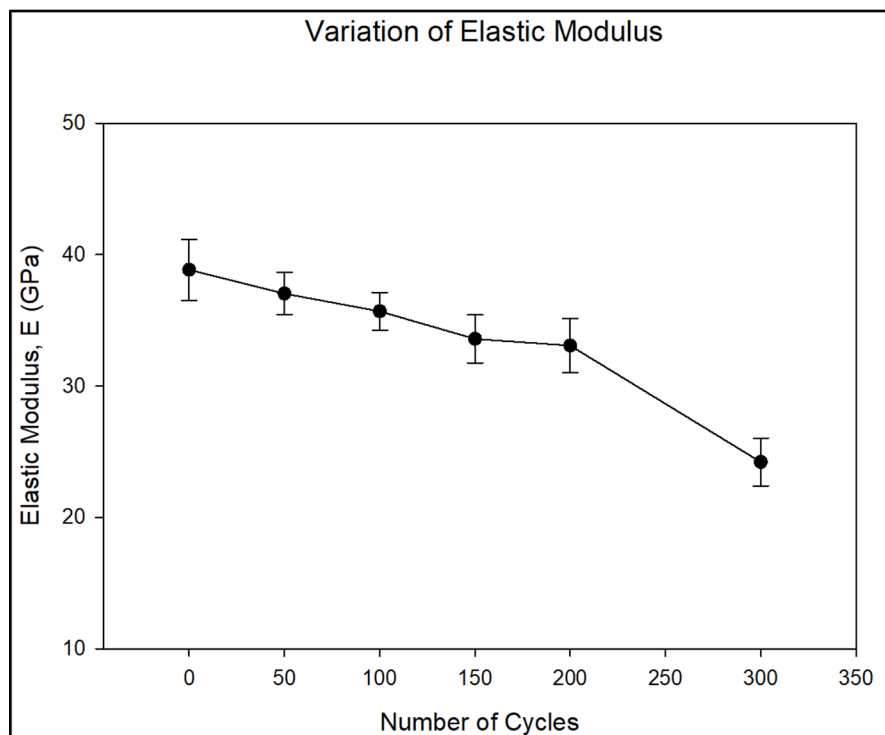


b) Ball 2

Figure 8.3 Variation of Elastic Modulus of SAC305 Balls with Shear Cycling



a) Ball 3



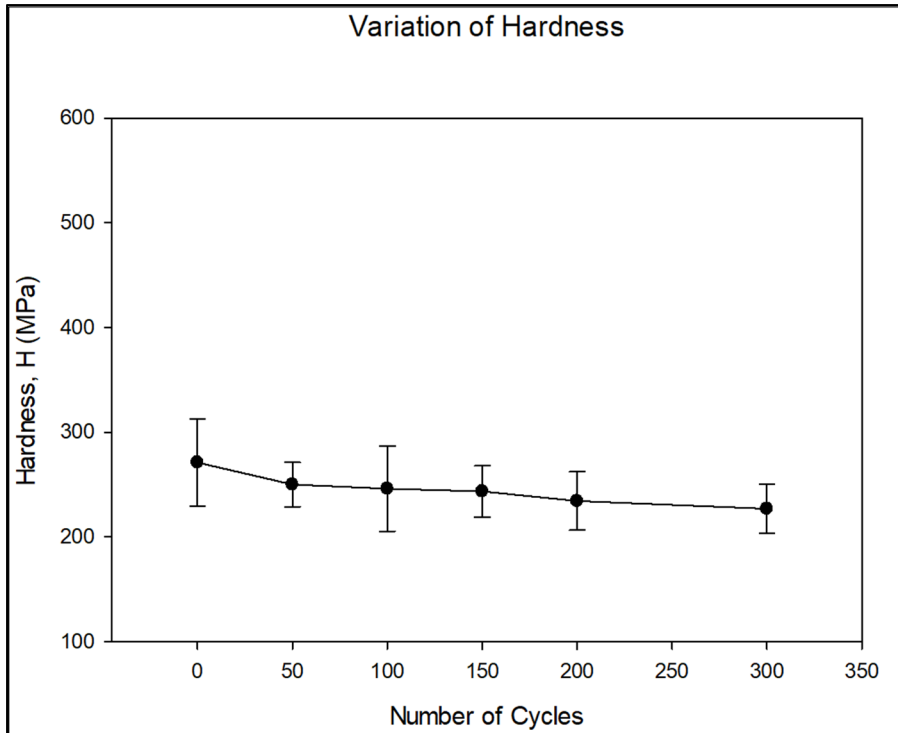
b) Ball 4

Figure 8.4 Variation of Elastic Modulus of SAC_Q Balls with Shear Cycling

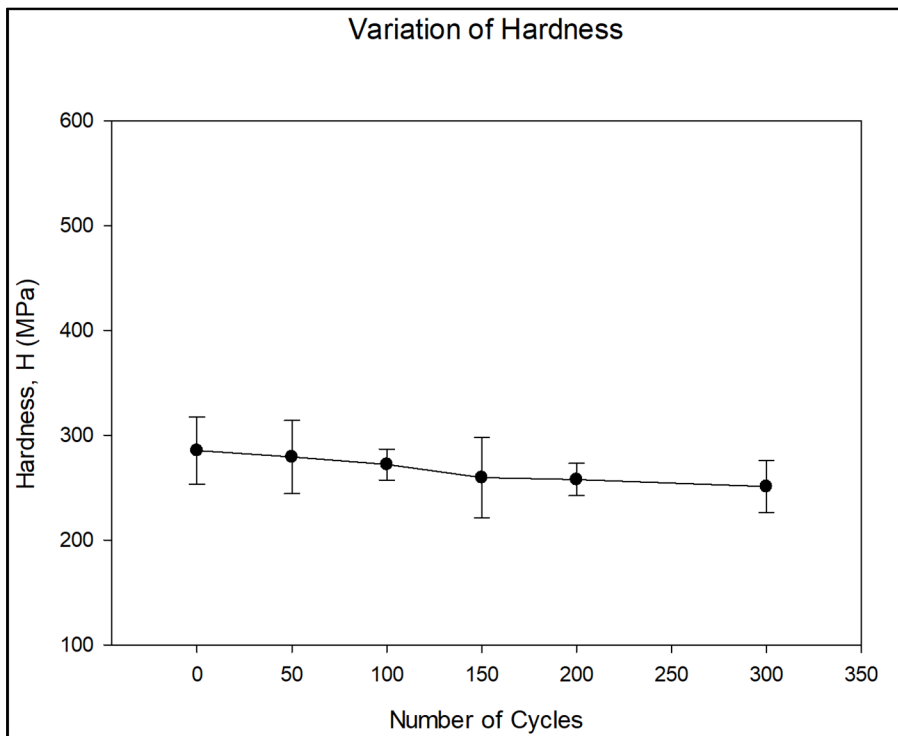
The evolution in hardness of the SAC305 and SAC_Q Balls with mechanical cycling has been summarized in Table 8.2. A 16.29% decline in hardness was observed for Ball 1 while a 12.04% decline in hardness was observed for Ball 2 after 300 cycles. A 19.78 % decline in hardness was observed for Ball 3 while a 15.06 % decline in hardness was observed for Ball 4 after 300 cycles. This evolution in hardness with mechanical cycling have been presented in Figs. 8.5 and 8.6 for SAC305 and SAC_Q, respectively. We once again observe that the hardness values of the SAC_Q Balls were much larger in value, but they presented a much larger reduction in values, possibly due to the larger energy stored with each mechanical cycle.

Table 8.2 Cycling Induced Changes of Hardness of SAC305 and SAC_Q

Cycling Duration (Cycles)	Hardness (MPa)			
	SAC305 Balls		SAC_Q Balls	
	Ball 1	Ball 2	Ball 3	Ball 4
0	270.91	285.32	502.03	478.49
50	249.87	279.19	480.15	455.03
100	245.94	272.05	459.85	445.99
150	243.34	259.53	430.29	434.26
200	234.15	257.71	413.59	428.21
300	226.79	250.98	402.75	406.44



a) Ball 1



b) Ball 2

Figure 8.5 Variation of Hardness of SAC305 Balls with Shear Cycling

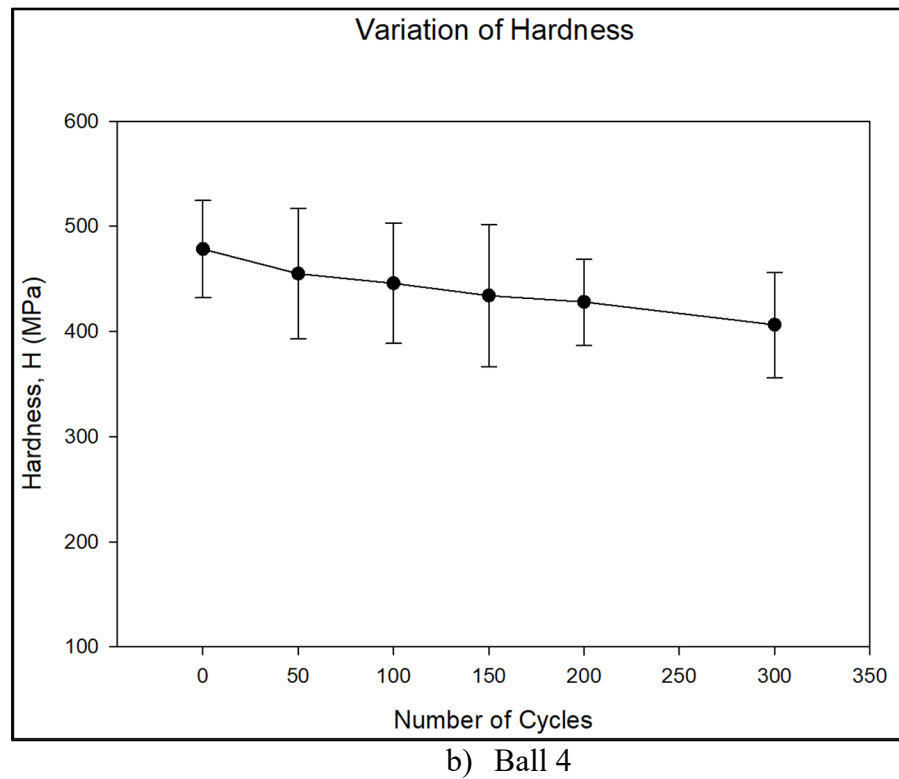
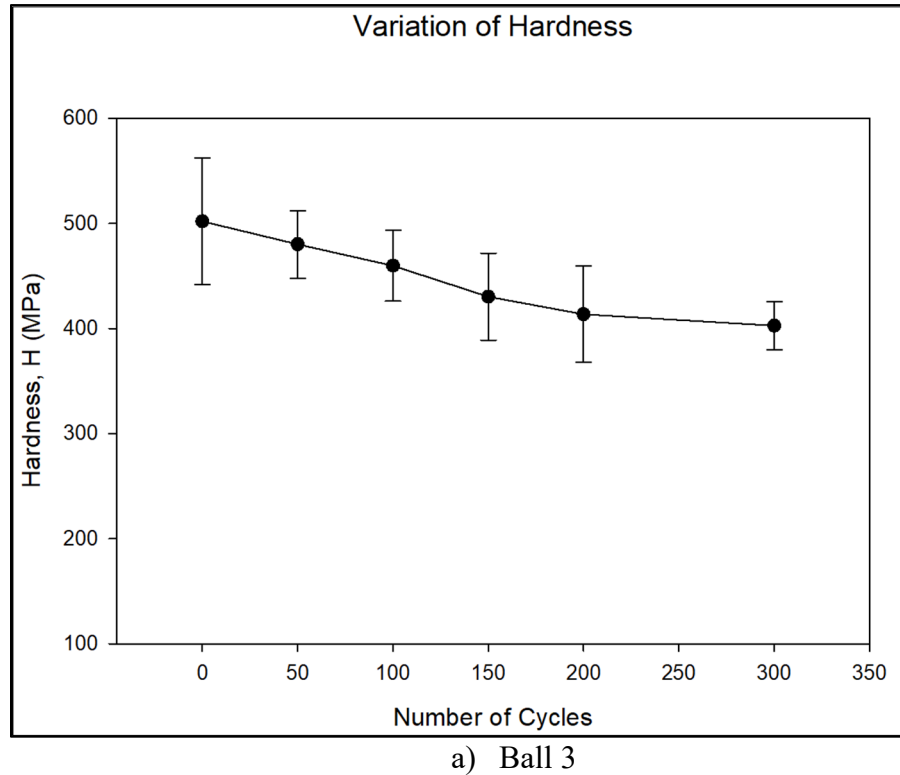


Figure 8.6 Variation of Hardness of SAC_Q Balls with Shear Cycling

8.4 Evolution of Creep Properties

A different set of solder balls were used to investigate the creep resistance change due to shear mechanical cycling for SAC305. A constant 30 mN load with a dwell time of 900 seconds was used to conduct the nanoindentation creep tests. After initial testing (0 cycles), the samples were then cycled for various durations (e.g. 50, 100, 150, 200 and 300 cycles) with a maximum peak force of 600 gf. After each cycling duration, nanoindentation creep testing was performed. Similar to the previous section, only the results of two different balls for both alloys will be presented.

A loading profile with a loading rate of 30 mN/s followed by a steady load of 30 mN (reference segment) was used for the creep nanoindentation tests. The 30 mN load was held for 900 seconds at the holding stage of the profile. The probe was then unloaded at a rate of -6 mN/s. A log hyperbolic model was used to fit the indentation depth vs time data obtained from the creep tests:

$$h = C_1 \ln(1+t) + C_2 \tanh(C_3 t) + C_4 t + C_5 \quad (8.2)$$

where h is the indentation depth, t is the test time and C_1 to C_5 are the fitting constants. The model suggested by Mayo and Nix [140] was used to determine the creep strain rate:

$$\dot{\varepsilon} = \frac{1}{h} \frac{dh}{dt} \quad (8.3)$$

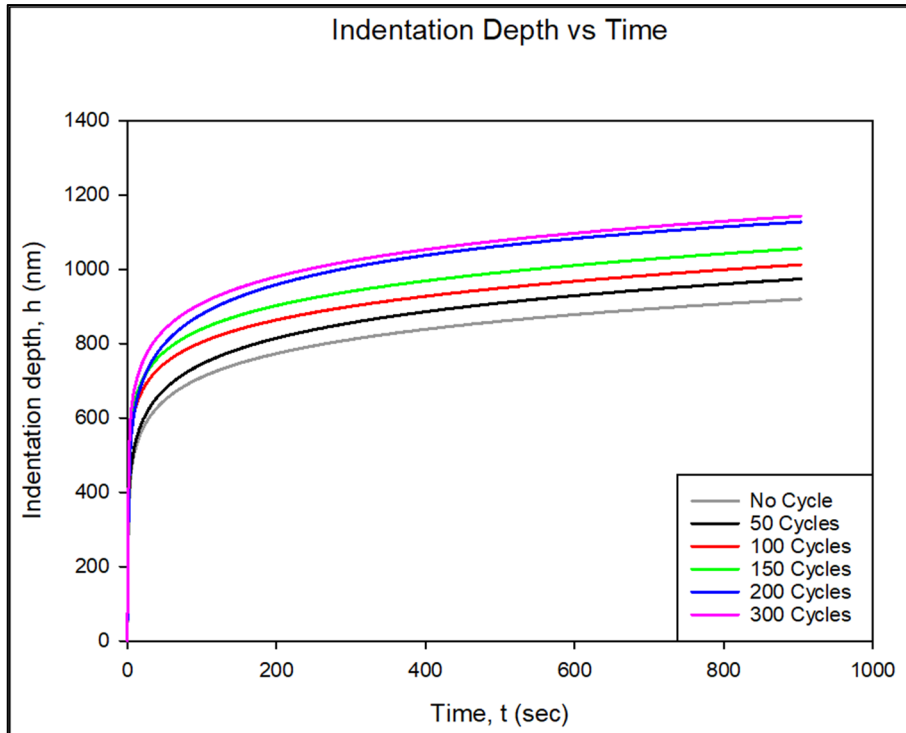
where $\dot{\varepsilon}$ is the creep strain rate. The creep response was then fit using the following exponential model:

$$\dot{\varepsilon} = C_1 e^{C_2 \sigma} \quad (8.4)$$

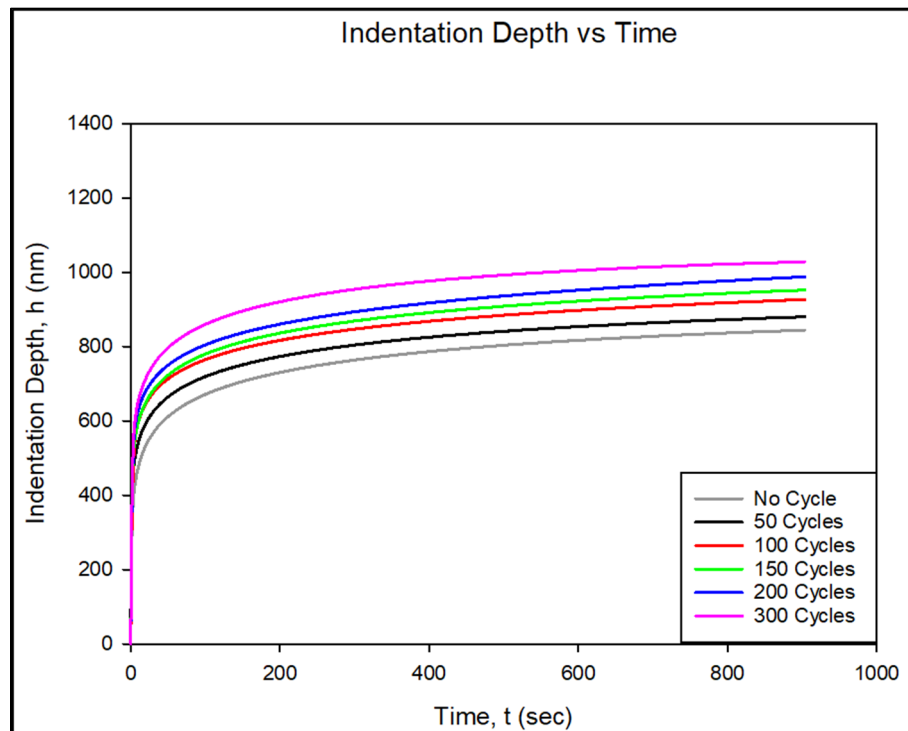
where σ is the stress and C_1 and C_2 are the fitting constants.

Figs. 8.7 and 8.8 illustrate the creep displacement vs. time curves for each duration of prior shear cycling of SAC305 and SAC_Q balls, respectively. The averaged creep data for both cases were fit with equation 8.2 to obtain the indentation depth vs time curve for each duration of cycling. It was recorded that the nanoindentation creep displacement and the total displacement both lead free alloys joints increased with increased number of prior shear cycling. This was observed to be a common trend for all the four joints presented in this paper. The indentation depths achieved with time for SAC305 was observed to be a lot higher than those obtained for SAC_Q as can be seen in the following figures.

The Tabor relationship [130] was used to approximately evaluate the yield stress by dividing the hardness by 3. Figs. 8.9 and 8.10 show the variation of creep strain rate with the applied stress for each duration of prior cycling. The curves in both figures were fit with equation 8.4. From the curves, it is understood that the creep strain rate at a fixed stress level increased with the increased duration of prior mechanical shear cycling for both materials. These increases were approximately 32X for SAC305 ball 5 and 24X for SAC305 ball 6 after 300 cycles for a stress level of 100 MPa. At the same stress level, the increase was about 3X for ball 7 and 9X for ball 8 after 300 cycles. From these observations, we can infer that SAC_Q has a better creep resistance with mechanical cycling than SAC305.

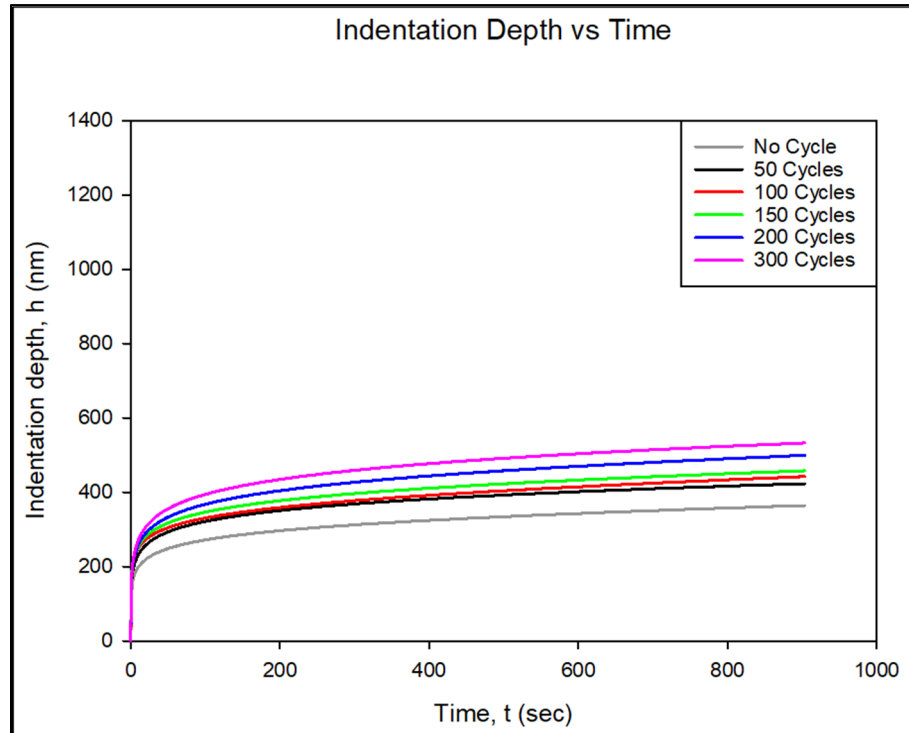


a) Ball 5

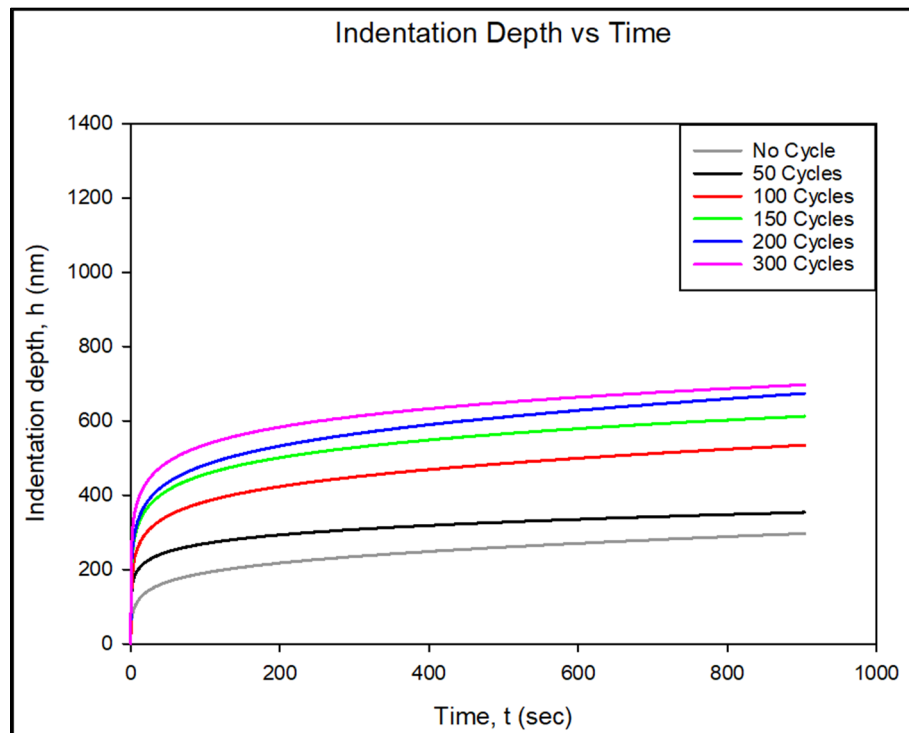


b) Ball 6

Figure 8.7 Creep Displacement vs. Time Curves for SAC305 Solder Balls

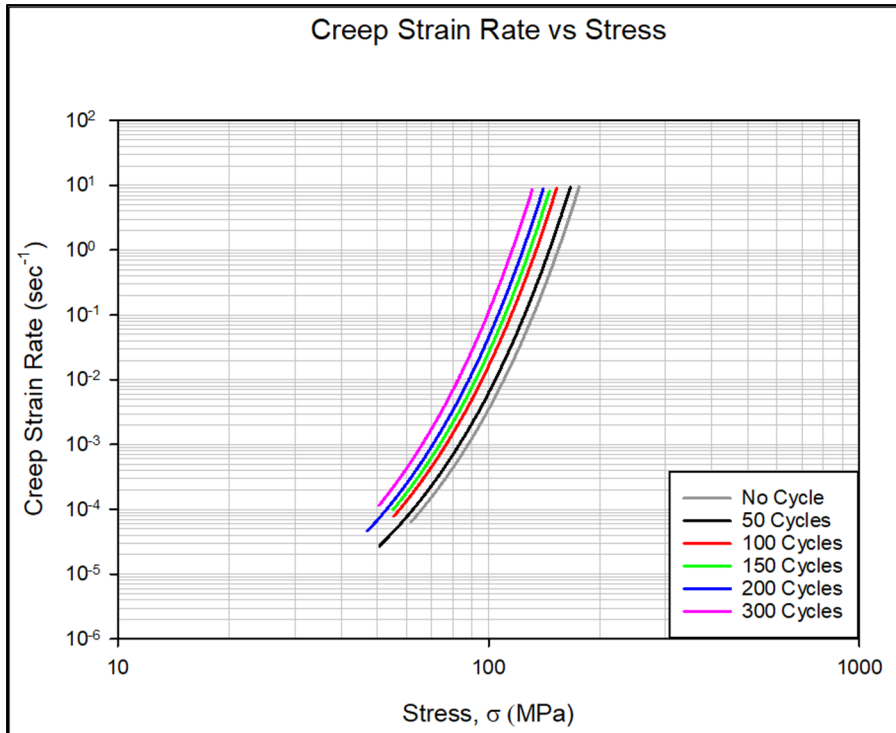


a) Ball 7

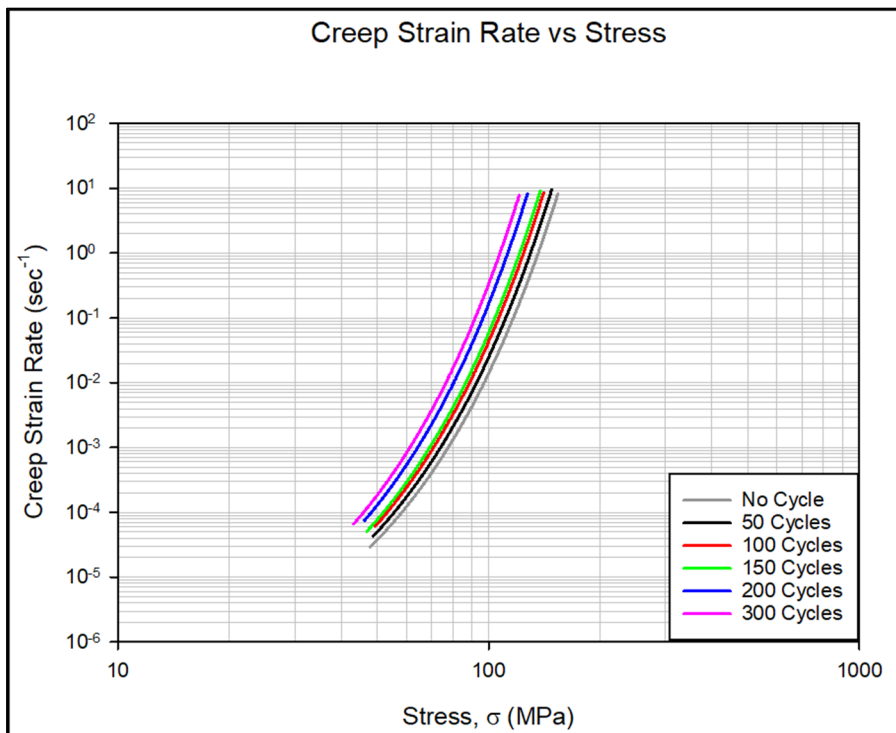


b) Ball 8

Figure 8.8 Creep Displacement vs. Time Curves for SAC_Q Solder Balls

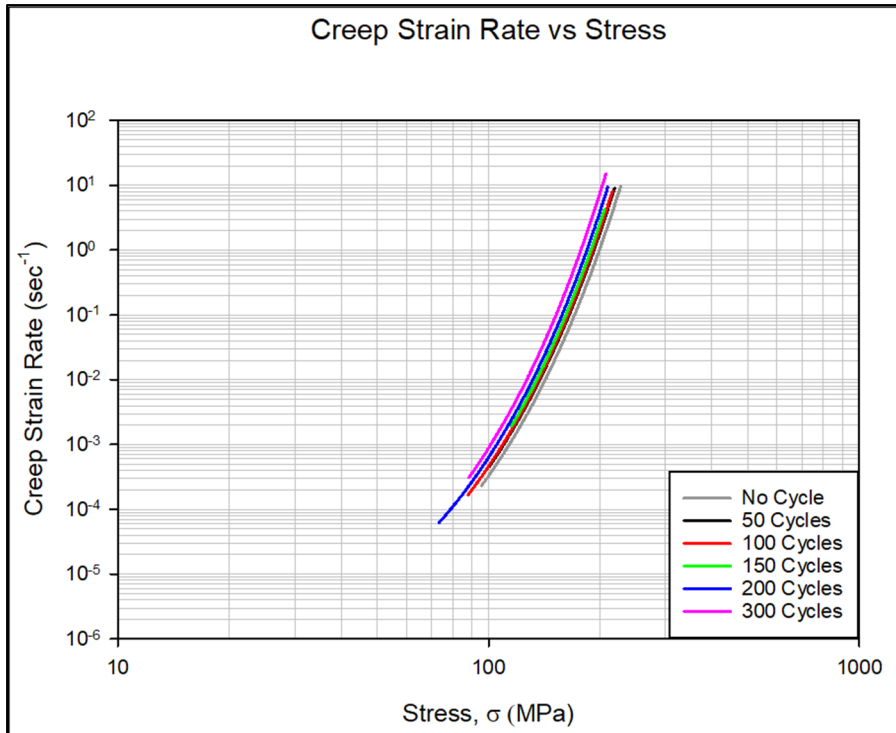


a) Ball 5

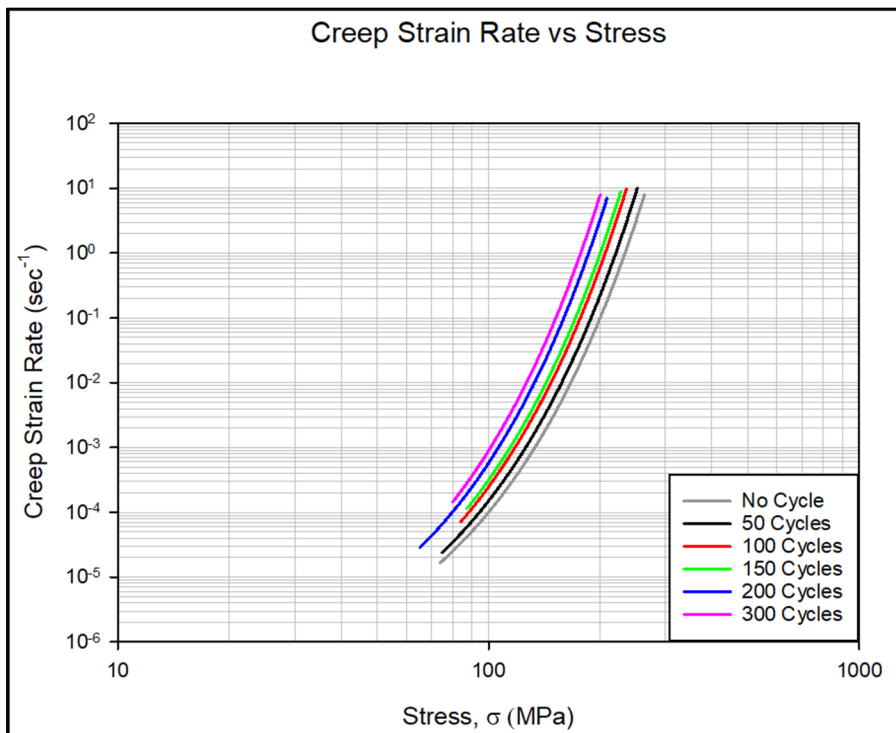


b) Ball 6

Figure 8.9 Creep Strain Rate vs. Stress curves for SAC305 Solder Balls



a) Ball 7



b) Ball 8

Figure 8.10 Creep Strain Rate vs. Stress curves for SAC_Q Solder Balls

It can be inferred from all the above results that mechanical cycling reduces the elastic modulus, hardness and creep strain rate. The mechanical behavior of a solder test can be closely attributed to its microstructure. Any changes in the microstructure of SAC305 and SAC_Q solder with cycling can give us an indication as to why we observe a decrease in its mechanical properties. In a previous chapter, we observed that changes in microstructure of SAC305 and SAC_Q solder uniaxial samples of a fixed region with mechanical cycling. As the cycling progressed, microcracks began developing along the subgrain boundaries of the β -Sn dendrites. With cycling, these cracks grew in length subsequently leading to the creation of larger transgranular cracks. It can thus be deduced that the decrease of the above measured mechanical properties were due to this sort of damage accumulation.

8.5 Finite Element Modeling

A Finite Element Model of the isothermal shear mechanical cycling of individual solder balls was developed in ANSYS APDL. This model was then run with the appropriate loading and boundary conditions. The results that were obtained was then validated with experimental data conducted by another group. This would allow us to estimate the results that would be obtained when run under other loading conditions.

8.5.1 Model Development and Loading Conditions

With the aid of the various cross-sections taken of the specimens, different dimensions of the solder balls were measured using the polarized optical microscope. These dimensions were then used to build and mesh the desired model as depicted in Fig 8.11. A symmetric half model was used for the finite element simulations. SOLID185 elements were used to mesh the entire structure as these elements can be used with a wide

range of nonlinear constitutive models, including hyperelasticity, viscoelasticity, in addition to plasticity and viscoplasticity.

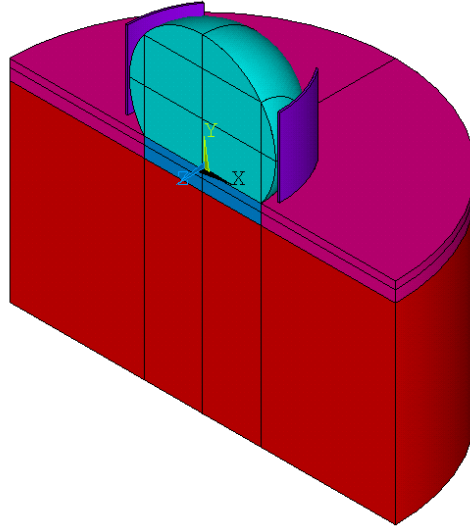


Figure 8.11 3D Symmetric Half Model of the Solder Ball Package

The Anand viscoplastic material model [52] was employed to accurately model the behavior of the SAC305 joints. A set of 9 Anand model parameters for SAC305 was previously evaluated by the authors in reference [45]. These values are tabulated in Table 8.3.

The remaining packaging materials (copper pad, stainless steel shear fixture, solder mask and PCB) were assumed to be linear elastic materials. The directional behaviors of the PCB was included by using orthotropic material constants including temperature dependence. The material constants were measured using stress-strain and CTE (TMA) testing [45], and are tabulated in Table 8.4. All materials were assumed to be homogenous. Process variations and intermetallic growth were not included.

Table 8.3 Anand Parameters for SAC305

Constant Number	Anand Constant	Units	SAC305 No Aging
1	s_0	MPa	21.0
2	Q/R	1/K	9320
3	A	sec ⁻¹	3501
4	ξ	Dimensionless	4
5	m	Dimensionless	0.25
6	h_0	MPa	180000
7	$\hat{S}$	MPa	30.2
8	n	Dimensionless	0.0100
9	a	Dimensionless	1.78

Table 8.4 Table of Material Properties

Material	Elastic Modulus (GPa)	Shear Modulus (GPa)	Poisson's Ratio	CTE [1/°C]
Copper Pad	129.0		0.34	16.3 x 10 ⁻⁶
Stainless Steel	200		0.30	24 x 10 ⁻⁶
Solder mask	3.10		0.30	30 x 10 ⁻⁶
PCB	19.30 (xy, -40 C)	8.71 (xy, -40 C)	0.11 (xy)	14.5 x 10 ⁻⁶ (xy)
	13.20 (xy, 125 C)	5.95 (xy, 125 C)		
	8.48 (z, -40 C)	3.80 (xz, yz; -40 C)	0.39 (xz, yz)	67.2 x 10 ⁻⁶ (z)
	5.84 (z, 125 C)	2.60 (xz, yz; 125 C)		

A constant displacement mechanical cyclic loading condition was applied to the model and studied. The stainless steel shearer was displaced by a range of ± 2.95 mm at a displacement rate of about 11.75 mm/s. The package assembly was assumed to be in a stress-free state at 25 °C (room temperature), with no initial residual stresses/deformations induced in the manufacturing process. The samples were also simulated to have been cycled at room temperature. In all of the models, it was found that the cycle to cycle variation of the solder stress/strain behavior became stable by the third cycle. The values

after the 5th cycle were extracted to evaluate the dissipated plastic work per cycle (ΔW) that was used to estimate fatigue life. The half model was appropriately constrained along the axes of symmetry, so that for a node in a symmetry plane, the displacement component perpendicular to the symmetry plane was required to be zero. Perfect bonding was assumed at all interfaces between different materials. Iterative solution procedures were used due to the nonlinear material and kinematic (large deformation) characteristics of the model.

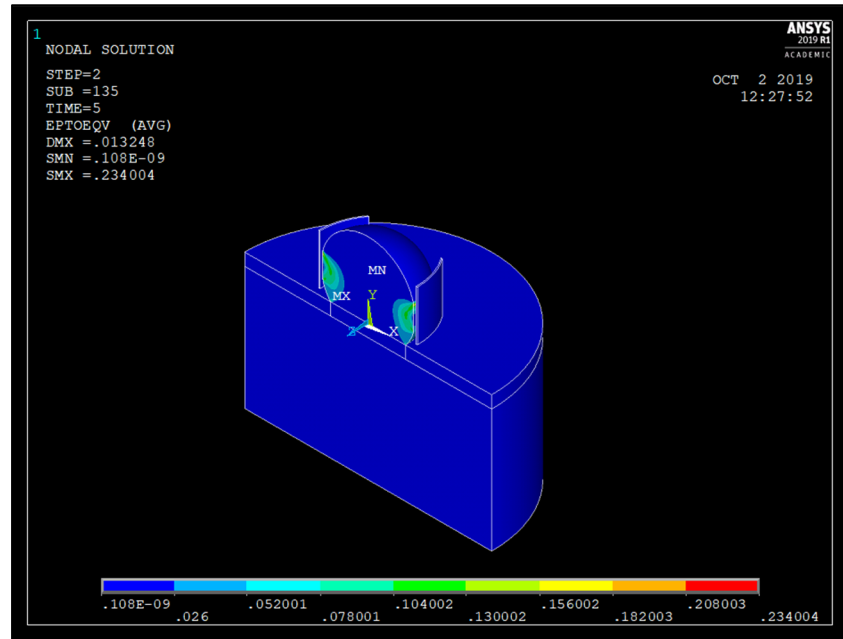
8.5.2 Finite Element Results

In this section, the results obtained for both the loading conditions will be presented and discussed. The plastic work accumulated after 5 cycles was extracted from the FEA models. This was then compared with the experimental data obtained [141]. From the above reference, it was observed that the characteristic fatigue life for SAC305 balls for the specified load (≈ 24 MPa) was about 781 for the ImAg surface finish and about 491 for the ENIG surface finish. The strain contours obtained after 5 cycles is illustrated in Figure 8.12. The maximum strain was observed to have occurred on the lower interface of the solder ball and the copper pad and it was observed that the solder balls did indeed fail at about that region. To minimize the mesh dependency of the calculated plastic work values in the balls, it is conventional to calculate volume averaged values across a set of N elements along the crack path using the relationship:

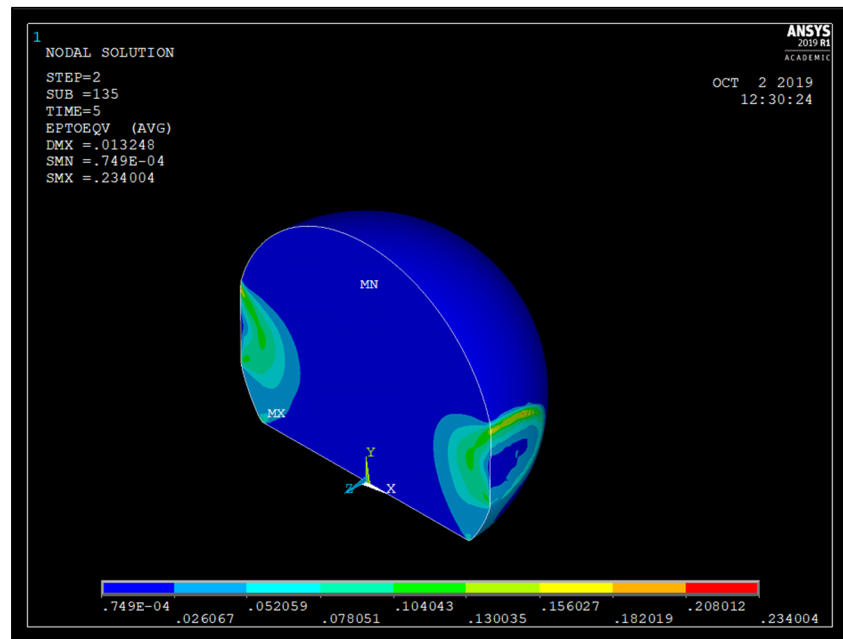
$$(W_{pl})_{Averaged} = \frac{\sum_{i=1}^N (W_{pl})_i V_i}{\sum_{i=1}^N V_i} \quad (8.5)$$

where $(W_{pl})_i$ and V_i are the elemental values of the accumulated plastic work and Volume. By considering only the critical region of the bottom layer of the solder ball, the average

plastic work accumulated per cycle for the solder ball, ΔW , was calculated using eqn. 8.5 to be approximately 0.45 MJ/m^3 .



(a) Entire Sample



(b) Solder Ball

Figure 8.12 Strain Contours Obtained after Isothermal Shear Cycling

Darveaux [142] developed a life estimation methodology for solder joints based on the energy dissipation (ΔW). The procedure uses a pair of power law type relations for estimating crack initiation and crack growth rate in solder joints:

$$N_i = K_1 (\Delta W)^{K_2} \quad (8.6)$$

$$\frac{da}{dN} = K_3 (\Delta W)^{K_4} \quad (8.7)$$

where N_i is the number of thermal cycles to crack initiation, da/dN is the crack growth rate (assumed constant) occurring after crack initiation, ΔW is the energy dissipation per cycle in the solder sample (e.g. critical solder ball), and K_1 , K_2 , K_3 , and K_4 are material constants. Once the crack location and path are known in the solder joint, the number of cycles to failure can be estimated using:

$$N_f = N_i + \frac{a_c}{\left[\frac{da}{dN} \right]} \quad (8.8)$$

where N_f is the number of cycles to failure, a_c is the length of the fully developed crack at failure/fracture, and N_i and da/dN are calculated from ΔW using Eqs. (8.6-8.7). Values of K_1 and K_2 for SAC305 solder have been determined using uniaxial (mechanical) fatigue testing of SAC305 samples [45]. Calculation of parameters K_3 and K_4 was also conducted previously [143]. The values of these constants have been tabulated in Table 8.5. The crack length path measured for the isothermal mechanical cycling of these solder balls was measured to be approximately 0.78 mm.

Table 8.5 Failure Model Material Constants

Aging Time	Board Finish	K ₁	K ₂	K ₃	K ₄
No Aging	ImAg	37.97	-2.8	0.027	3.08
	ENIG			0.038	3.41
	ENEPIG			0.033	3.05

Notes: Units of K₁ and K₃ are (Cycles/MPa^{K₂}) and (mm/Cycle-MPa^{K₄})

By using the values of the constants and the value of ΔW obtained from the finite element simulation in Equations 8.6, 8.7, and 8.8, we can estimate the fatigue life of the solder balls. These values have been tabulated in Table 8.6. By taking a look at the values in Table 8.6, the finite element model was able to fairly deduce the fatigue life for the ImAg board finish but needs refinement to estimate the fatigue life for the ENIG board finish.

Table 8.6 Correlation of Finite Element Estimations and Experimental Data

Board Finish	Experimental Fatigue Life	Estimated Fatigue Life	% Difference
OSP	694	-	-
ImAg	781	694	11
ENIG	491	669	36

8.6 Summary and Discussion

In this study, we have explored the effect of shear mechanical cycling on damage accumulation in SAC305 and SAC_Q solder joints in a 3x3 BGA sample. Such studies are needed to help improve fatigue models for lead free solders. The solder balls were cycled individually with a peak load of 600 gf by a Micromechanical tester for various durations (50, 100, 150, 200, 300 cycles), all of which were below the fatigue life of these joints. After each cycling duration, nanoindentation experiments were conducted on the samples to test for changes in elastic modulus, hardness, and creep response. It was

observed that the elastic modulus decreased by about 27-32% while the hardness reduced by about 12-16% after 300 cycles for SAC305. SAC_Q joints experienced a decline of about 37-43% in elastic modulus and a decline of about 15-20% in hardness after 300 cycles. Although the values of elastic modulus and hardness of SAC_Q were greater than that of SAC305, the larger decline in values can be attributed to the larger amount of damage accumulation in SAC_Q. The creep properties of the lead free solder joints also decreased with shear mechanical cycling. It was seen that for a stress level of 100 MPa, the creep strain rate increased by about 24X to 32X after the end of 300 cycles for SAC305. On the other hand, SAC_Q experienced an increase of about 3X to 9X after 300 cycles at the same stress level. It can thus be deduced that SAC_Q has better creep properties than SAC305 after undergoing shear mechanical cycling. We have also seen that with the cycling of lead free solder samples, small intergranular cracks form. These cracks tend to grow along the subgrain boundaries and increase in length with cycling, thus weakening the β -Sn dendrites and finally leading to the creation of larger transgranular cracks. A finite element model was also developed to estimate the fatigue life of SAC305 solder balls when subjected to isothermal shear cycling. The model had a fairly good agreement with the ImAg board finish but needed more improvement for the ENIG board finish. The model, however, was able to estimate the failure regions accurately and can be developed further to help deduce the fatigue life of solder balls when subjected to shear cycling under other loading conditions.

CHAPTER 9

EFFECTS OF MECHANICAL CYCLING IN SHEAR ON LEAD FREE SOLDER JOINTS

9.1 Introduction

Previous studies conducted on the effect of mechanical cycling on the material behavior of lead free solders have been performed on bulk samples subjected to tension and compression. The goal in this study was to study the load-displacement curves and the microstructure of lead free solder joints when they are subjected to mechanical shear cycling. Experiments conducted on actual solder joints would help us gain a better understanding on the real life effects of shear cycling. The test assemblies in this study were (3X3) BGA sandwiched SAC305 solder joints which were about 0.75 mm diameter lead free solder balls which were formulated by reflowing solder spheres soldered onto 0.55 mm diameter Cu pads on FR4 coupons. All the solder joints were cycled simultaneously using the MT-210 micro-tester along with a newly designed fixture. The load-displacement curves obtained were recorded and studied. For the microstructural portion of the study, the specimens were initially held in epoxy preforms and polished to expose the solder joints. The samples were then extracted and some of these were aged at 125 °C for 10 days. Both the aged and non-aged specimens were then isothermally mechanically cycled at either room temperature or at 100 °C. The differences in the microstructure due to mechanical cycling of the joint under different conditions, namely, aging, or high temperature mechanical cycling, or a combination of both were then observed. The micrographs before and after the cycling were captured by an optical microscope. The other objective of this study was to also observe if recrystallization played

a part in the ultimate failure of the joints when subjected to shear mechanical cycling for each of the conditions. Finally, a finite element model was built to simulate the experiment and the results were compared with actual data.

9.2 Experimental Procedure

For this study, sandwiched SAC305 solder joints were tested. Both aged and non-aged solder joints were used here. Each sample that was used in this study consisted of 9 reflowed SAC305 sandwiched joints arranged in a 3x3 Ball Grid Array (BGA). The substrate in the samples were FR-4 PCBs. The joints in consideration had an average diameter of about 30 mils (approximately 762 μm) and reflowed on to copper pads with a nominal diameter of 20 mils (approximately 550 μm). The ball-to-ball pitch was about 3 mm. The average height of the joints was approximately 0.5 mm while the specimen had an overall height of about 2.5 mm. Fig. 9.1 represents a sample specimen used in these experiments. An Immersion Silver surface finish was applied on the specimens. The samples were stored in a freezer at a temperature of -40 °C to hinder any aging effects.

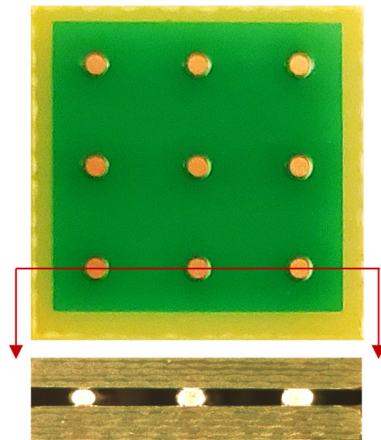


Figure 9.1 SAC305 BGA Samples (3 x 3 Arrays)

To observe changes in the microstructure of the SAC305 joints with mechanical cycling, the samples were first inserted into slots created in epoxy preforms. One such

sample fit inside an epoxy preform is shown in Fig. 9.2. The slot created in the epoxy preforms had a similar thickness to that of the specimens to ensure that a tight fit is created for even polishing. This method would allow us to not only evenly support the sample for polishing, but also be able to extract the sample after polishing. The specimens were polished on either sides to verify that the load distribution on the sample would be equal on both sides.

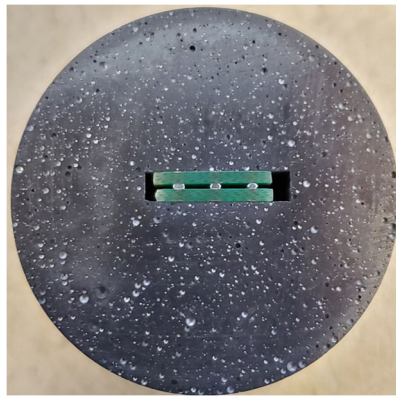


Figure 9.2 Polished SAC305 Joints in Slotted Epoxy Preforms

The polished sample was then extracted from the epoxy resin and cycled using the microtester shown in Fig. 3.7. The thermal chamber visible in Fig. 3.7 was used to maintain an isothermal environment to conduct the shear mechanical cycling. Fig. 3.26 shows a set of newly designed fixtures used to grip and cycles these BGA samples. The sample is tightened using screws at various locations to ensure the cycling is uniform. The left end of the microtester is attached to the linear actuator that allows the arm to move back and forth while the right end of the microtester is held stationary. The load cell is attached to the stationary arm of the microtester. The samples were cycled in a shear direction until failure was noticed. The polarized optical microscope presented in Fig. 3.16 was used to observe changes in the grain morphology of the solder joints when subjected to isothermal

mechanical cycling. The microstructures were recorded after selected cyclic intervals. Any aged samples that were used in this study were aged in an environmental chamber for 10 days at a temperature of 125 °C.

9.3 Evolution of Mechanical Cycling Load-Displacement Curves

Fresh SAC305 BGA Joints were polished in the methods described in the previous section. Some of these samples were then aged for 10 days at a temperature of 125 °C in a thermal oven. All the samples were initially studied under the optical microscope to record the initial microstructure and to observe the number of grains and grain orientation differences. The specimens were then cycled at stage speed of 0.002 mm/s at a displacement range of ± 0.018 mm. After every 200 cycles, the samples were again viewed under the optical microscope to track any changes in the microstructure and grain morphology due to the isothermal shear mechanical cycling of these joints. These steps were repeated until the failure of the samples. As the cyclic tests were conducted at a constant displacement condition, the plastic displacement range, (length of the displacement axis intercepts), was approximately the same for all the tests.

Fig. 9.3 represents a force-displacement graph typically obtained when non-aged specimens were mechanically cycled at room temperature. Initially, the samples experienced a hardening phenomenon in which the resistive load of the specimens actually increased with the progress in cycling. This subsequently increased the magnitude of the hysteresis loop area as well. This phenomenon continued up to the 400th cycle, beyond which, the peak stress and the loop area began decreasing until the average fatigue life of approximately 1620 cycles was reached.

The graph shown in Fig. 9.4 was obtained after cycling the 10 days aged joints at room temperature. Unlike the previous case, only a cycling softening phenomenon was observed here, where the loop area and the peak stress both decreased as the cycling progressed. The average failure life obtained for 10 days aged joints was about 940 cycles.

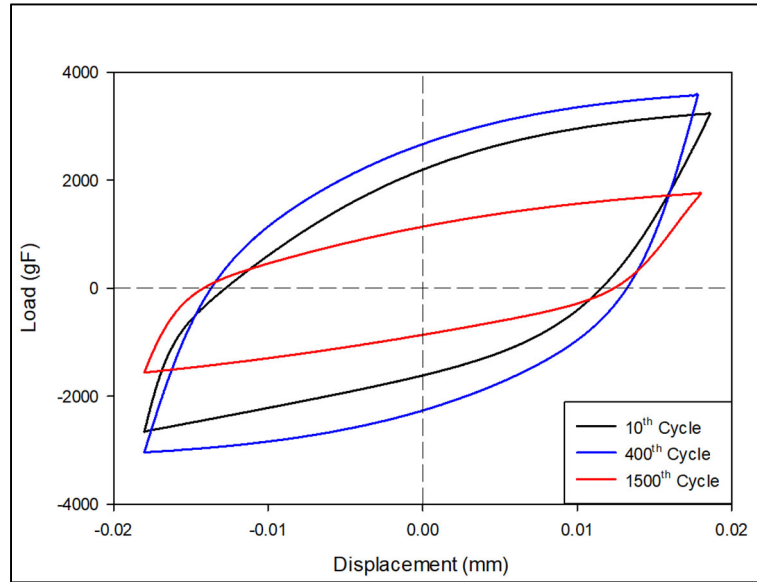


Figure 9.3 Typical Force-Displacement Graph for Non-aged Samples Cycled at 25 °C

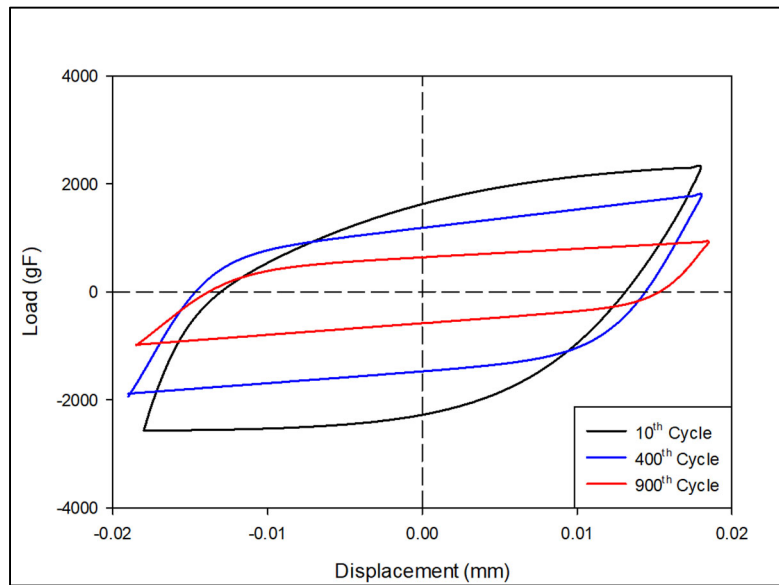


Figure 9.4 Typical Force-Displacement Graph for Non-aged Samples Cycled at 25 °C

9.4 Evolution of Microstructure of SAC305 Joints with Mechanical Cycling

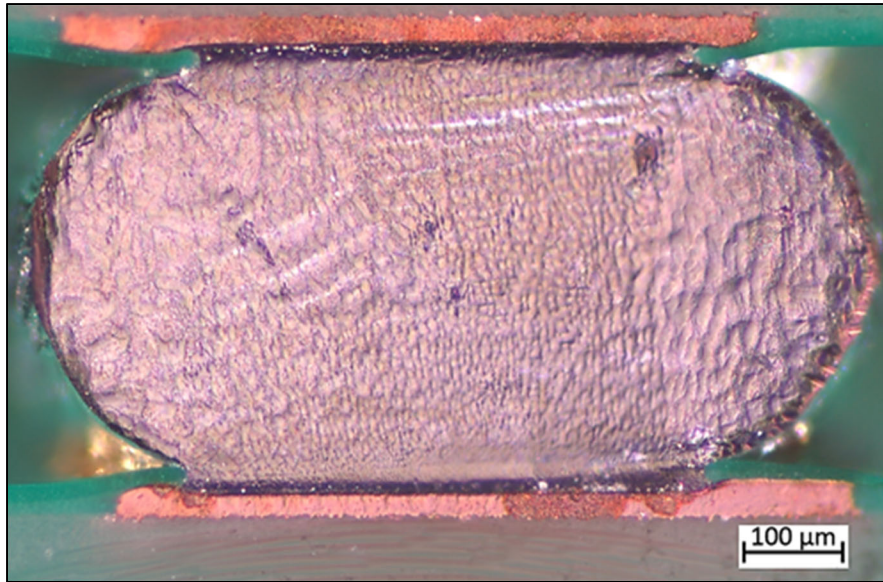
9.4.1 Non-aged Joints Cycled at Room Temperature (25 °C)

The series of micrographs presented in Fig. 9.5 depicts the evolution of the microstructure in a polished corner SAC305 non-aged sandwiched solder joint when mechanically cycled at room temperature. Each joint was polished such that the Beta-tin dendrites were exposed. Eutectic regions containing Ag_3Sn and Cu_6Sn_5 intermetallic particles enclose the beta-tin dendrites. The intermetallic particles help to strengthen the solder joint by obstructing dislocation motion.

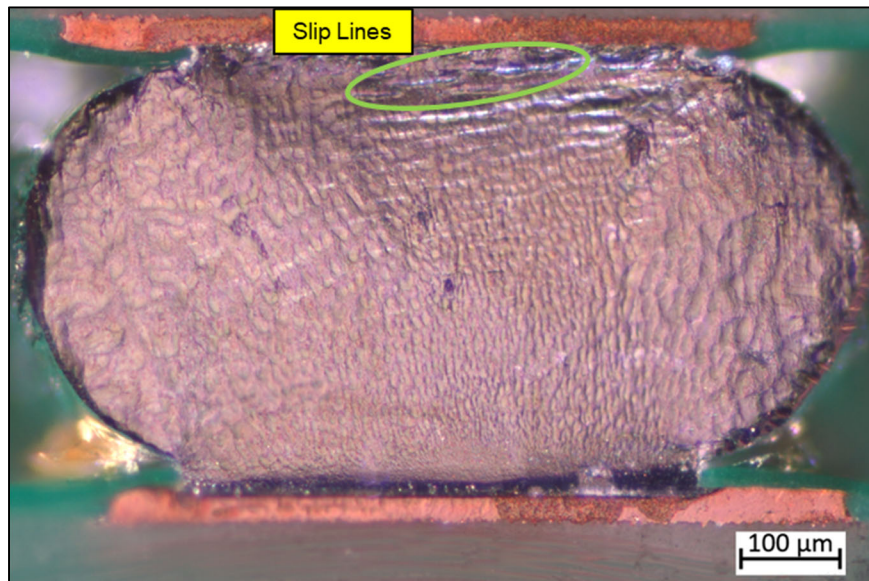
We can observe that the joint is a single grained joint from the initial micrograph. No prominent changes were noticed in the first 600 cycles. Slip lines were visible in the micrograph after about 800 cycles as observed in Fig. 9.5(b). One of these slip lines has been circled in the figure as well. With the progress in cycling, the slip lines were much more noticeable and damage along these slip lines became more severe with cycling. The changes continued prior to the crack propagation stage. Some intrusions and extrusions were also noticed in the microstructure after 1200 cycles as highlighted in Fig. 9.5(c).

Zoomed in images of cracks occurring at the corner of two different solder joints has been presented in Fig. 9.6. The images were taken after about 1600 shear cycles. By comparing the cross-polarized optical micrographs with its corresponding bright-field micrographs, it was determined that the different colors introduced in the optical images were due to the uneven surface of the joint and not brought about by any recrystallization phenomenon. In addition, the authors did not observe any indication of the formation of new grains along the crack path as is evident from the series of images in Fig. 9.6. The images also point out the initiation of cracks from around the copper pad region. This is

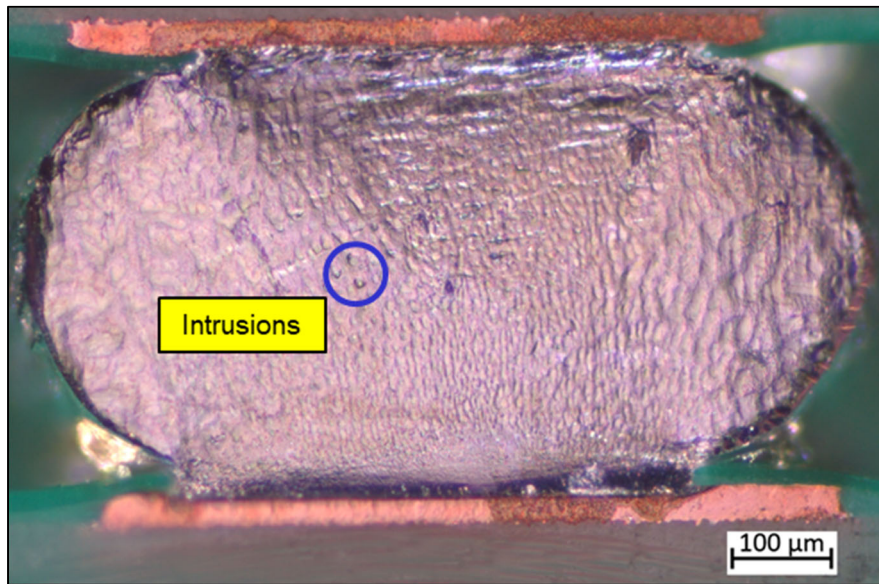
due to the accumulation of plastic strain mainly around these regions and thus most of the cyclic softening phenomenon will be experienced in these areas. However, the crack propagation phase was noticed to span over a very short period of the whole fatigue life. This is unlike the case observed for thermal cyclic loading, where the crack propagation stage occupies most of the fatigue life [144].



a) Initial Microstructure

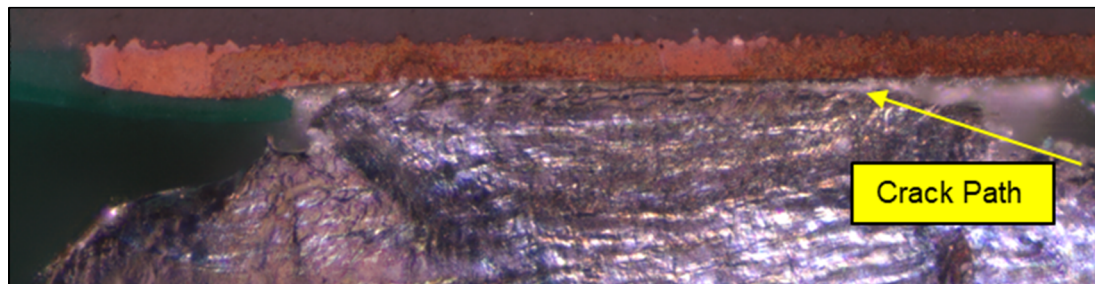


b) Microstructure after 800 Cycles at 25 °C

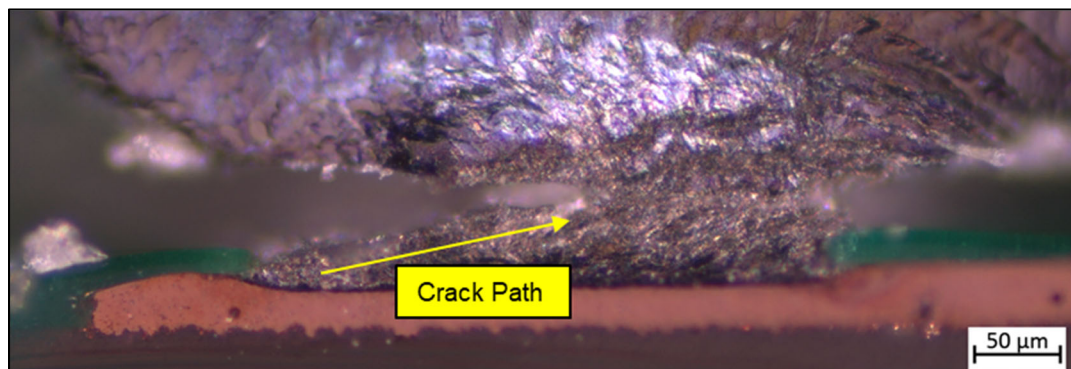


c) Microstructure after 1200 Cycles at 25 °C

Figure 9.5 Degradation of Microstructure with Mechanical Shear Cycling at 25 °C (Non-aged SAC305)



a)

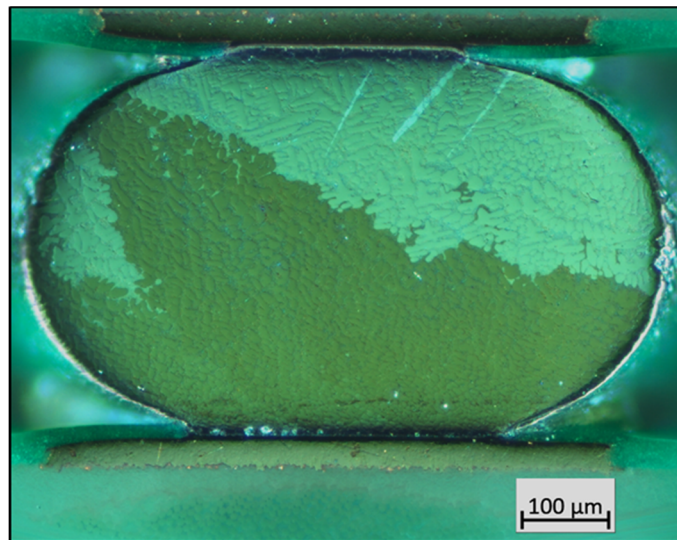


b)

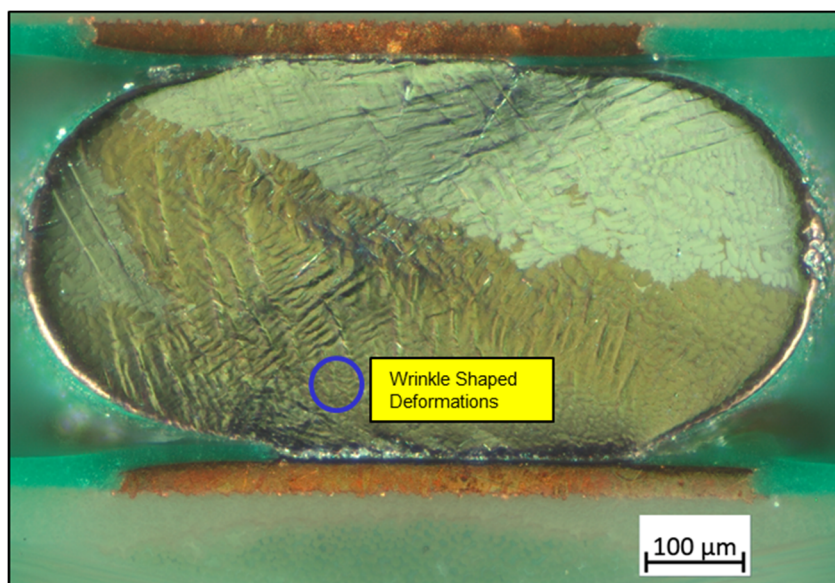
Figure 9.6 Magnified Images of Cracks (Non-aged SAC305)

9.4.2 Non-aged Joints Cycled at Elevated Temperature (100 °C)

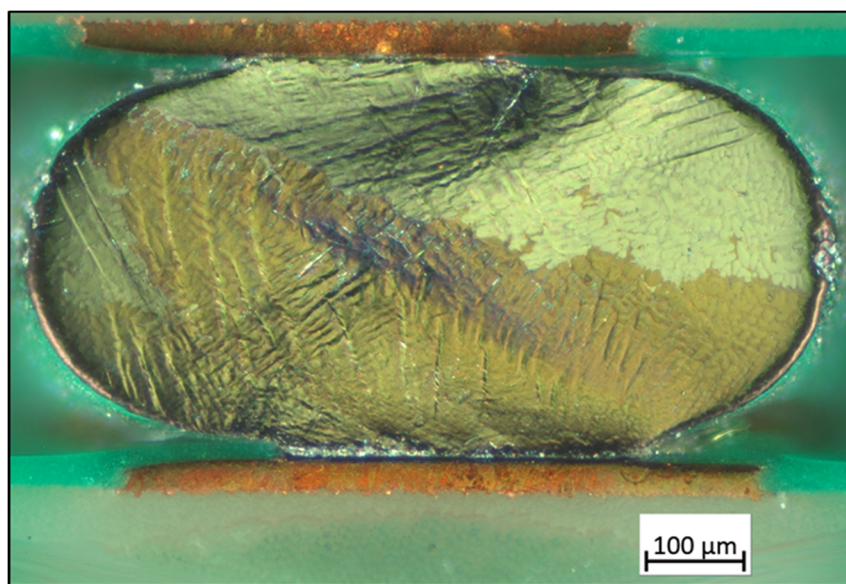
Similar shear experiments were conducted on SAC305 solder joints at a test temperature of 100 °C. The micrographs obtained from various stages during the cycling experiment are illustrated in Fig. 9.7. When compared with the images obtained from joints cycled at room temperature, it can be deduced that the joints cycled at 100 °C accumulated more damage for the same duration of cycling. Slip lines were found to be in two directions for the different grained joints and no signs of any recrystallization assisted crack were found when the joints were cycled at an elevated temperature as well. Another significant observation was the formation of abundant wrinkle shaped damage accumulation on the surface of the solder joints. These deformations were not along the slip lines and were observed to occur all over the joint. This was a common phenomenon across all the joints that were cycled at an elevated temperature. The crack propagation stage was observed to be a lot shorter than when cycled at room temperature. The damages in the microstructure were observed after 400 cycles. The deformations became more severe after 600 cycles. The average fatigue life of samples that were cycled at 100 °C was about 820 cycles.



a) Pristine Microstructure



b) Microstructure after 400 cycles at 100 °C

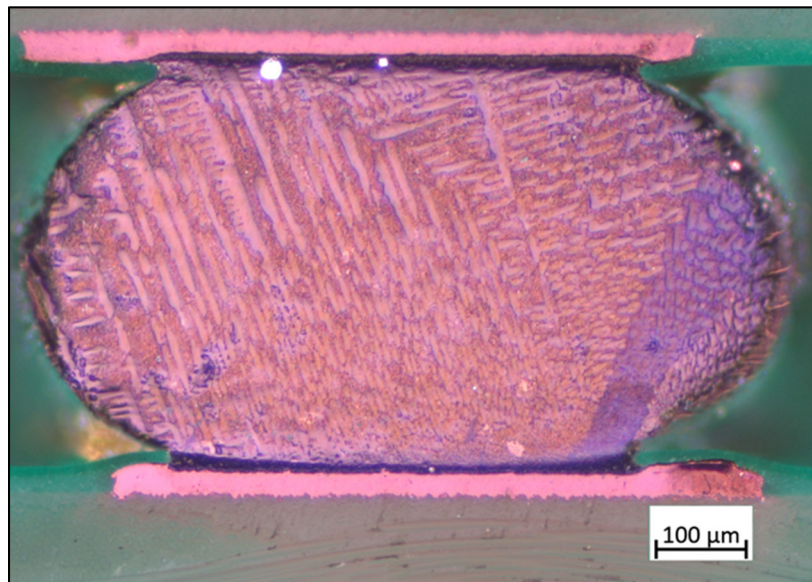


c) Microstructure after 600 cycles at 100 °C

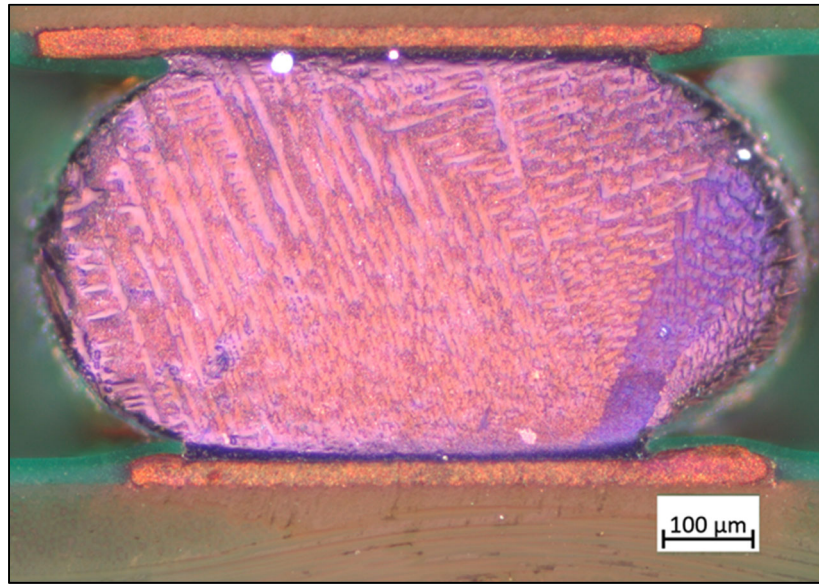
Figure 9.7 Degradation of Microstructure with Mechanical Shear Cycling at 100 °C
(Non-aged SAC305)

9.4.3 10 days aged Joints Cycled at Room Temperature (25 °C)

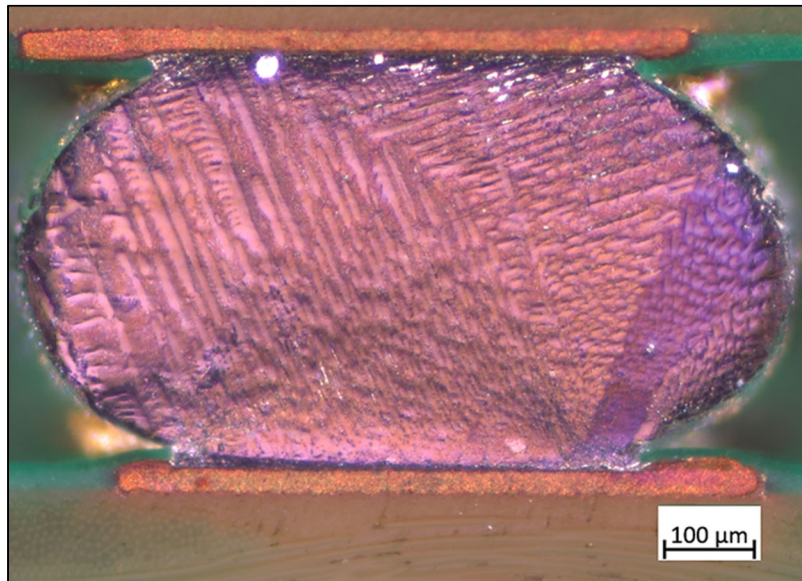
Some of the SAC305 joints were aged for 10 days at 125 °C. Figs. 9.8 (a) and (b) represent the cross-polarized optical images of the microstructure of one such aged joint before and after aging. It was observed that the aging condition does not introduce any additional grains, but has an oxidation effect on the PCB substrate and copper pads which change color after the aging duration. The evolution of the microstructure with mechanical cycling have been depicted in the other micrographs of Figure 9.8. Slip lines began to appear after about 400 cycles, before which no significant degradations were observed. After about 800 cycles, the slip lines were clearly visible and it was observed to be in similar directions as that seen in the non-aged samples. Prior to failure, no new grains were found along the crack path as seen in the micrographs presented in Fig. 9.9. As was observed with the room temperature cycling of non-aged samples, cracks nucleated from areas in the vicinity of the copper pads, and no cracks initiated from the interface of the grain boundaries of the multi-grained SAC305 joints.



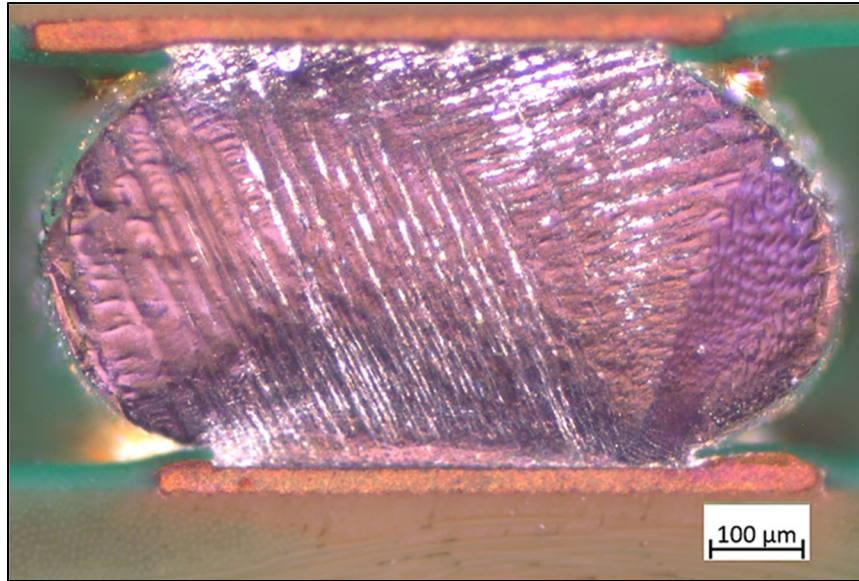
a) Non-aged Pristine Microstructure



b) Microstructure after 10 days Aging

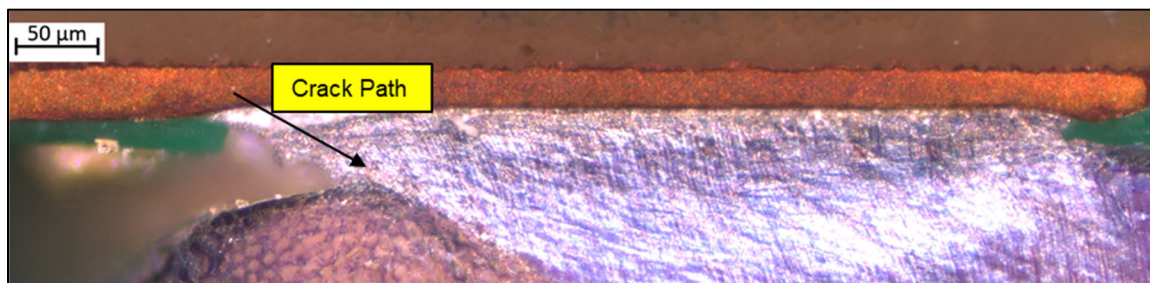


c) Microstructure after 400 cycles at 25 °C

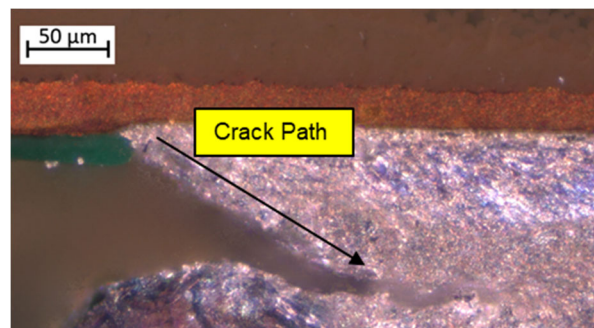


d) Microstructure after 800 cycles at 25 °C

Figure 9.8 Degradation of Microstructure with Mechanical Shear Cycling at 25 °C
(SAC305, Aged at 125 °C for 10 days)



a)

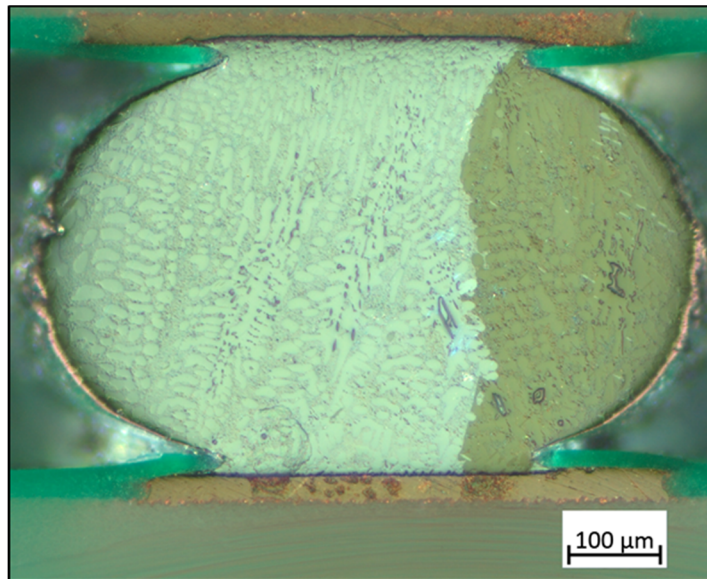


b)

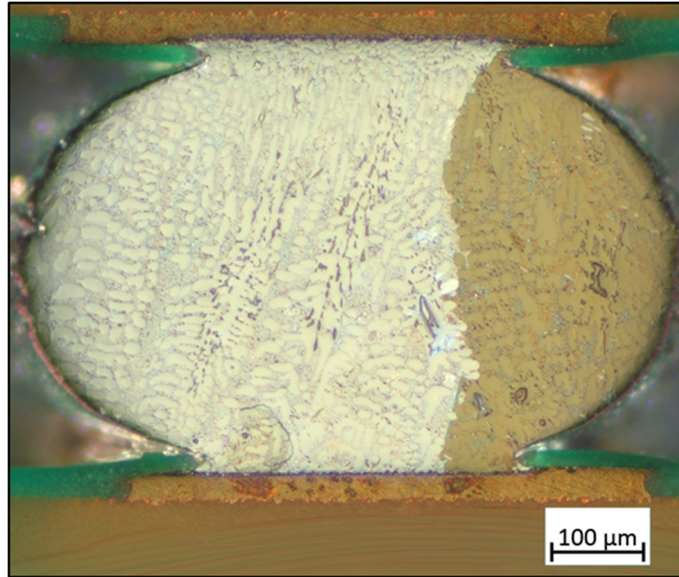
Figure 9.9 Magnified Images of Cracks (Pre-aged SAC305 Specimens)

9.4.4 10 days aged Joints Cycled at Elevated Temperature (25 °C)

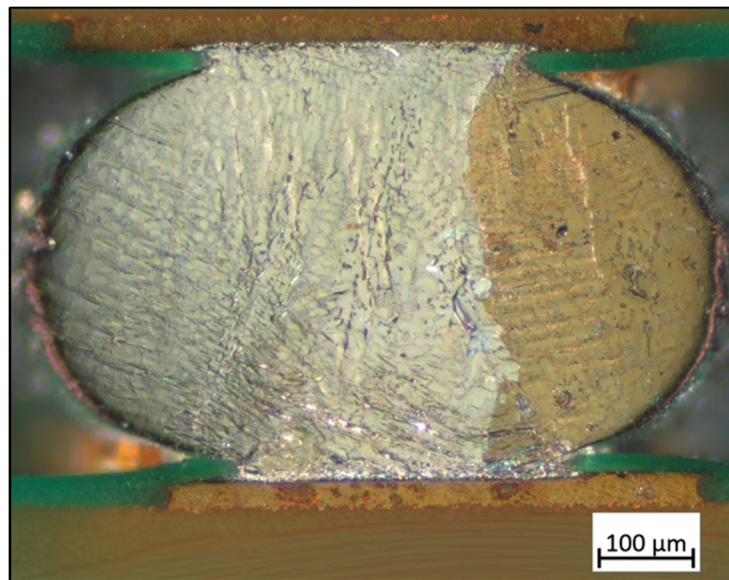
Some of the aged joints were also cycled at 100 °C. These joints were subjected to the worst of all the four conditions. The drastic effect that this condition had on the solder balls is evident in the micrographs presented in Fig 9.10. When aged samples were cycled at 100 °C, the samples failed the quickest (about 570 cycles) when compared to the other conditions. For the same cyclic strain range, these samples demonstrated the most changes in the microstructure. Severe damages were observed just after 200 cycles along the slip lines. The wrinkled shaped damages were also observed in this case as was noticed with non-aged samples cycled at 100 °C. We can thus deduce that these additional deformations are caused due to mechanical cycling at elevated temperatures. The slip line deformations became more destructive after 400 cycles. The crack propagation stage was observed to be very quick, as observed with non-aged samples cycled at 100 °C.



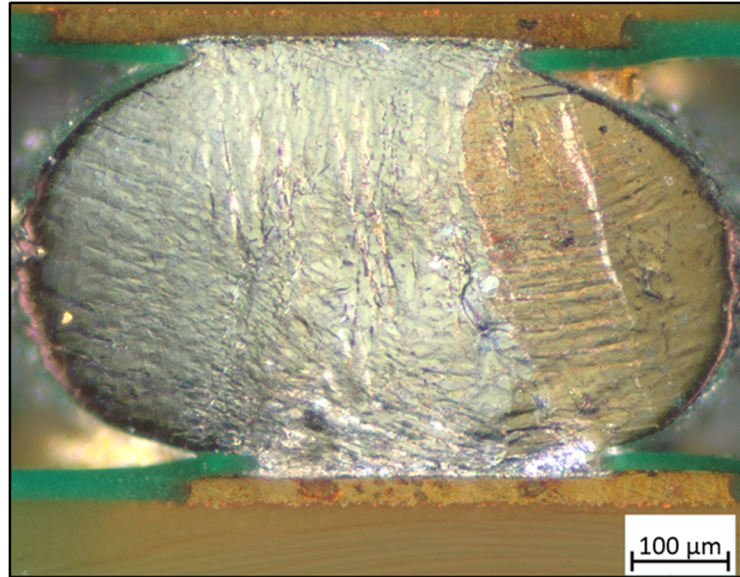
a) Non-aged Pristine Microstructure



b) 10 days Aged Microstructure



c) Microstructure after 200 cycles at 100 °C



d) Microstructure after 400 cycles at 100 °C

Figure 9.10 Degradation of microstructure with mechanical shear cycling at 100 °C (SAC305, aged at 125 °C for 10 days)

9.4.5 Discussion Regarding No Recrystallization

One can deduce that these joints were deformed plastically by observing the hysteresis loops obtained from the cyclic experiments. The hysteresis loop also indicates that with every cycle, a significant portion of strain energy is accumulated within these joints. An amount of this strain energy is stored in the form of dislocations. A higher energy state is always unfavorable and thus this excessive strain energy is reduced either of two phenomenon, recovery and recrystallization. The recovery process involves the distribution of newly formed dislocations to reduce the overall change in stored energy introduced within the joint. This is achieved by relocating the dislocations without any shifting of the grain boundaries itself, thus retaining the identity of the crystal but the dislocations are rearranged to achieve a lower energy state. The recrystallization process, however, involves the shifting of the grain boundaries itself to create new grains which do

not contain any defects. Both the above mentioned processes work against each other, and thus, if most of the stored strain energy is used in the recovery process, the recrystallization phenomenon will not have ample energy to initiate. No recrystallization was observed when non-aged SAC305 joints were mechanically cycled up to failure. This indicates that there was insufficient energy for recrystallization to begin and most of the stored strain energy was used in the recovery process.

9.5 Finite Element Modeling

A Finite Element Model of the isothermal shear mechanical cycling of the sandwiched BGA specimen was developed in ANSYS APDL. This model was then run with the appropriate loading and boundary conditions. The results that were obtained was then validated with experimental data conducted by another group. This would allow us to estimate the results that would be obtained when run under other loading conditions.

9.5.1 Model Development and Loading Conditions

With the aid of the various cross-sections taken of the board, different dimensions of the sandwiched 3 x 3 BGA solder joints were measured using the polarized optical microscope. These dimensions were then used to build and mesh the desired model as depicted in Fig 9.11. A symmetric half model was used for the finite element simulations. SOLID185 elements were used to mesh the entire structure as these elements can be used with a wide range of nonlinear constitutive models, including hyperelasticity, viscoelasticity, in addition to plasticity and viscoplasticity.

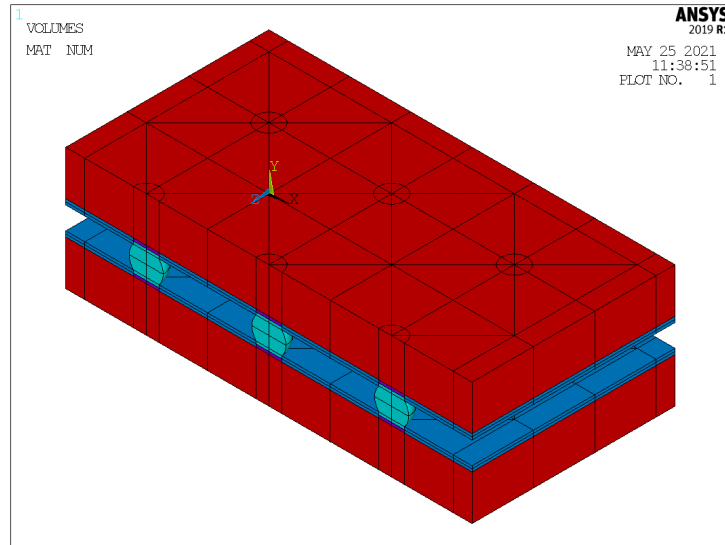


Figure 9.11 3D Symmetric Half Model of the Sandwiched Package

The Anand viscoplastic material model [52] was employed to accurately model the behavior of the SAC305 joints. A set of 9 Anand model parameters for SAC305 was previously evaluated by the authors in reference [45]. These values are tabulated in Table 9.1.

The remaining packaging materials (copper pad, solder mask and PCB) were assumed to be linear elastic materials. The directional behaviors of the PCB was included by using orthotropic material constants including temperature dependence. The material constants were measured using stress-strain and CTE (TMA) testing [45], and are tabulated in Table 9.2. All materials were assumed to be homogenous. Process variations and intermetallic growth were not included.

Table 9.1 Anand Parameters for SAC305

Constant Number	Anand Constant	Units	SAC305 No Aging
1	s_0	MPa	21.0
2	Q/R	1/K	9320
3	A	sec ⁻¹	3501
4	ξ	Dimensionless	4
5	m	Dimensionless	0.25
6	h_0	MPa	180000
7	$\hat{S}$	MPa	30.2
8	n	Dimensionless	0.0100
9	a	Dimensionless	1.78

Table 9.2 Mechanical Properties of Other Components of the Package

Material	Elastic Modulus (GPa)	Shear Modulus (GPa)	Poisson's Ratio	CTE [1/°C]
Copper Pad	129.0		0.34	16.3 x 10 ⁻⁶
Solder mask	3.10		0.30	30 x 10 ⁻⁶
PCB	19.30 (xy, -40 C)	8.71 (xy, -40 C)	0.11 (xy)	14.5 x 10 ⁻⁶ (xy)
	13.20 (xy, 125 C)	5.95 (xy, 125 C)		
	8.48 (z, -40 C)	3.80 (xz, yz; -40 C)	0.39 (xz, yz)	67.2 x 10 ⁻⁶ (z)
	5.84 (z, 125 C)	2.60 (xz, yz; 125 C)		

Two mechanical cyclic loading conditions were applied to the model and studied. The first loading condition displaced the top surface of the package by a range of ± 0.02 mm at a displacement rate of into 0.02 mm/s while the second loading condition displaced the top surface of the package by a range of ± 0.018 mm at a displacement rate of into 0.02 mm/s. The loading conditions have been illustrated in Fig 9.12. For both the loading conditions, all three displacements of the bottom surface of the package was kept zero to

prevent any rigid body motions. The package assembly was assumed to be in a stress-free state at 25 °C (room temperature), with no initial residual stresses/deformations induced in the manufacturing process. The samples were also simulated to have been cycled at room temperature. In all of the models, it was found that the cycle to cycle variation of the solder stress/strain behavior became stable by the third cycle. The values during the third cycle were extracted to evaluate the dissipated plastic work per cycle (ΔW) that was used to estimate fatigue life.

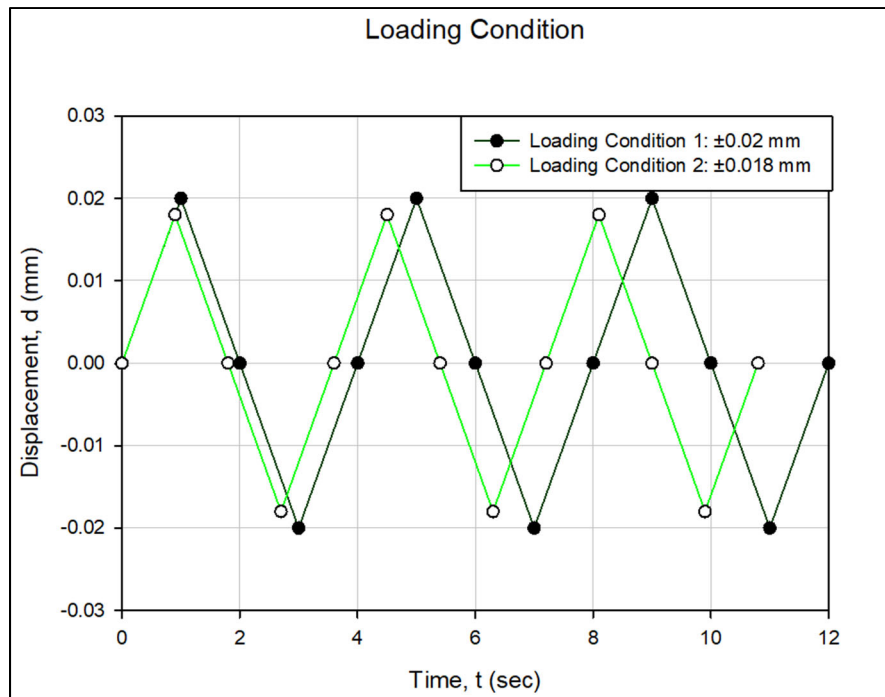


Figure 9.12 Mechanical Cycling Loading Profile

The half model was appropriately constrained along the axes of symmetry, so that for a node in a symmetry plane, the displacement component perpendicular to the symmetry plane was required to be zero. Perfect bonding was assumed at all interfaces between different materials. Iterative solution procedures have been used due to the nonlinear material and kinematic (large deformation) characteristics of the model.

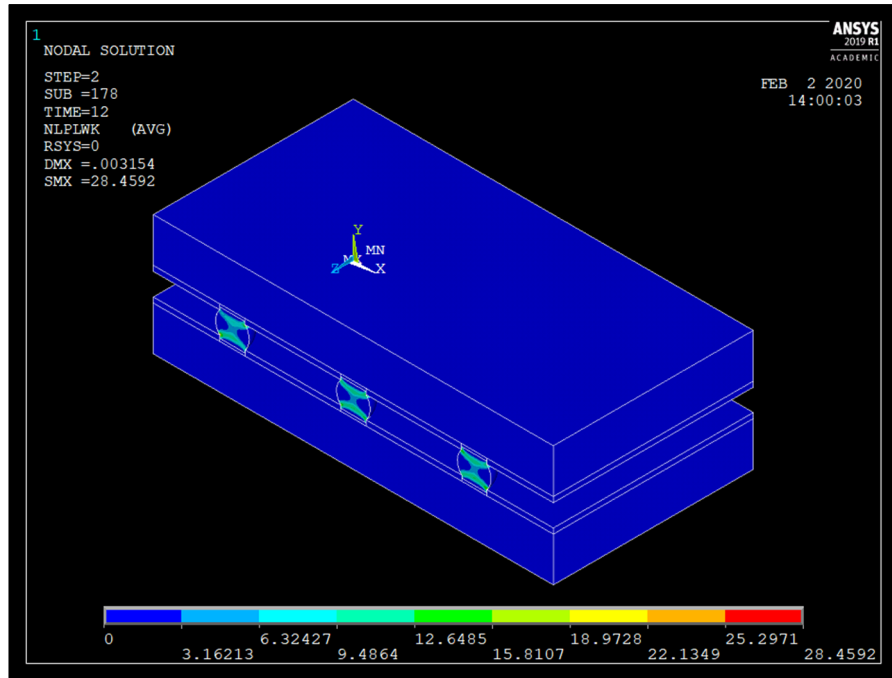
9.5.2 Finite Element Results

In this section, the results obtained for both the loading conditions will be presented and discussed. The plastic work accumulated after 3 cycles was extracted from the FEA models. This was then compared with the experimental data obtained.

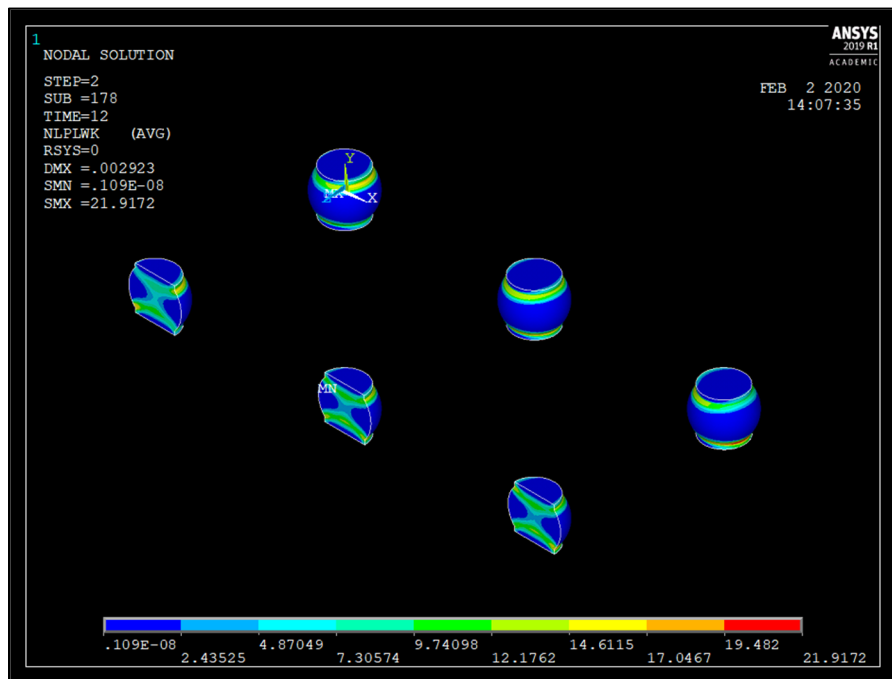
For loading condition 1 (± 0.02 mm), the plastic work dissipated contours obtained are presented in Fig. 9.13. To minimize the mesh dependency of the calculated plastic work values in the joints, it is conventional to calculate volume averaged values across a set of N elements along the crack path using the relationship:

$$(W_{pl})_{\text{Averaged}} = \frac{\sum_{i=1}^N (W_{pl})_i V_i}{\sum_{i=1}^N V_i} \quad (9.1)$$

where $(W_{pl})_i$ and V_i are the elemental values of the accumulated plastic work and Volume. For loading condition 1, the average plastic work accumulated per cycle for the whole specimen was calculated using eqn. 9.1 to be approximately 1.544 MJ/m^3 . Multiplying this with the total volume of the geometric model, results in an initial plastic work of around $0.515 \times 10^{-3} \text{ J}$. The actual cyclic data obtained for this specific loading condition is shown in Fig. 9.14. From the experimental graph of work against number of cycles in Fig. 9.14 [145], it was observed that the initial plastic work accumulated in the sample during the initial cycles was approximately $0.5 \times 10^{-3} \text{ J}$. Thus, the error in initial plastic accumulated work for cycling under loading condition 1 calculated from the finite element model was about 3%.



a) Entire Sample



b) Solder Joints

Figure 9.13 Plastic Work Accumulated Contours for Loading Condition 1

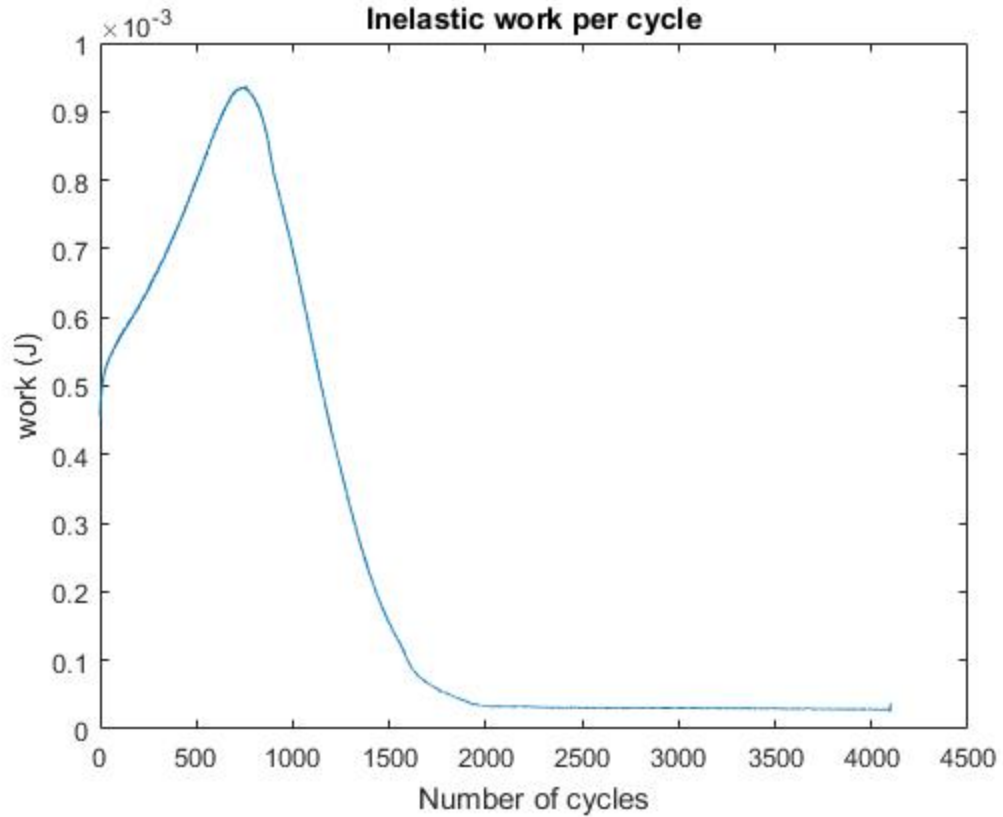
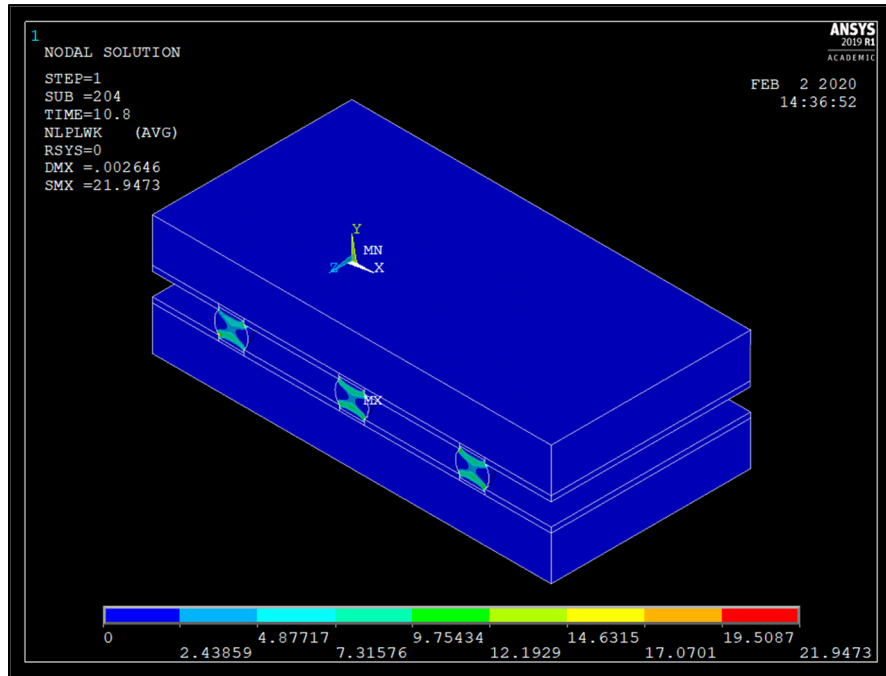
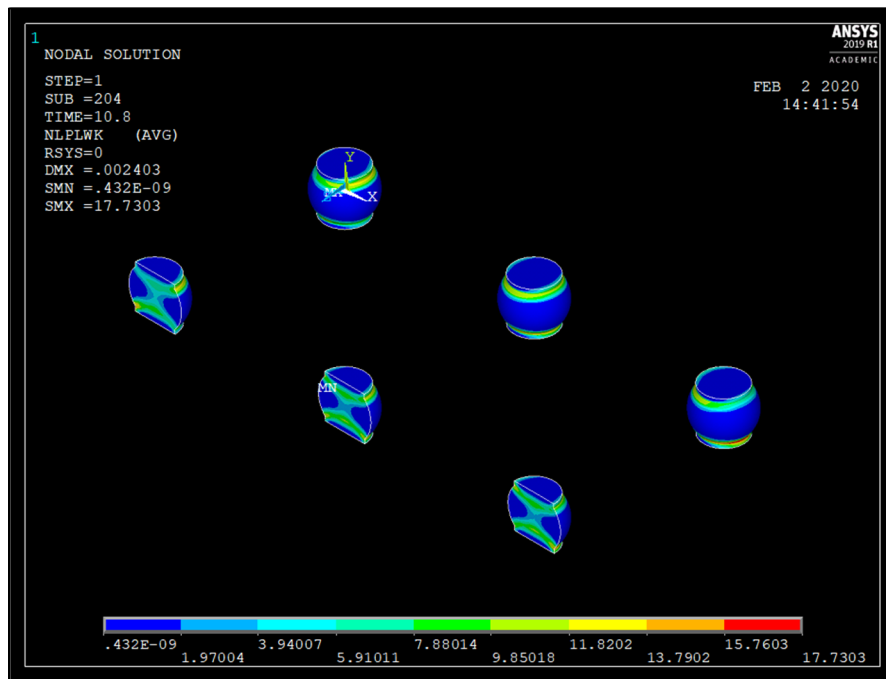


Figure 9.14 Experimental Plastic Work Accumulated vs Number of cycles (Loading Condition 1) [145]

Similarly, the plastic work dissipated contours obtained for loading condition 2 (± 0.018 mm) are presented in Fig. 9.15. The contours obtained were similar to that observed for loading condition 1. For loading condition 2, the average plastic work accumulated per cycle for the whole specimen was approximately 1.228 MJ/m^3 and an initial plastic work of around $0.409 \times 10^{-3} \text{ J}$. The actual cyclic data obtained for this specific loading condition [145] is shown in Fig. 9.16, from which it was observed that the initial plastic work accumulated in the sample during the initial cycles was approximately $0.4 \times 10^{-3} \text{ J}$. Thus, the error in initial plastic accumulated work for cycling under loading condition 2 calculated from the finite element model was about 2.25%.



a) Entire Sample



b) Solder Joints

Figure 9.15 Plastic Work Accumulated Contours for Loading Condition 2

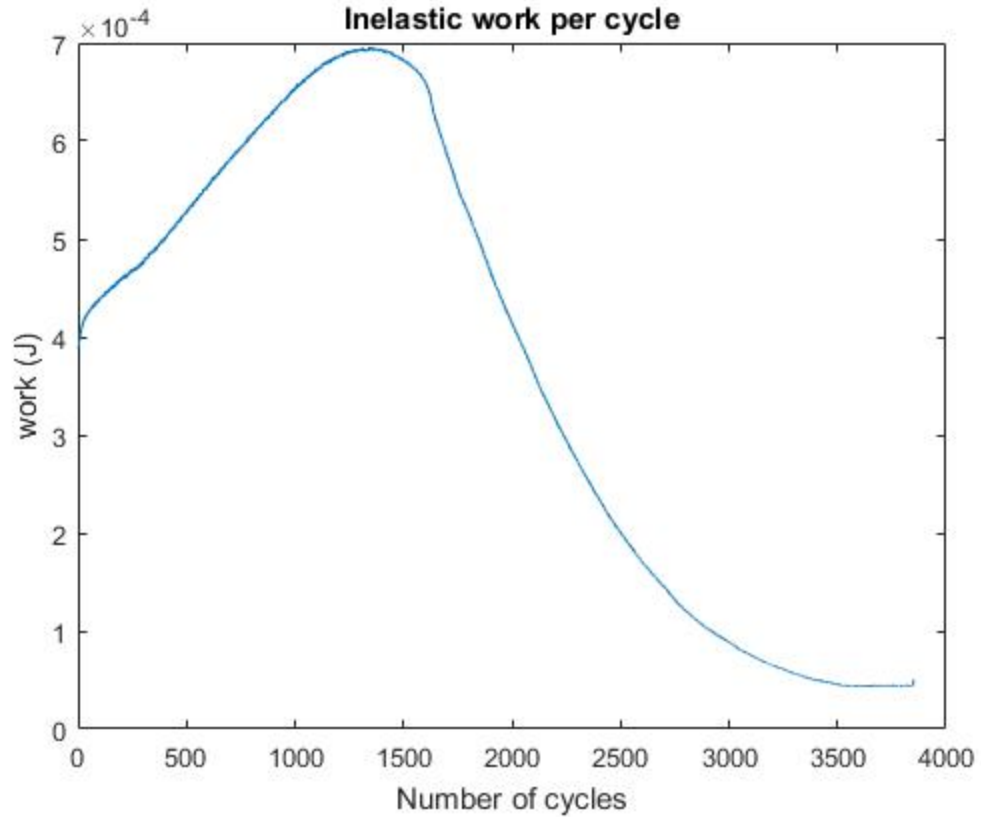
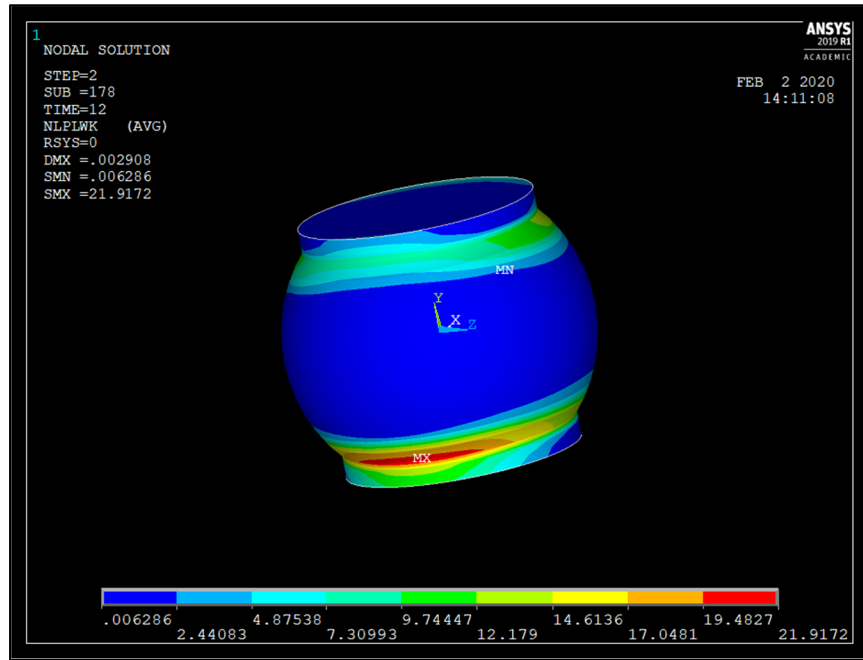


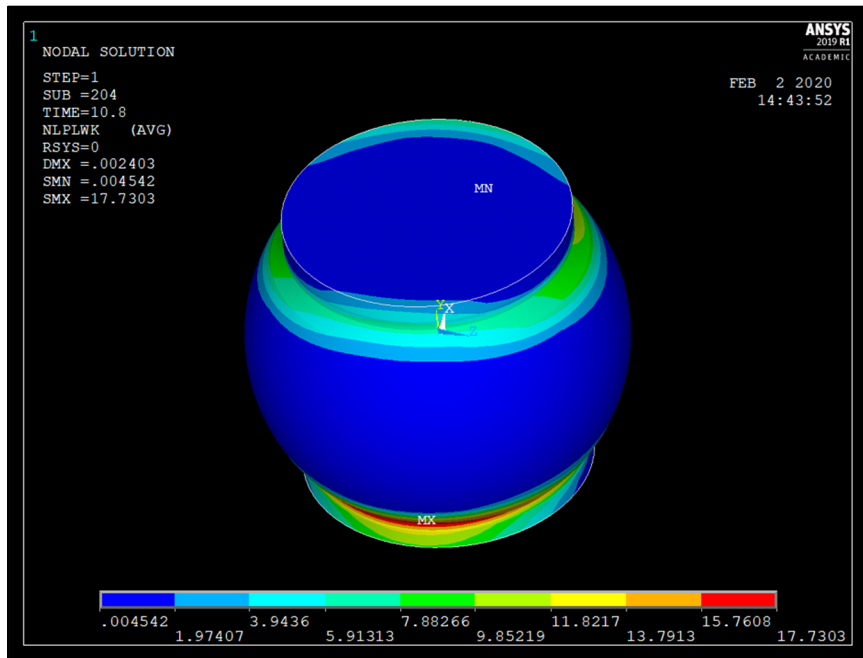
Figure 9.16 Experimental Plastic Work Accumulated vs Number of cycles
(Loading Condition 2) [145]

It was observed that the model estimated the plastic accumulated work fairly accurately for both the loading conditions. It can thus be concluded that this model should be able to estimate the initial plastic work accumulated for other loading conditions as well. In doing so, it will provide a better insight on how one can use this data to estimate a fatigue life for solder joints under shear loading conditions. Another important criteria which ensured that the model could accurately simulate the loading condition was the region of the critical zone in the critical solder joint of the sample. Fig 9.17 shows the critical region obtained for the critical joint subjected to both the loading conditions. The region was at the same location for both the conditions and this region agreed with the observations

obtained from the experiments. Actual experiments on the samples revealed that the solder joints failed near regions depicted by the critical locations of the solder joints.



a) Loading Condition 1



b) Loading Condition 2

Figure 9.17 Plastic Work Accumulated Contours for Critical Solder Joint

9.6 Summary and Discussion

In this chapter, the changes in the cycling behavior and microstructure of SAC305 solder joints subjected to shear mechanical cycling were investigated. Both non-aged and joints aged at 125 °C for 10 days were tested at test temperatures of 25 °C and 100 °C up to failure. The joints were cycled isothermally using a thermal chamber at a strain range of ± 0.018 mm and a stage speed of 0.002 mm/s. The hysteresis curves recorded from the tests were studied and compared. The microstructure of the samples were observed at various cyclic intervals using a polarized optical microscope. The damage accumulated with cycles are visible in the form of slip lines. These slip lines start to become more prominent with increased cycle numbers. Wrinkle shaped deformations not associated with the slip lines were observed on the surface of solder joints that were cycled at elevated temperatures. As can be estimated, cracks nucleated from regions near the corner of the copper pad. The crack propagation stage, however, occupied a small fraction of the fatigue period, which is unlike the situation observed in thermal cyclic loading. This propagation period was observed to be even shorter in samples that were cycled at 100 °C. There were no signs of recrystallization along the crack path when joints were mechanically cycled (for all four cases used in this study) which is typically the method by which joints subjected to thermal cyclic loads fail. A finite element model was developed to be able to estimate the plastic work accumulated per cycle for isothermal shear cycling of the sandwiched BGA packages used. Two different loading conditions were utilized for the simulations and the initial plastic work dissipated was compared with the experimental data. It was observed that the finite element model estimated the plastic work fairly accurately and the critical zone obtained for the critical solder joints also matched with the

experimental portion of this chapter. This would allow one to further develop a model to better understand the effect of mechanical cycling on lead free solder joints.

CHAPTER 10

SUMMARY

10.1 Literature Review

Literature review has been done on conventional and doped lead free solders, cyclic stress-strain behavior of these alloys, effects of thermo-mechanical cycling on solder microstructure, and effects of aging on solder fatigue properties and microstructure.

The mechanical properties of a solder are strongly influenced by its microstructure, which is controlled by its thermal history including its solidification rate and thermal exposures after solidification. Aging of lead free solders leads to changes in their constitutive and failure behaviors. For example, research in the literature has shown that aging leads to large reductions in solder material properties including shear strength, elastic modulus, nanoindentation joint modulus and hardness, high strain rate mechanical behavior, creep response, and Anand model parameters. Other studies have shown that aging causes severe reductions in uniaxial cyclic stress-strain curves and fatigue life, shear cyclic stress-strain curves and fatigue life, fracture behavior, drop reliability, and thermal cycling reliability.

Dopants have been found to strongly influence the properties and behaviors of lead free solders. For Example, Bi helps to reduce solidification temperature, increases strength by means of precipitation hardening, helps to reduce IMC (Intermetallic Compound) layer thickness, and also reduce aging induced decrease of mechanical properties in lead free solder materials. Ni helps to improve thermal fatigue life and drop test performance by refining Sn grain size and reducing the IMC layer formation near the Cu pad. The effects

of rare earth (RE) elements and nanoparticle addition on the properties of lead free solder was also discussed in this chapter.

High temperature and larger strain range cyclic tests leads to the decrease of both tensile and constitutive mechanical properties. These evolutions in the properties of SAC305 lead free solder was recorded and discussed in this dissertation.

The changes in solder mechanical behavior are a result of the evolution of the solder microstructure that occurs during aging. The most well-known and widely observed changes are coarsening of the Ag_3Sn and Cu_6Sn_5 intermetallic compounds (IMCs) present in the eutectic regions between beta-Sn dendrites. Several researchers have proposed empirical models to describe the growth of these secondary phase particles as a function of aging temperature and aging time, and related this growth to mechanical property changes.

10.2 Experimental Procedure

All the experimental procedures and the data processing steps were presented in chapter 3. For uniaxial tensile testing, micro-scale tensile specimens were prepared in a rectangular shaped hollow glass tube using a vacuum suction method. Typical dimension of the uniaxial tensile specimens were 80 (length) X 3 (width) X 0.5 (height) mm. Uniaxial tensile tests were performed using a micro tension torsion testing system.

For cyclic stress-strain testing, newly designed micro-cylinder shaped uniaxial lead free solder test specimens have been prepared in glass tubes using the same vacuum suction process. The cylindrical specimens were introduced to solve the buckling problem during compression in the cyclic or fatigue testing. The cyclic testing was performed using a tension/torsion thermo-mechanical test system. Gripping fixtures were designed and added to the test system to grip the cylindrical specimens. After introducing the cylindrical

specimens and gripping fixtures, we have solved many problems such as buckling during compression, specimen breaks in the grip, and slippage during testing. After testing, empirical models were utilized to represent the stress-strain, creep, and cyclic stress-strain experimental data. To process the cyclic stress-strain data and calculate the areas of the associated hysteresis loops, a pair of hyperbolic tangent empirical models has been used to represent the tension portion and the compression portion of the stress-strain behavior in each cycle. Then, hysteresis loop area (plastic strain energy density), plastic strain range and peak stress can be determined for each cycle.

To study the solder microstructural changes subjected to both cyclic loading and aging, SAC305 and SAC_Q solder samples were formed with Reflow cooling profiles, and then microscopy cross-sections were prepared. After polishing, small indentation marks were added to the cross-sections to facilitate locating the identical regions of interest. Then the microstructural changes in the fixed regions during aging were examined using SEM. To study the effects of mechanical cycling on the solder microstructure, uniaxial samples with various durations of prior mechanical cycling have been prepared. The cycled microstructures were then examined using SEM.

To study the evolution of mechanical properties at joint level, we have explored the effect of shear mechanical cycling on damage accumulation in SAC305 solder joints in a 3x3 BGA sample. Nanoindentation experiments were performed using Hysitron TI950 TriboIndenter.

Newly designed fixtures allowed the gripping of sandwiched SAC305 solder joints. These samples were then aged for 0 days or 10 days at a temperature of 125 °C. The joints were then subjected to both room temperature and elevated temperature and the

microstructure studied after certain durations of shear mechanical cycling to record the changes brought to the microstructure with shear cycling. These images were recorded by a cross polarized optical microscope. The joints were also studied for any recrystallization effects induced by the shear mechanical cycling.

Finite Element Models were also built to study the effect of shear mechanical cycling on solder balls and joints. The results were validated with other experimental data to ensure the proper functioning of the model. Such models would allow one to better estimate the fatigue life of lead free alloys when subjected to mechanical shear loading.

10.3 Evolution of Mechanical Properties in Lead Free and Doped Lead Free

Solders during Cyclic Loading

In this chapter, the effects of isothermal mechanical cycling on damage accumulation in lead free SAC305, SAC_Q (SAC+Bi), and Innolot (6 Element Alloy) lead free bulk solder alloys have been explored. In particular, we have examined both the evolution of the solder microstructure and the changes occurring in the solder constitutive behavior (stress-strain and creep) as cycling progresses during fatigue testing. Such knowledge is necessary to develop accurate fatigue criteria for lead free solder alloys. In addition to quantifying damage accumulation during cyclic testing, we have also measured the evolution of the cyclic stress-strain behavior (hysteresis loop area, plastic strain range, and peak stress) during single fatigue tests run to failure. For the mechanical cycling experiments, cylindrical uniaxial SAC305 and SAC_Q lead free solder test specimens have been prepared using a vacuum suction reflow process within glass tubes. To study the evolution of the cyclic stress-strain behavior during a single fatigue test, the uniaxial specimens have been subjected to cyclic stress/strain loading at constant temperature until

fatigue failure. The cyclic stress-strain curves after various durations of cycling have been extracted, and a four parameter hyperbolic tangent empirical model has been used to fit the entire cyclic stress-strain curve at each chosen increment of cycling. From the recorded cyclic stress-strain curves, the evolution of the solder hysteresis loops with number of cycles has been characterized. Also, the dependencies of the hysteresis loop area (plastic strain energy density), plastic strain range, and peak stress on the duration of cycling have been studied. The first two parameters are often considered to be driving forces for fatigue damage, and used in fatigue life estimation models, such as Morrow model and Coffin-Manson model. Near fatigue failure, the decreases in loop area and peak stress were similar for all the three alloys. The plastic strain range remained essentially constant during the cycling for both materials. To study the effects of cycling on damage accumulation and reduction of the constitutive behavior of SAC305, SAC_Q, and Innolot, uniaxial samples have been prepared and subjected to various durations of prior isothermal mechanical cycling (e.g. 0, 50, 100, 300, 600, 900, 1200 cycles) that were below the fatigue life of the materials. Thus, the prior cycled samples contain different amounts of cycling induced material damage. The cycled samples were then subsequently subjected to stress-strain or creep testing. Using the data from the various tests, we have been able to characterize and quantify the cycling induced damage through the observed changes of several mechanical properties (elastic modulus, yield strength, ultimate strength, and creep strain rate) with the amount of prior cycling. The decrease in the creep strain rate were especially significant for both materials due to exacerbated grain boundary sliding from intergranular fatigue cracking. The cycling induced material property evolutions observed in the two lead free alloys have been correlated with the observed changes in their microstructures that

occurred during the cycling. Special polished test specimens were prepared so that the microstructural evolution of a small fixed region in the solder specimen could be followed as the cycling progressed. It was observed that the cycling induced damage consisted primarily of small intergranular cracks forming along the subgrain boundaries within dendrites. These cracks continued to grow as the cycling continued, resulting in a weakening of the dendrite structure, and eventually to the formation of large transgranular cracks. The distribution and size of the intermetallic particles in the inter-dendritic regions were observed to remain essentially unchanged.

10.4 Effect of Elevated Temperature Cycling on the Properties of Lead Free

Solder Alloys

In this chapter, the effects of elevated temperature mechanical cycling on the damage accumulation in lead free alloys were investigated. SAC305 and SAC_Q uniaxial cylindrical samples were prepared and subjected to constant strain cyclic tests at a test temperature of 100 °C. The hysteresis loops at various intervals were recorded and the different cyclic properties (peak stress, plastic strain range, and plastic strain energy density) were extracted from these hysteresis loops. This allowed us to evaluate the reduction of the peak stress and hysteresis loop area with cycling. The SAC305 samples cycled at room temperature demonstrated a higher peak stress and loop area for the same strain range, and these values decreased less than that observed for SAC305 samples cycled at 100 °C. This suggests larger weakening of the alloy during elevated temperature cycling. When comparing both the alloys, the SAC+Bi alloy demonstrated a higher peak stress and loop area for the same strain range and these values decreased less than that observed in

SAC305. This suggests the better cyclic performance of Bi doped alloys at elevated temperatures.

To gain a better knowledge of the damage accumulation accompanying the elevated temperature mechanical cycling, pristine SAC305 and SAC_Q samples were mechanically cycled for durations of 0, 50, 100, 300 and 600 cycles at temperatures of $T = 100\text{ }^{\circ}\text{C}$. These prior cycled specimens were then either subjected to room temperature tensile or creep tests to quantify the damage accumulated in the samples due to the cyclic tests. The SAC305 samples cycled at $100\text{ }^{\circ}\text{C}$ proved to have a large damage accumulation with the same number of cycles as deduced from the results when compared to the room temperature cycled SAC305 samples, while SAC_Q exhibited a superior performance in the tensile and creep properties when cycled for the same durations at $100\text{ }^{\circ}\text{C}$. However, the SAC_Q samples could not be cycled up to 600 cycles at $100\text{ }^{\circ}\text{C}$ due to the larger plastic work dissipated per cycle.

An SEM microstructural study was conducted alongside to view the changes in the microstructure and correlate them to the degradations that occur with cycling. Polished rectangular cross-sectioned samples were indented to mark regions of interest which were tracked after each cycling duration. Slip bands and microcracks were evident in the SAC305 samples cycled at $100\text{ }^{\circ}\text{C}$ after only 25 cycles. These features were observed in the SAC_Q samples after the 75th cycle. The microcracks, for SAC305 ($100\text{ }^{\circ}\text{C}$ cycling) were observed to occur on the interface of the eutectic and β -Sn regions while they occurred on the sub-grain boundaries of the β -Sn dendrites for the SAC305 ($25\text{ }^{\circ}\text{C}$ cycling) and SAC_Q ($100\text{ }^{\circ}\text{C}$ cycling), with slip lines being visible towards the latter cycles. With increased durations of cycling, the slip lines became more visible while the microcracks

grew in length and the slip line damage was the major visual damage accumulation mechanism for both the alloys cycled at 100 °C. The cracks then propagated in a direction along the slip lines as cycling progressed. No cracks were observed to nucleate in the eutectic phases and cracks were seen there only during the propagation stage.

10.5 Effect of Strain Range on the Cyclic and Constitutive Properties of Lead

Free Solder Alloys

In this chapter, the effects of different strain ranged mechanical cycling on the damage accumulation in lead free alloys were investigated. Uniaxial cylindrical SAC305, SAC_Q and Innolot samples were prepared and subjected to constant strain cyclic tests at 7 different strain ranges at a test temperature of 25 °C. Initial hysteresis loops for each cyclic condition of each alloy were recorded and plotted. These initial hysteresis loop data were then used to construct charts of initial plastic work accumulated vs applied cyclic strain range. Such charts would allow the estimation of the initial plastic work dissipated at any given strain range for the particular strain rate used.

The SAC305 chart was then used to estimate the strain ranges to obtain an initial plastic work, ΔW , of approximately 0.25, 0.5, 0.75, and 1 MJ/m³. Uniaxial cylindrical SAC305 samples were then cycled at these 4 strain ranges at room temperature for 600 cycles. Similarly, strain range levels needed to obtain an initial plastic work, ΔW , of approximately 0.25, 0.5, 0.75, and 1 MJ/m³ at a cycling temperature of 100 °C were also evaluated. SAC305 samples were then also cycled at these 4 strain levels at 100 °C for 600 cycles. The hysteresis loops obtained for all the conditions were recorded and the different cyclic properties (peak stress, plastic strain range, and plastic strain energy density) were extracted from these hysteresis loops. This allowed us to evaluate the

decrease of the peak stress and hysteresis loop area with cycling. It also allowed the comparison in the difference in evolution of cyclic properties when SAC305 was cycled at the same initial plastic work accumulated at the two cycling temperatures. The cyclic properties also reduced with number of cycles at higher applied strain ranges. It was observed that the elevated temperature cycling brought about a much larger decrease in the cyclic properties despite cycling at the same initial plastic work accumulated level. This suggests that the decrease in properties brought about by elevated temperature cycling is much harsher than the decrease in properties brought about by cycling at a larger strain.

To gain a better knowledge of the damage accumulation accompanying the isothermal mechanical cycling, pristine SAC305 samples were mechanically cycled for durations of 0, 50, 100, 200, 300 and 600 cycles at temperatures of $T = 25$ and $100\text{ }^{\circ}\text{C}$. These prior cycled specimens were cycled at strain ranges previously mentioned to obtain specific values of initial plastic work accumulated. These pre-cycled samples were then subjected to room temperature monotonic tensile tests to quantify the damage accumulated in the samples due to the cyclic tests. The trends observed in the evolution of the Elastic modulus, UTS, and yield stress were similar to that observed with the changes in hysteresis loop area and peak stress evolution. It was once again observed that SAC305 samples cycled at $100\text{ }^{\circ}\text{C}$ proved to have a larger damage accumulation with the same number of cycles and same initial plastic work when compared with its room temperature counterpart.

Overall, all these data can be incorporated in a constitutive model to better model the viscoplastic nature of lead free alloys in electronic packages. All most all the models used to estimate solder life fatigue assumes the plastic work accumulated within the lead free solder joint to be constant. This is not the case, and with the usage of this data, one

should be able to better estimate the changes in various properties of lead free joints when subjected to cyclic loading.

10.6 Aging Effects on the Cyclic Properties of SAC305 Lead Free Solder

In this chapter, an investigation on the effects of aging on the cyclic stress-strain behavior of reflowed SAC305 lead free solders was examined. Using a micro tester machine equipped with a newly designed gripping fixture, uniaxial isothermal cyclic tests were conducted. For preparing the testing specimens, a reflowed (RF) solidification profile was used, which produced microstructures of SAC305 similar to that observed in industrial solder joints. Cylindrical uniaxial test specimens of reflowed SAC305 were formed in circular cross-section glass tubes using a vacuum suction process. Specimens aged at 125 °C for different aging times (0, 1, 2, 5, 10, and 30 days) were subjected to cyclic (tension/compression) mechanical loading at room temperature under strain controlled cycling at different strain levels. After fitting the cyclic stress-strain curves obtained from the tests, the evolution of the cyclic stress-strain behavior with aging has been characterized in terms of peak stress, hysteresis loop, and plastic strain range. It was observed that the hysteresis loop area, plastic strain range, and peak stress of SAC305 reduced while the plastic strain range increased with an increased aging period for any particular strain range. For any particular aging condition, it was observed that the hysteresis loop area, peak stress and the plastic strain range, all increased with an increase in applied strain range.

Plots of initial plastic work accumulated vs applied strain range were then constructed to help facilitate the deduction of initial plastic work at any given aging condition and applied strain range. Such curves would aid in allowing the cycling of different aged SAC305 samples such that the initial plastic work dissipated would remain

the same, thus allowing a more comprehensive comparison between the effect of aging on SAC305 lead free solder alloy.

The effects of prior aging on damage accumulation occurring in the microstructure of SAC305 subjected to mechanical cycling was also studied. Uniaxial rectangular cross-sectioned samples were prepared and polished so that the microstructural changes could be tracked after the initial aging, and then subsequently with mechanical cycling. In particular, we examined the microstructural changes that occurred in small fixed regions in the solder samples, rather than using several different regions. Regions of interest near the center of the sample were marked using small indents formed with a nanoindentation system. Samples were then subjected to aging at 125 °C for various durations to produce several different initial microstructures. Scanning electron microscopy (SEM) was used to investigate the aging induced microstructural changes in the regions of interest in the solder sample. After aging, the samples were then subjected to mechanical cycling. After various durations of cycling (e.g. 0, 10, 25, 50, 100, 200, 300 cycles) that were below the fatigue life of the materials, the regions of interest were again examined using SEM. Using the recorded images, the microstructural evolutions in the fixed regions were observed, and the effects of the initial aging on the results were determined. It was found that the number of IMC particles decreased while the average diameter of the particles increases significantly due to the initial aging.

After the cyclic loading was performed on the sample, it was observed that the cycling induced damage consisted primarily of small intergranular cracks forming along the sub-grain boundaries within the β -Sn dendrites. These cracks continued to grow in size as the cycling continued, resulting in a weakening of the dendrite structure, and eventually

resulting in the formation of large transgranular cracks. The distribution and size of the intermetallic particles in the inter-dendritic regions were observed to remain essentially unchanged with the application of the mechanical cyclic load. Relative to the non-aged samples, there were significant differences observed in the rate and intensity of the micro crack growth occurring in the heavily aged samples that began with much coarser microstructures. In particular, there were tiny cracks visible in the eutectic regions even after a small number of loading cycles (e.g. 25-50 cycles). These cracks eventually helped the transgranular cracks develop more quickly. In comparison to the non-aged SAC305 samples, the IMC region was less effective in pinning and blocking the intergranular cracks. Therefore, it was found that aged SAC305 samples exhibited more damage compared to the non-aged samples after the same cycling duration.

10.7 Evolution of the Mechanical Properties of Individual Lead Free Solder Balls Subjected To Cyclic Loading

In this study, we have explored the effect of shear mechanical cycling on damage accumulation in SAC305 and SAC_Q solder joints in a 3x3 BGA sample. Such studies are needed to help improve fatigue models for lead free solders. The solder balls were cycled individually with a peak load of 600 gf by a Micromechanical tester for various durations (50, 100, 150, 200, 300 cycles), all of which were below the fatigue life of these joints. After each cycling duration, nanoindentation experiments were conducted on the samples to test for changes in elastic modulus, hardness, and creep response. It was observed that the elastic modulus decreased by about 27-32% while the hardness decreased by about 12-16% after 300 cycles for SAC305. SAC_Q joints experienced a decline of about 37-43% in elastic modulus and a decline of about 15-20% in hardness after 300

cycles. Although the values of elastic modulus and hardness of SAC_Q were greater than that of SAC305, the larger decline in values can be attributed to the larger amount of damage accumulation in SAC_Q. The creep properties of the lead free solder joints also decreased with shear mechanical cycling. It was seen that for a stress level of 100 MPa, the creep strain rate increased by about 24X to 32X after the end of 300 cycles for SAC305. On the other hand, SAC_Q experienced an increase of about 3X to 9X after 300 cycles at the same stress level. It can thus be deduced that SAC_Q has better creep properties than SAC305 after undergoing shear mechanical cycling. We have also seen that with the cycling of lead free solder samples, small intergranular cracks form. These cracks tend to grow along the subgrain boundaries and increase in length with cycling, thus weakening the β -Sn dendrites and finally leading to the creation of larger transgranular cracks. A finite element model was also developed to estimate the fatigue life of SAC305 solder balls when subjected to isothermal shear cycling. The model had a fairly good agreement with the ImAg board finish but needed more improvement for the ENIG board finish. The model, however, was able to estimate the failure regions accurately and can be developed further to help deduce the fatigue life of solder balls when subjected to shear cycling under other loading conditions.

10.8 Effects of Mechanical Cycling in Shear on Lead Free Solder Joints

In this chapter, the changes in the cycling behavior and microstructure of SAC305 solder joints subjected to shear mechanical cycling were investigated. Both non-aged and joints aged at 125 °C for 10 days were tested at test temperatures of 25 °C and 100 °C up to failure. The joints were cycled isothermally using a thermal chamber at a strain range of ± 0.018 mm and a stage speed of 0.002 mm/s. The hysteresis curves recorded from the

tests were studied and compared. The microstructure of the samples were observed at various cyclic intervals using a polarized optical microscope. The damage accumulated with cycles are visible in the form of slip lines. These slip lines start to become more prominent with increased cycle numbers. Wrinkle shaped deformations not associated with the slip lines were observed on the surface of solder joints that were cycled at elevated temperatures. As can be predicted, cracks nucleated from regions near the corner of the copper pad. The crack propagation stage, however, occupied a small fraction of the fatigue period, which is unlike the situation observed in thermal cyclic loading. This propagation period was observed to be even shorter in samples that were cycled at 100 °C. There were no signs of recrystallization along the crack path when joints were mechanically cycled (for all four cases used in this study) which is typically the method by which joints subjected to thermal cyclic loads fail. A finite element model was developed to be able to estimate the plastic work accumulated per cycle for isothermal shear cycling of the sandwiched BGA packages used. Two different loading conditions were utilized for the simulations and the initial plastic work dissipated was compared with the experimental data. It was observed that the finite element model estimated the plastic work fairly accurately and the critical zone obtained for the critical solder joints also matched with the experimental portion of this chapter. This would allow one to further develop a model to better understand the effect of mechanical cycling on lead free solder joints.

10.9 Recommendation of Future Work

Following future work can be performed to extend the findings of this dissertation:

- Characterize and quantify the effects of aging on grain size and grain orientation of lead free solders in the same region using polarized light microscope and EBSD.
- Use the same procedures and methods to study the effects of aging and cycling on the microstructure of different percentage weights of Bismuth doped lead free alloy.
- Quantitatively analyze the aging induced evolution of IMC particles in fixed regions due to mechanical cyclic loading.
- Study the effect of elevated temperature and strain range on doped lead free alloys such as SAC_Q and Innolot.
- The experimental procedure to evaluate the mechanical properties evolution of individual solder joint presented in this dissertation can be used to perform creep testing under shear loading. Anand parameters can also be evaluated using these experimental results.
- Use the results obtained for the changes in mechanical properties with cycling to re-evaluate Anand parameters at a certain temperature and strain range. Plugging the revised Anand parameters in a Finite Element Model will give a better estimate of the estimation of fatigue life of solder joints. This would be possible as the finite element model would now not assume the hysteresis loop of the lead free solder to be constant throughout the whole cycling process.
- Study the effect of mechanical cycling on Low Temperature Solders.
- Develop alternative models (other than Anand Model) to better estimate cyclic loading of lead free solders.

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