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CHARACTERIZATION OF MICROSTRUCTURAL EVOLUTION OF CREPT, AGED AND THERMOMECHANICALLY FATIGUED EUTECTIC Sn-Ag SOLDER JOINTS USING ORIENTATION IMAGING MICROSCOPY

Ву

Adwait U. Telang

A THESIS

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Department of Chemical Engineering and Materials Science

ABSTRACT

CHARACTERIZATION OF MICROSTRUCTURAL EVOLUTION OF CREPT, AGED AND THERMOMECHANICALLY FATIGUED EUTECTIC Sn-Ag SOLDER JOINTS USING ORIENTATION IMAGING MICROSCOPY

By

Adwait U. Telang

Single shear lap eutectic Sn-Ag solder joints with copper substrate were subjected to isothermal aging, creep at room and elevated temperature, and thermomechanical fatigue (TMF). Orientation Imaging Microscopy (OIM) studies reveal how the crystallographic orientations are correlated with microstructural features in the solder joints. Certain misorientations appear to be energetically favored during solidification, which have twin and Σ boundary type relationships. Changes in the crystallographic orientations, grain size and misorientation angles between grains, occurring due to subsequent heating and/or deformation were documented and analyzed. The gross texture components remained unchanged after creep and aging. High temperature processes caused some grain boundary motion and modest grain growth. Observations made in three different regions of a specimen that underwent 150 and 370 TMF cycles indicate that no significant changes occurred, though grain boundaries moved, resulting in slight grain growth. This study shows that the lead-free solder joints are multi-crystals, so that deformation is very heterogeneous and sensitive to crystal orientation, strain and the temperature history.

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ACKNOWLEDGMENTS

I would like to convey my deepest thanks to my advisor, Dr.T.R.Bieler whose constant support and encouragement provided the driving force for this research project. I would also like to thank Dr.K.N.Subramanian for his excellent tutelage and support during the course of my Master's Program. Observing and learning from the unique ways in which both these professors approach problems has been a rewarding experience of my graduate studies for which I am extremely grateful.

I also extend my thanks to Dr.D.Liu for being a member on my committee. Many thanks to Dr.J.P.Lucas and Dr.M.A.Crimp for generously sharing their facilities for such studies to transpire.

I would like to express my appreciation to my colleagues Mr.J.G.Lee, Mr.H.Rhee, Mr.B.Simkin, Mr.B.C.Ng, Mr.P.Sonje, Dr.F.Guo and Dr.S.Choi for their support and camaraderie.

Finally, my deepest gratitude to my family who have always stood by me, providing me the much needed perspective and strength and the inspiration to keep going.

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Images in this thesis are presented in color

INTRODUCTION

Soldering is a joining technique being used since ancient times (2000 to 3000 years¹). Soldering, like welding joins two materials on heating them to a temperature with a filler material whose liquidus does not exceed 450°C. In the past several decades solder joints have made valuable connections in automobiles, aircrafts, spacecrafts, computers, televisions and electronics.

Soldering has emerged as an integral part of the electronic assembles where it helps in connecting several devices on to printed circuit boards (PCB). Several new packaging technologies like surface mount technology (SMT), ball-grid array (BGA), plated through-hole (PTH) and multi-chip modules (MCM), have made it possible to reduce the size of the electronic packages. Miniaturization of electronic components has led to smaller electronic packages and an increase in the interconnection density. However, the higher package density and the reduction of joint dimensions has compelled the solder joints to perform better.

So far, no other technique has been developed to replace solder as an interconnect to the same level of performance. Individual components such as chips, resistors, capacitors, etc. are mounted via SMT or PTH type assemblies on PCBs with the solder as the only interconnect in most cases (except when larger components need to be mounted and then screws and straps are used). These solder joints not only serve as mechanical supports, which physically incorporate such components onto the PCB, but also as electrical connections for linking the components to the electronic circuitry. Smaller electronic packaging puts forth several constraints on the solder interconnect and necessitates better inherent properties in the solder to resist failure during operation. About 70% of failure in most electronic circuitry is solder related, which makes it important to understand their deformation behavior and modes of failure.

Lead-based solders have been used for several decades as solder interconnect material. Table I shows some of the lead-based solders along with their melting temperatures. However, since lead is toxic, environmental concerns and international trade restrictions have warranted the eradication of lead from all electronic based components²⁻⁵. Several alternatives have been sought in this regard to replace lead-based solders, some of which are given in Table I. Eutectic tin-silver (96.5Sn-3.5Ag by wt.%) based solders have gained particular interest in recent years due to their higher melting temperatures (221°C) as compared to lead-tin (37Pb-63Sn wt.%) solders (183°C). There is sufficient data on mechanical properties and microstructural details of lead-based solders, however much less is known about tin-based solders, which has different characteristic properties than Sn-Pb solders. Therefore the selection of the right material to replace Sn-Pb solders is still in the embryonic stages because deformation characteristics and failure modes for eutectic Sn-Ag have not been fully characterized. Different damage accumulation processes occur in service, at different times during the life cycle that can lead to the ultimate failure of a solder joint. Hence there is need to study Sn-based solders to ascertain their usefulness as good solder interconnects.

Alloy	Melting Range		Bulk Tensile	Brinell	
(wt.%)	(°C)		Strength	Hardness	
	Liquidus	Solidus	(MPa)	(BHN)	
Sn-based			JA		
90Sn-10Ag	221	295	61.5	12.9	
95Sn-5Ag	221	245	-	-	
96.5Sn-3.5Ag	221	221	61.5	12.9	
95Sn-5Sb	233	240	43.9	11.4	
80Sn-20Zn ⁷	200	270			
50Sn-50In ⁷	117	125			
42Sn-58Bi	138	138			
95Sn-3.5Ag-1Cd-0.5Sb ⁸	218	221			
99.3Sn-0.7Cu ²	227	227			
95.5Sn-4Cu-0.5Ag ²	216	216			
Pb-based					
90Pb-10Sn ⁸	225	300			
80Pb-20Sn	183	277	-	-	
70Pb-30Sn	183	257	41.9	8.7	
50Pb-50Sn	183	216	-	-	
37Pb-63Sn	183	183			
97.5Pb-1Sn-1.5Ag	309	310			
93.5Pb-5Sn-1.5Ag ⁷	296	301			
52Pb-30Sn-18Cd ⁷	145	145			
32Pb-16Sn-52Cd ⁸	96	96			
50Pb-50In ⁷	180	209			

Table I. Composition and melting temperature of various solders.

* All data can be found in Ref.6, exceptions are the ones with specific reference numbers.

Deformation is inherent in any material put in service over a period of time. One of the most important deformation processes is TMF, wherein the solder material goes through several cycles of repeated reversed stress states and relaxation as a consequence of temperature variations. The stresses can be induced by mechanical means, but during service it is most often due to the coefficient of thermal expansion (CTE) mismatches between solder/substrate/component. Anisotropic behavior of Sn plays an important role ⁹ at stress levels possible in a joint. Bulk tin expands much more (along the c-axis) on heating as compared to the copper substrate, leading to various stress states within the joint depending on the relative orientation of crystals. Lee et.al.⁹ have made an attempt to look at the stresses that can arise within a solder joint as a consequence of different crystallographic orientations of the Sn grains over a 165° range of temperature.

Another important factor which needs consideration during deformation is that eutectic Sn-Ag solder does not have a soft phase like Pb in Sn-Pb solder which can accommodate the stresses resulting from the anisotropic deformation of Sn, hence retained stress in the solder joint leads to extensive stress accumulation during the large temperature excursions (which can range from sub-zero to 150°C) in typical automotive under-the-hood type of situations. This stress is not completely relaxed even after long periods of time¹⁰, resulting in nucleation of micro cracks that progress with time and combine to cause failure of the joint^{11,12}. Most solder joints tend to fail in regions close to the interphase boundary in the solder matrix due to the solder being relatively soft as compared to the hard interphase intermetallic layer formed between the solder and the substrate. Other prominent processes occurring during service are aging and creep. Solder joints can experience high homologous temperatures during service. Under normal operating conditions temperatures of around 150°C (423°K) are common. This temperature is greater than $0.5T_m$ for Tin-silver solder (where $T_m=221°C$, i.e. 494°K) and hence there is recrystallization, recovery and grain growth occurring simultaneously in the solder joint. Solder joints not only serve as electrical connections but are also mechanical supports to the surface mount components and hence need to have good creep properties which are of great concern at such high homologous temperatures.

There is a strong correlation between the microstructure and the properties exhibited by the solder material^{2,13,14}. There is also a direct correlation between microstructure and the crystallographic orientations that go with the microstructure. Such crystallographic orientations can affect fracture behavior and mechanics, corrosion resistance, precipitation and recrystallization. Texture studies have been carried out for several years using X-rays and the technique has allowed theories to be developed that describe how preferred crystallographic arrangement in the material evolves during service conditions. Orientation Imaging Microscopy (OIM) using Electron Backscattered Diffraction Pattern (EBSP) is the latest development in the texture arena, and it enables us to visualize the direct correlations between texture and the microstructure.

Humphreys¹⁵ in his overview paper discusses how EBSP is used to investigate phase distributions, grain and subgrains and grain boundary characteristics in Al alloys. Randle et.al.¹⁶ discuss how grain boundary misorientations and texture affect anomalous grain growth in the nickel-based alloy Nimonic PE16 for particular heat treatment conditions. Tai et.al.¹⁷ in their paper have used the EBSP technique to study the

crystallographic orientations of PZT ceramics and their average and local preferred orientations. Hasegawa et.al.¹⁸ discuss how EBSP technique was used to study the mechanisms of microstructure development caused by static and dynamic recrystallization in pure nickel. Hong et.al.¹⁹ showed that the magnetic property of grain orientation in electrical steels is strongly dependent on the texture orientation of the secondary recrystallized grains. Herein the grain size and texture during primary recrystallization influence the evolution of secondary recrystallization and hence the magnetic properties of grain oriented electrical 3% Si steels. Bieler and Semiatin²⁰ used EBSP to examine heterogeneous deformation mechanisms in hot working of Ti-6Al-4V.

The investigations illustrated above indicate that EBSP was effective to study how changes in the microstructure cause changes in the properties. There is need to know how properties change during service in Sn-based solders to predict reliability. Thus EBSP may help identify mechanisms of change that affect properties of solder joints. Since no prior studies with respect to crystallographic orientations and texture have been carried out in Sn-based solders, EBSP has a broad scope with the ability to explore a variety of issues of concern in solder material.

1.1 BACKGROUND FOR TEXTURE AND OIM

Texture is defined as a collective distribution of crystallographic orientations in a polycrystalline aggregate²¹. Gaining control of texture gives enhanced ability to optimize properties at the production level and hence reduce costs. X-ray diffraction has been the most commonly used technique to determine texture of materials (macro texture). However, X-rays provide no better spatial resolution than the area illuminated by the beam, which can be quite small when a synchrotron source is used²² (which is a high intensity parallel and focused X-ray beam). Hence a technique based on X-rays is usually only good for macrotexture determination, where no information about misorientations between grains can be obtained.

The transmission electron microscope (TEM) and scanning electron microscope (SEM) using electrons as their primary source can also be used for microtexture determination. Microtexture is defined as *a population of individual crystallographic orientations, which are usually related to one or more features in the microstructure.* TEM can be used where precise measurements of grain and subgrain orientation or misorientations are desired. However, the TEM process is quite tedious because multistage specimen preparation is time consuming. Since only a small electron transparent region in the foil is obtained, the orientation of only a small fraction of the total volume of the thin foil is obtainable, and hence it is difficult to have statistical certainty that observations correlate to the bulk changes and texture in the specimen. Also, TEM needs off-line diffraction pattern indexing, though recently on-line techniques are commercially available (hkl Channel Acquisition software version 4.2 by HKL

Technology, Inc., Blaakildevej, 17k, Hobro, DK-9500, Denmark and OIM data collection software version 3.5 by TSL Laboratories, Draper, UT).

Grain orientations can be measured in the SEM with two techniques, namely Selected Area Channeling (SAC) and Electron Back-scatter Diffraction Pattern (EBSP). In SAC one measures the orientation of individual grains, but the limited angular range (15°) and spatial resolution of ~15 μ m are some of the drawbacks along with the lack of automation. Thus, it is tedious to measure the entire set of local orientations to correlate with the microstructure of the specimen. One can obtain a "mesotexture" for the larger grains, which provides us information about particular grains and their orientation relationships with neighbors. (See Figure 1).

On the other hand, EBSP has a better spatial resolution (100-300nm) and angular range (up to about 80°), possibly easier specimen preparation and on-line acquisition of data and analysis, which allow large regions of the specimen to be analyzed quickly with reasonably good accuracy. Hence it has gained popularity in a very short time. The texture one obtains with EBSP is called microtexture where crystallographic information of individual crystals / grains and the grain boundaries is delineated in the form of a two-dimensional map, where every pixel corresponds to the spot from where it was taken on the specimen during the scan. This map is called an Orientation Imaging micrograph and this technique is called Orientation Imaging Microscopy (OIM). These maps correlate very well with the microstructural features and give an insight into the crystallographic orientations, which is invaluable for comprehensively studying any material and material behavior. Solder joints are often about 100µm thick; hence conventional X-rays are

impracticable. SAC is time consuming and hence the EBSP technique is a favorable technique to analyze texture of solder joint specimens rapidly.



Figure 1. An illustration of the relationship between (a) micro and (b) mesotexture. Macrotexture shows 14 grains oriented with respect to the specimen X, Y and Z. (a) Microtexture correlates specific grains and their crystal orientations; three different grain orientations have three different c-axis poles (shown by red, blue and green colored grains)²³ in Figure 1(c). (b) Mesotexture shows grain boundary relationships (shown by dotted and black thick lines) which help reveal grain boundary character that is statistically described in Figures 1(d) misorientation distribution function (MODF) and Figure 1(e) histograms of misorientation angle.



Figure 1(c). Pole figure with point plots and a Gaussian distribution that shows the macrotexture.



Figure 1(d). Misorientation distribution functions (MODF).



Figure 1(e). Histograms of misorientation angle.

1) Electron Back-scatter Diffraction Pattern (EBSP): Incoherent scattering of elastically scattered electrons in a thick specimen gives rise to Kikuchi patterns. These electrons are sometimes called diffusely scattered electrons. Elastic scattering takes place due to deflection of the electrons by the positive charge of the nucleus, taking into account the negative charge of the revolving electrons and near collision with electrons. The interaction volume[•] in the solid specimen gives rise to the effect of back-scattering. Most of these electrons are deviated by about 90° from the incident direction during backscattering. These electrons then undergo both elastic and inelastic scattering in the interaction volume before they re-enter the unit hemisphere of solid angle from which they entered²⁴. The diffracted beam will be in the form of cones which when projected onto a screen in 2-dimension appears to look like parabolas. These parabolas appear as two parallel lines (called Kikuchi lines) if one is to view them near the optic axis. These Kikuchi lines are fixed (both angular and spatial) for a particular crystal structure and they are the projections of the geometry of the lattice planes in a crystal²³. The angles between the Kikuchi lines (or Kikuchi bands as they are sometimes called) along with the stereographic projection are used to index the Electron Back-scatter Diffraction pattern (EBSP) or Kikuchi pattern. (Figure 2)

Interaction volume is the volume inside the specimen in which interactions between the incident electrons and those from the specimen take place. This volume depends on 1) the atomic number of the material of the specimen; higher atomic number implies more electron absorbance and hence a smaller interaction volume, 2) the accelerating voltage; higher voltages implies more penetration and generation of a larger interaction volume and 3) the angle of incidence for the electron beam; the greater the angle (further from normal) the smaller the interaction volume.



(a)



Figure 2. Kikuchi pattern of tin crystal. (a) Pattern showing Kikuchi bands and (b) after indexing.

2) Microtexture Data Collection: Automated EBSP (Electron Backscattered Pattern) is the most preferred technique to study micro and meso textures. The essential components for this method of data collection are an SEM with a stage tilt control, a phosphor screen viewed with a CCD video camera and a TV, all interfaced with a computer for on-line data analysis. Figure 3 is a simple sketch illustrating EBSP formation and data collection. The specimen is mounted in a holder and tilted to an angle of 70°. The electron beam trajectory strikes the surface of the specimen emitting backscattered electrons, which hit the phosphor screen and are converted into optical light pulses transmitted through optical fiber cable, which are then reconverted into an electronic signal and displayed on a TV screen. The computer captures the screen image (which is typically averaged to improve resolution) and indexes the pattern in real time to give a crystal orientation recorded in terms of "Euler" angles. The beam then moves to the next spot and the procedure is repeated.



Figure 3. Typical setup for EBSP.

3) Sample Preparation and Mounting: The sample preparation for EBSP is relatively straightforward. The surface need not be absolutely flat, though it needs to be free from any oxide layer, impurities and contamination since the penetration depth for back-scatter electrons is approximately 20nm. This can be easily accomplished by ultrasonic cleaning, plasma cleaning (incases where high temperatures is not a problem since surface temperature of 300°C are reached) followed by storing the specimen under a nonoxidizing environment²⁵. Surface deformation can cause difficulty in indexing the EBSP pattern (which may get distorted or become less distinct) and there are various levels of deformation, which can be tolerated for indexing. Since the angular relationships between bands remains unchanged, it might be possible to index the EBSP, however the confidence in indexing depends on the extent of deformation, as the intensity of the Kikuchi bands decreases with increase in deformation. It is believed that the usual metallographically prepared specimens are not suitable for EBSP and additional steps are required. As a general rule it is proposed that all specimens undergoing EBSP analysis be chemically etched, electro polished or metallographically polished with 20nm silica particles in an alkali suspension²⁶. Once the specimen is ready, it needs to be mounted onto the sample holder taking care that there is no surface damage or contamination during mounting.

The specimen is mounted in a specimen holder, which is designed so that the surface of the specimen makes an angle of about 70° ²³ with the horizontal axis. Precise tilt is an integral part of the calibration setup. Calibration is achieved using a Silicon single crystal with [001] as the surface normal. The geometrical aspects and procedure for calibration can be found in Ref 23.

Spatial resolution is dependent on the acceleration voltage, probe current, working distance and tilt angle. Higher acceleration voltage is preferred to increase the size and depth of the interaction volume, however 20-25kV seems to be an optimum value. Probe currents of the order of about 5mA or higher are considered optimal. Shorter working distances are preferred to minimize focusing problems. A decrease in the angle from 75° to 60° results in the increase in spatial resolution, with a loss in the proportion of backscatter electrons to absorbed electrons^{27,28}. Hence a 70° angle is considered most favorable²⁹ in majority of cases, though in a particular system one can obtain better conditions by tweaking one of these parameters with a trial and error approach.

4) Pattern Recognition: The EBSP pattern can be used to identify crystal structure. The EBSP pattern first obtained is studied for rotation angles and mirror planes which can be easily accomplished by looking at the intersecting Kikuchi bands. Further, calculations of angles between various bands on the EBSP and the spacing between parallel bands can be determined. Further information on crystallographic details that can be obtained from an EBSP are found in references^{25,30-32}.

Once all the above steps are completed and a region of interest is located, the beam is rastered over a chosen area of specimen. The scan coils of the SEM are turned off (i.e. the spot mode), and the beam interacts with only a small volume of the specimen to display an image of the EBSP on a low-light TV monitor. The computer program indexes the zone axes and Kikuchi bands and measures the inter-axial and inter-planer angles. The orientation is determined in real time and a pixel of orientation data is stored before the beam is automatically positioned to the next spot and the process repeated.

Being an automated process, several such areas can be scanned one after the other. The dataset is subsequently post-processed to generate maps and plots.

OIM (orientation imaging microscopy) assigns a color to particular crystal orientation, so that a given crystal orientation has the same color. Misorientations between grains can be visualized by the degree of color difference. Thus microstructure and crystal orientation are simultaneously displayed on a map. Figure 1 shown earlier reveals the different ways information obtained from an OIM scan can be processed for better understanding of the data. Figure 7 shows crystal orientations being expressed using color-coded maps and inverse pole figures. Grain size, shape, distribution, etc., are available and can be deduced with the software. The distribution of grain boundary misorientations (i.e. high, low, twin, special boundaries) are expressed as misorientation distribution functions (MODF) or histograms of misorientation angle.

The fast and relatively easy automated setup, along with simple sample preparation and user friendliness, EBSP allows researchers to explore topics such as recrystallization, grain size, grain growth and microstructural evolution. The software allows us to represent texture in terms of pole figures or OIM maps. The grain boundary distribution can be studied with the help of misorientation distribution functions (MODF) and histograms of misorientation angles. The plots along with the OIM maps serve as effective tools to understand grain boundary formation and changes. Hence OIM is an indispensable tool for better understanding and characterization of the highly dynamic microstructure of solder in a joint configuration.
1.2 LITERATURE REVIEW

A) Thermal and Mechanical Processes Experienced by a Solder Joint in Service:

1) Aging:

All solder joints undergo aging with time, which results in the alteration of the microstructure. Depending on the temperature to which the solder joint is exposed during service, the rate of aging can differ. Higher temperatures naturally cause faster aging. Solder joints in typical automotive under-the-hood service conditions are exposed to temperatures of around 150°C, while those in computer related applications experience temperatures of 50-80°C. Such high temperatures cause dynamic recrystallization and grain growth to occur within the solder microstructure. This change in the microstructure alters the properties of the solder joint and changes the way the solder material would respond to further deformation processes.

In eutectic Sn-Ag solder joints aging produces coarsening of the Ag₃Sn particles in the solder matrix³³. The intermetallic layers of Cu₆Sn₅ and Cu₃Sn also grow in thickness from approximately 0.7 μ m to about 10 μ m after aging at 150°C for 1000 hours³³. Jang et.al. observed a 2 μ m thick Cu₆Sn₅ layer after a 60 seconds reflow of a eutectic Sn-Ag solder at 250°C³⁴.

The material in the joint configuration can undergo three basic types of stress / strain imposition: 1) time dependent monotonic loading e.g. tensile loading, shear loading and creep and 2) cyclic deformation as in fatigue³⁴.

2) Creep:

Creep is the progressive deformation of a material under constant stress at an elevated constant temperature. It is typically seen that the strength of a material decreases with increase in temperature, which is due to a corresponding increase in diffusion of atoms at elevated temperatures. This can strongly affect the mechanical properties of the material. Higher temperatures also give additional mobility to dislocations, which can move by the climb process and additional slip-systems can come into play³⁶. At elevated temperatures the strength of the material depends on both the strain rate and the time for which it is being strained. Therefore the number of variables increases as the temperature increases.

Creep deformation becomes important in human time frames at temperatures greater than $0.5T_m$ (where T_m is the melting point of the material in absolute temperatue). The creep experiment involves loading a tensile specimen at a constant temperature (>0.5T_m) and at a constant load. The strain (extension) of the material is measured as a function of time. Hence studies based on creep experiments can take several hours to even years to achieve the desired information. A typical creep curve is shown in Figure 4. The curve can be ideally divided into three regions; primary, secondary and tertiary creep. The creep rate given by $d\epsilon/dt$ decreases rapidly with time initially during primary creep and is approximately constant in the secondary creep region. The initiation of tertiary creep is an important point on the creep curve since after this point the creep rate increases steadily until the specimen ultimately fractures. A long secondary creep period or a very low minimal creep rate is essential for longer service life of the specimen.



Time, t

Figure 4. Typical creep curve showing the three stages of creep. Curve A is constant-load test; curve B, constant-stress test³⁶.

Representation of the secondary creep deformation by a mathematical equation commonly uses the Dorn equation,^{37,38} which is given by:

$$\frac{d\gamma_s}{dt} = \frac{CGb}{kT} (b/d)^p (\tau/G)^n D_o \exp(-Q/RT)$$

where,

 $d\gamma_s / dt =$ steady state creep rate,

G = shear modulus,

b = Burger's vector,

T = absolute temperature,

d = grain size,

 τ = applied stress,

 $D_o =$ frequency factor,

Q = activation energy for the deformation process,

n = strain exponent,

p = grain size exponent, and

C = constant characteristic of the underlying mechanism.

Darveaux and Banerji³⁹ developed constitutive relations, which describe the behavior of solder joints under a wide range of conditions. They showed that eutectic Sn-Ag solder absorbed larger strain prior to failure as compared to 60Pb-40Sn solder joints.

However, creep properties are affected by several factors. Higher melting temperature solders exhibit lesser creep deformation if the applied strain is small (which is usually the case in typical electronic packages)⁴⁰. Guo et.al.⁴¹ and Choi et.al.^{42,43} have shown that recently developed composite solders and alloyed solders are more creep

resistant than non-composite counterparts due to the intermetallic interphase layer formed between the solder matrix and the composite reinforcement.

Testing solder joints rather than bulk solders is the best way to study the deformation and microstructural effects of solder, since the microstructure of bulk specimens is different from solder joints. Solder interconnect material in the joint configuration experiences several convoluted and varied effects, which lead to their deformation and ultimate failure that cannot be realized if solder material is tested alone³⁹. Darveaux and Murty's⁴⁴ test results show that creep properties of solder in joint configuration is quite different than bulk solder, with joints showing better creep resistance due to the effect of joint constraints.

Creep seems to be the dominant deformation process as one reaches temperatures greater than $0.5T_m$. Igoshev et.al.⁴⁵ show that accumulation of grain boundary defects start early during creep and last for 70-80% of the specimen's life. Depending on the stress states during creep at high temperatures, failure can take place via transcrystalline (at lower stresses) or inter-crystalline (at higher stresses) cracking. Darvaeux and Banerji³⁹ have shown that dislocation climb is the main factor contributing to creep deformation, however stress states in the solder joint can change and failure can occur by fatigue.

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3) Thermomechanical Fatigue (TMF):

Fatigue failures are those wherein the material subjected to repeated fluctuating stress states fails at a much lower stress than that required to cause failure in a single application of load³⁶. Figure 5 gives some examples of fatigue cycles. Figure 5(a) shows a sinusoidal curve with complete reversal of stresses. Figure 5(b) shows repeated reversals in stress however the stress range is always on the positive (tensile) side. Figure 5(c) shows a random fatigue cycle and Figure 5(d) shows cyclic deformation with hold times.



Figure 5. Typical fatigue stress cycles. (a) Repeated reversed sinusoidal stress curve with complete reversal of stresses, (b) repeated stress reversals on the positive (tensile) side, (c) random fatigue cycle and (d) cyclic deformation with hold times.

Though Coffin-Manson's law states that the fatigue cracking in steel is related to the total plastic strain, in solder alloys with low melting temperatures, the fatigue behavior is related to the equivalent strain amplitude and change in the properties with temperature. The fracture is not only dependent on fatigue undergone by the joint but is also dependent on creep at such temperatures⁴⁶. Harada and Satoh⁴⁶ have developed an empirical equation to estimate the thermal fatigue life of 96.5Sn-3.5Ag solder. Their equation gives a modeling approach to crack growth as a result of fatigue. It does not attempt to model crack nucleation, which is a more critical phenomenon with reference to solder interconnects^{11,12}. The equation is given by

$$N_f = C \ln((Aa_f + B)/B)f^m \cdot exp(Q/kT_{max}) \Delta \varepsilon eqmax^{-n}$$

where,

 N_f = thermal fatigue life,

 $\Delta \varepsilon eqmax = maximum equivalent strain range,$

Q =activation energy,

 T_{max} = maximum temperature,

f = frequency of temperature cycle,

k = Boltzmann's constant,

A = 5.87,

$$B = 1.40 \times 10^3$$
,

C = 0.234,

n = 1.2, and

m = 0.33.

Fatigue strength of a material usually increases with a decrease in temperature. At higher temperatures, transition from fatigue failure to creep can occur and transcrystalline fatigue failure may change to intercrystalline creep failure³⁶. In general a material with high temperature creep strength will have better fatigue properties at high temperatures. Fine grain size gives better fatigue properties at lower temperature. As the temperature increases, the differences in fatigue properties of coarse and fine grain sized materials are reduced with increasing temperature until high temperatures are reached. At high temperatures creep dominates and hence a large grain size is preferred for creep resistance.

The stresses produced during fatigue need not only be mechanically induced. Fluctuating thermal stresses induced by differential heating and cooling and due to differences in the coefficient of thermal expansion (CTE) in the material can lead to stress accumulation, which leads to failure of the material. Such repeated application of thermal stresses of low magnitude that leads to failure is called thermomechanical fatigue. A simple equation showing the stress developed in a constrained material cycled between two different temperature extremes is given by:

$\sigma = \alpha E \Delta T,$

where,

 σ = stress developed in the material,

 α = coefficient of thermal expansion,

E = elastic modulus, and

 ΔT = change in temperature.

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The life prediction of a solder joint is dependent on the susceptibility of a solder joint to TMF. TMF in a solder joint can occur due to the cyclic changes in the temperature experienced by the joint. Also the various constituents in the electronic assembly play an important role during such temperature fluctuations, since different constituents have different CTE. The plastic strain accumulation is thus a function of the total change in the temperature, the rate and the magnitude at which this change occurs and the inherent constraints in the electronic assembly and its interaction with the time dependent and cyclic deformation processes occurring in the solder alloy⁴⁷. A constitutive equation taking into account these several processes is needed and a model for this needs to be developed.

B) Eutectic Tin-Silver Solder

1) Crystal Structure and Directional Properties:

Eutectic Sn-Ag solder (96.5%Sn-3.5%Ag in wt.%) consists of two phases, β -Sn and the Ag₃Sn intermetallic phase. The β -Tin has a Body-Centered Tetragonal unit cell with four atoms occupying the positions 0 0 0, 0 ½ ¼, ½ 0 ¼, ½ ½ ½ in the unit cell as shown in Figure 6⁴⁸. It is a squashed tetragonal cell with a=b=5.8197 Å and c=3.175 Å⁴⁸, with c/a = 0.5456.



Figure 6. β -Sn unit cell (lattice parameters, a=b= 5.8197 Å and c=3.175 Å⁴⁸).

The Sn unit cell has four closed packed directions⁴⁹; [001], [111], [100] and [101] and four closed packed planes⁴⁹; (100), (110), (101) and (121). At different temperatures different slip systems come into play. Table II shows the various possible slip systems at different temperatures. Twin formation is seen frequently in tin and Table II also shows the possible twinning plane and direction for tin. Twins have low energies associated with them and if a twin is formed during solidification, internal strains at its boundaries are so small or absent that recrystallization is not promoted in such cases^{50,51}.

	Gli	Twinning Systems				
Low Temp. (~20°C)			High Tem	p. (~150°C)		
Slip Plane	Slip Direction	Critical Stress, kg/mm ² *	Slip Plane ^{49,53}	Slip Direction	Plane ⁵⁰	Direction ⁵⁰
(100)	[001]	0.19	(110)	[-111]	(301)	[-301]
(110)	[001]	0.13				
(101)	[10-1]	0.16				
(121)	[10-1]	0.17				

Table II. Possible slip and twin systems in β -Sn.

* Critical stress in kg/mm² at 20°C and 0.01% impurity.

All data can be found in Ref.52, except the ones specially marked.

The Young's Modulus in the a-direction is 54 GPa and in the c-direction is 85 GPa as shown in Table III⁵⁴. The CTE for tin in the a-direction is 15.45 x 10^{-6} /°C and 30.5 x 10^{-6} /°C in the c-direction⁵⁴. However, tin is highly anisotropic and the value of

modulus changes with crystal direction as calculated by Lee et.al.⁹ The anisotropy of tin in the solder joint can lead to very high stress accumulation, which can contribute to the failure of the solder joint, assuming elastic deformation is imposed on bi-crystals.

The Ag₃Sn has an orthorhombic structure with a=5.968 Å, b=4.7802 Å, c=5.1843 Å⁵⁵. The Ag₃Sn undergoes a transformation at about 60°C⁵⁵. However the nature of this transformation is unknown and X-ray studies have shown that there is no lattice change⁵⁷.

	Cu	Sn		Cu ₆ Sn ₅	Cu ₃ Sn
	(Polycrystal)	(Single crystal)		(Polycrystal)	(Polycrystal)
		a-dir.	c-dir.		
Young's Modulus, GPa	117	85	54	85.56	108.3
Shear Modulus, GPa				50.21	42.41
Hardness, Gpa	2.94	0.98		3.71	
Thermal Expansion, x	17.1	15.4	30.5	16.3	19.0
10 ⁻⁶ /°C					
Thermal Diffusivity,				0.145	0.24
cm ² /sec					
Resistivity, μΩ-cm	1.7	11.5		17.5	8.93
Density, g/cc	8.9	7.3		8.28	8.9

Table III. Constants for various solder joint constituents⁵⁴.

2) Microstructure of Eutectic Tin-Silver:

The microstructure of eutectic Sn-Ag solder consists of Ag₃Sn precipitated in a tin matrix surrounding the so-called tin cells. The grain boundaries are difficult to reveal, as effective etchants for this system are not well established. Depending on the cooling rate one can obtain variations of this basic microstructure. Under equilibrium cooling the most favorable growth direction for tin is along the $[110]^{58}$ crystal direction, which gives rise to dendritic arms in the microstructure. With rapid cooling one sees a more or less equiaxed microstructure, with \Box tin cells surrounded by the eutectic mixture. Microstructures of eutectic Sn-Ag solder joint are given in Figure 7. It is evident that a microstructure produced with a low soldering temperature and a fast cooling rate would yield an optimal solder joint since low soldering temperature implies lesser superheat into the system and similarly a fast cooling rate would prevent dendrite formation and produce a more equiaxed microstructure^{33,59}.



Figure 7. SEM micrographs revealing microstructure of eutectic Sn-Ag solder. (a) Solder joint showing equiaxed Sn-cells surrounded by Ag_3Sn intermetallic, (b) same joint revealing dendritic Sn-cells in some other region and (c,d,e and f) higher magnification micrographs of particular regions of the same joint. These features are common to all solder joints depending on the maximum temperature reached during soldering and the cooling rate.

The microstructure in the joint configuration with copper substrates is slightly different due to dissolution of copper into tin, which gives rise to scallop shaped Cu₆Sn₅ and Cu₃Sn intermetallic phases at the copper-tin interphase as well as dispersed hexagonal rod-like Cu₆Sn₅ particles in the tin matrix. Cu₆Sn₅ is found at the interface with the Sn-rich phase and the Cu₃Sn found between Cu and Cu₆Sn₅ (and is closer to the Cu-rich phase to maintain thermodynamic equilibrium). The lattice parameters for hexagonal Cu₆Sn₅ are as follows: a = 4.192 Å and c = 5.037 Å⁵⁵. The thickness of this intermetallic layer is dependent on the heating and cooling rate used during joint fabrication. A higher temperature facilitates a thicker intermetallic layer. Depending on the amount of heat input into the system during the entire soldering process as explained by Guo et.al.⁶⁰, the total volume of the secondary intermetallic phases of Sn with Cu can be controlled. Higher heat input (thermal flux) allows greater diffusion and hence thicker intermetallic interphase layers. Aging at higher temperatures also increases the thickness of this layer as well as the size of Cu₆Sn₅ particles due to the increased solubility and faster diffusion of copper into tin.

The properties of the solder are affected by the microstructure and the constraints imposed by the copper substrate in the joint configuration. The microstructure can be changed and controlled by addition of several alloying elements (Cu, Ni, Ag, Bi, Sb, In) in small proportions. The Ag₃Sn in the eutectic Sn-Ag solder matrix increases the hardness and strength of the solder. Copper addition (up to 1wt.%) slightly adds to the strength of Sn-3.5Ag solder joint⁶¹. The intermetallic Cu₆Sn₅ formed due to the dissolution of the Cu substrate increases the strength of the solder joint even more with a loss in ductility.

C) Lead-based Solders

1) Microstructure of Lead-based Solders:

Lead-free solders have posed several metallurgical challenges to the research community. Addition of secondary constituents like copper, nickel, bismuth, etc. to the eutectic Sn-Ag solder matrix to enhance wettablility and decrease the melting point, has led to the formation of intermetallics at the interphase / boundary and in the solder matrix. These intermetallics change / affect the microstructure of the joint during formation and results in variation of performance. It has been observed that the microstructure in case of Pb-based solders affects the fatigue properties of the solder¹³. Hence there is need to study the evolution and changes that occur in the microstructure and mechanical properties due to such additions. Lead-based solders are well studied in this regard and though there is a distinct difference in the behavior of lead-based solders and lead-free solders with respect to microstructure and its evolution, the knowledge and insight gained from studying lead-based solders can greatly enhance our understanding of how the microstructure of lead-free solders evolve / change.

Microstructure of the Sn-Pb eutectic alloy (63% Sn-37% Pb): The solder joint is a composite with solid, hard metallic elements bound in a relatively softer solder matrix forming the bulk of the interconnect. Intermetallics formed in the solder joint, affect and present different properties than those exhibited by the matrix tested alone. The solidification microstructure is governed essentially by the rate of cooling. Faster cooled joints reveal a fine equiaxed microstructure whereas slower cooling leads to a lamellar microstructure. Synonymously, smaller joints cool faster and therefore exhibit a finer microstructure as compared to thicker joints (~0.5-1mm thick), which reveal a coarse lamellar microstructure. Faster cooling rate implies a lesser time for diffusion and hence slower growth of the lamellae². Since solder joints are used at high homologous temperatures in most cases, the microstructure evolves with time and hence affects the way the joint behaves and contributes to be a strong interconnect.

Eutectic microstructure: Eutectic Sn-Pb is the most popular solder being used. The microstructure is well studied and it gives valuable information to base our studies upon. Though there is no direct correlation between Sn-Pb solders and Sn-Ag solders, the information and data we can obtain from Sn-Pb solders is invaluable.

Morris et.al.¹⁴ have studied the microstructure of Sn-Pb solders and have seen how the microstructure affects the joint properties. On relatively slow cooling, eutectic Sn-Pb solders give rise to a microstructure shown in Figure 8(a), which consists of lamellae of the two solid solutions simultaneously crystallizing and forming parallel plate-like colonies when the two phases are present in equal volumes. This is the classic "eutectic" microstructure. If one of the two phases dominates then the other forms a rodlike morphology within the matrix of the larger volume phase.

Mei et.al.⁶² has also compared the effect of cooling rate on the microstructure of the Sn-Pb solder. Slow cooling gives the eutectic microstructure as above. Very rapid cooling however results in the Pb-rich and Sn-rich phases, which solidify as equiaxed grains mixed with each other (See Figure 8(c)). Intermediate cooling rate gives rise to variations between the eutectic and the rapid solidification microstructure. Eutectic lamellar microstructure is typically observed on the PCB and on ceramic chip leads whereas the fine equiaxed microstructure is seen on surface mount components⁶¹ (surface

mount components have a smaller mass and faster heat dissipation as compared to PCBs and large ceramic chip leads).



Figure 8. Effect of cooling rate on the microstructure of eutectic Sn-Pb solder. (a) Furnace cooling, (b) air cooling and (c) quenching (from Ref.14).

2) Evolution of Sn-Pb Microstructure:

During solidification of the solder, when the temperature is lowered below the eutectic temperature, the equilibrium solute content of the primary solution is greatly decreased. The super saturation of the solute results in the precipitation of the solute in the matrix as fine particles since solid-state diffusion is relatively slow. Hence one finds small crystallites of Sn distributed in the Pb-rich phase². The Sn-rich phase is more "pure" than the Pb-rich phase due to lesser dissolution of Pb in Sn. The volume of the Sn rich phase to the Pb rich phase is 73:27 for 63Sn-37Pb solders².

The eutectic microstructure with plate-like lamellae is out of equilibrium with respect to lowering the total surface energy of the interfacial area per unit volume. There are two ways to address this issue to achieve thermodynamic equilibrium with respect to surface and interfacial energy. Firstly, the lamellae can be converted into spheroidal particles, which would coarsen and grow with time. Such spherodization usually initiates at the boundary between the so-called colonies of the eutectic microstructure, where there are several differently oriented colonies approaching each other. The spherodization once initiated at such boundaries slowly consumes the entire grain. The second way the microstructure can change is when the joint undergoes plastic deformation at high homologous temperatures. In such a case the solder material may recrystallize and evolve into finer equiaxed grains⁶³⁻⁶⁷. The recrystallization occurs along bands of shear that develop due to inhomogeneous deformation in the solder joint. This sudden change can deteriorate the fatigue life of such solder drastically.

As Morris et.al.¹⁴ point out, fine, equiaxed microstructures obtained by rapid solidification have smaller interfacial surface area per unit volume, e.g. Figures 8(a and

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c), which in turn helps to lower interfacial surface energy. Such grains do not undergo recrystallization under load, since deformation causes grain boundary sliding and dislocation activity that helps keep the grain size small. However they can grow and coarsen which can affect the creep properties and other properties related to grain size.

Finally, at high homologous temperatures, the rate of diffusion, especially in the intermetallic layer in case of Cu (from component) and Sn (from the solder), is very high. This causes increase in the intermetallic layer and simultaneous depletion of the Sn from the near intermetallic region, which gives rise to a Pb-rich layer/band near the interphase. This Pb-rich layer is comparatively very soft to the Cu_6Sn_5 intermetallic layer and hence there is a greater chance of crack nucleation, de-bonding and failure in this region. Thus a chemically inhomogeneous solder is created near the joint interfaces.

3) Effect of Eutectic Sn-Pb Microstructure on the Mechanical Properties of Solder Joints:

a) Creep: Figure 9 shows the creep curve adapted from Ref. 68 to show the creep behavior of a fine-grained eutectic Sn-Pb solder. In the high stress regime, the stress exponent is in the range of 4-7 and the activation energy is close to that of bulk diffusion. The deformation within the grains is controlled by bulk diffusion and it is stated that dislocation climb within the grains is the controlling creep mechanism. At slightly lower stresses, the stress exponent is 2 and the activation energy is close to grain boundary diffusion. One sees deformation predominantly concentrated at the grain boundaries, which makes grain boundary sliding the creep rate / deformation controlling mechanism. At still lower stresses the stress exponent has a value of about 3. The activation energy

has a value close to that of bulk diffusion. The creep rate controlling mechanism is believed to be bulk deformation, from dislocations that drag solute atoms, which accommodates deformation concentrated near the grain boundaries and hence maintains contact between grains.



Figure 9. Schematic illustrating the creep properties of fine-grained eutectic Sn-Pb solder from Ref. 68.

The microstructure of the solder affects the creep behavior and creep rate. An increase in the size of the colony of the eutectic Sn-Pb solder causes hardening of the solder, especially at lower strain rates as seen in Figure 9⁶⁹ and the intermediate creep rate in this case disappears (See dashed line in Figure 9). This phenomenon is particularly seen in eutectic Sn-Pb compositions, where the microstructure is relatively sensitive to the cooling rate. Addition of other elements can affect the creep rate by changing the grain size and solidification microstructure. Reynolds et.al.⁷⁰ have reason to believe that the creep behavior is affected by the creep behavior of the individual phases present in the joint¹⁴. The phase dominating the creep behavior can be identified by the microstructure. Hence it is essential to customize the microstructure to obtain the desired mechanical properties from the solder.

b) Shear Strength: Similar to dependence of creep behavior on microstructure, the shear stress is also affected by the microstructural changes. A fine grained joint in shear deforms by grain boundary sliding and it can sustain large deformations prior to failure. In this case the solder behaves in the superplastic manner. In case of the eutectic Sn-Pb microstructure, the shear strain is heterogeneously distributed throughout the microstructure, which allows partial recrystallization and shear band formation in the solder. These bands have finely divided grains, which then undergo superplastic deformation. The mechanical inhomogeneity causes further redistribution of straining in the joint and finally failure occurs along these shear bands¹⁴. It is observed that superplastically deformed material can sustain larger amount of strain to failure and hence crack growth rates are not fast.

c) Fatigue: Morris and Reynolds have stated in their paper¹⁴ that "microstructure should resist the formation of the persistent slip bands that concentrate cyclic deformation and nucleate fatigue cracks. To achieve this goal, one would have to homogenize the deformation and avoid the development of local stress concentrations within the material." From this point of view eutectic Sn-Pb solders are rather not suitable, especially due to the fact that solders usually experience high homologous temperatures in service. Recrystallization due to inhomogeneous deformation into a fine grain size under such conditions would lead to a soft microstructure. This creates early crack nucleation and subsequent failure in the interconnect. To achieve better fatigue properties in eutectic Sn-Pb solders, it is recommended that the eutectic microstructure be converted into a more equiaxed microstructure to homogenize the strain. This can be achieved by using a fast enough cooling rate so that a more equiaxed and homogeneous microstructure is obtained. However, to achieve a fine equiaxed microstructure so that the joint would be able to undergo superplastic deformation (which is possible in electronic circuitry owing to the usually low strain rates) and to prevent grain growth (due to the high temperatures experience by the solder joints) which will lead to decrease in the fatigue resistance of the solder joint, addition of other elements, which inhibit the formation of the eutectic microstructure, can prove very useful. Another way to improve fatigue resistance is to introduce a dispersed second phase, which will defer deformation of the matrix, however being softer than the matrix this secondary phase should be able to undergo deformation⁷¹.

To achieve better creep and fatigue properties it is important to achieve a stable fine grain microstructure, which does not coarsen with time, so as to facilitate

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homogeneous deformation in the solder joint. This is comparable to a similar phenomenon occurring in high temperature superalloys, which were developed for aerospace applications. Hence approaches used by researchers for aerospace alloys could also be used for solder joints. Out of the many such approaches, dispersion strengthening and precipitation hardening are two possible techniques. In precipitation hardening a fine secondary phase precipitates out homogeneously throughout the microstructure from the solid solution. In case of dispersion hardening, a suitable phase needs to be dispersed in the matrix by external means. These dispersed particles have to be of the appropriate size and the distance between the dispersoids have be to optimized so that the hard particles can stop the movement of the dislocations and hence hinder slip^{72,73}. Betrabet et.al.⁷² discuss the use of such approaches in their paper with respect to the advantages and disadvantages of such dispersoids and properties inherently required by the secondary phases in the eutectic Sn-Pb system.

d) Comparison of Sn-Pb and Sn-Ag solders: Satoh et.al.⁷⁴, Hwang et.al.⁷⁵ and Thwaites et.al,⁷⁶ have reviewed the tensile behavior of Sn-Pb solder. On comparison of the two solders for tensile properties, Sn-3.5Ag solders have comparable or slightly higher tensile strength than Sn-Pb solders^{2,74-76}.

Shear strengths of Sn-3.5Ag are comparable to those of Sn-Pb. In shear the plastic instabilities of the solder do not interfere with strength². During elongation experiments carried out by Satoh et.al.⁷⁴, Thwaites et.al.⁷⁶ and Timlinson et.al.⁷⁷, Sn-Ag had comparable elongation to Sn-Pb at room temperature and moderate strain rates, however Sn-Ag is less strain rate sensitive i.e. there will be greater elongations at higher strain rate and lower amount of elongations at lower strain rate⁷⁷.

Creep resistance of Sn-Ag solder is better than Sn-Pb in the 25-100°C range. In case of isothermal fatigue, eutectic Sn-Pb with a fine-grained equiaxed microstructure has a better fatigue life as compared to one with a lamellar structure. Sn-Ag is much better in fatigue as compared to Sn-Pb at high shear strain amplitudes as indicated by Guo et.al.⁷⁸. He attributes the better fatigue resistance of Sn-Ag solder to the resistance of Sn-Ag to crack initiation, rather than crack propagation, because Sn-Ag has the lowest crack growth resistance for the alloys studied by him. Other studies also show that Sn-Ag is more fatigue resistant at room temperature and at about 100°C, compared to Sn-Pb solders⁷⁶. In Sn-Pb solders localized microstructural coarsening occurs which decreases its fatigue resistance and leads to failure in the joint, which is not the case in Sn-Ag solders⁷⁹. However, more detailed studies are warranted in this respect to assess the mechanisms of thermomechanical fatigue damage in lead free solder.

1.3 AIM

Reliability of Sn-Ag solders as interconnects is vital to electronic packaging. So far there has been no model explaining physical understanding of failure mechanisms persistent in Sn-Ag solder. Scanning electron micrographs by themselves give no information on the crystallographic texture of the solder joint. Characterizing the microstructure crystallographically is critical to understand deformation processes occurring in the solder joints, as most of the mechanical properties are affected in some way or other by the microstructure of the solder joint. OIM using EBSP is an effective mean to identify physical changes in microstructure of Sn-Ag solder, which is different from Sn-Pb solder. This can give an insight on how to enhance the mechanical properties and achieve better solder interconnects. Hence, OIM is an ideal tool to study crystallographic orientations, grain shape and grain boundary changes resulting from solidification and subsequent deformation. Hence the subsequent investigations were carried out.

"This research is the first attempt of its kind that aims at characterizing tinbased eutectic Sn-Ag solder joints in the as-fabricated condition and subsequently subjected to aging, creep and thermomechanical fatigue processes using the Orientation Imaging Microscopy (OIM) technique, which utilizes Electron Backscattered Patterns (EBSP)."

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EXPERIMENTAL PROCEDURES

2.1 SOLDER JOINT PREPARATION

Copper half dog bone specimens (shown in Figure 10) were made by electro discharge machining (EDM) from a copper sheet of 0.5mm thickness. The copper dog bones were cleaned in 50% Nitric Acid (HNO_3) to remove the oxide layer that could create pores and de-wetting and hinder joint formation. The dog bones were placed in the apparatus developed by Rhee at Michigan State University, which eliminates the need for a mask that was required in previous studies^{1,2}. This method facilitates the making of solder joints of approximately 100µm with reasonable consistency (see fixture shown in Figure 10). Once the lower dog bone piece was laid in the apparatus, the eutectic Sn-Ag solder paste of approximately 0.2mm³ volume was placed on the free copper surface. The upper dog bone piece was then placed on top and the entire assembly was placed on a heated hot plate. The assembly was kept on the hot plate for about 50 seconds during which time the temperature reached 221°C (the melting point of the eutectic Sn-Ag solder). The temperature was further allowed to reach 260°C in another 5 seconds and then the entire assembly was removed from the hot plate and transferred to an Aluminum plate where it was allowed to cool down to room temperature. The initial cooling rate was about 150°C/min till about 150°C, after which it took about 7 minutes to cool down to room temperature.

The joint was taken out of the apparatus, taking care that no mechanical damage was induced and mounted in a jig for polishing. Though most of the solder (0.2mm³ volume) was involved in making the joint, some of the solder made up the fillets on

either side of the joint and some was extruded out from the edges when the upper dog bone was overlaid on the solder. This was ground off during polishing.



(a)



Figure 10. (a) Solder joint configuration. (b) Fixture for manufacturing solder joints used in this study. Copper dog bones were about 500 μ m in thickness, while the glass plate was ground to about 600 μ m thickness, which facilitates making 100 μ m thick solder joints.

Since OIM studies require the surface to be ideally free of oxide and deformation, careful polishing is needed. Polishing was achieved by mounting the specimen in a special jig (made of Aluminum) that prevents mechanical deformation during grinding and polishing. Figure 11 illustrates the way the joint was placed in the jig for the polishing operation. The jig was designed to support the joint on the flat edges, so that no deformation or bending was induced in the joint. To accommodate for the thickness of the joint, two identical copper strips (25mm x 3mm x 0.5mm, same as the dog bone halves without the extension used to make the joint) were placed alongside the tail of the copper dog bone on the joint side to prevent any bending or twisting of the joint during polishing. The edges of these joints were metallographically polished progressively with 600 and 1000 grit sandpapers. The specimen / jig were handled carefully to prevent specimen damage. Then, the same procedure was repeated on the other side of the specimen, which was then progressively polished with a 0.3µm alumina suspension followed by 0.05µm silica in an alkaline suspension, which was the final step in polishing. The side that underwent final 0.05µm polishing was used to study the microstructural evolution, whereas the other side, which had a flat surface, facilitated mounting the specimen on the stage during OIM. The specimen was observed under an optical microscope periodically while polishing. All specimens were metallographically hand polished with minimal force application during polishing. The final two steps were achieved by placing the joint (in the jig) with the surface to be polished on velvet paper mounted on a 6cm x 1cm x 0.5cm Aluminum block, applying the polishing media and then moving the jig back and forth without applying any force. The only force applied

was that of the weight of the jig, which is about 30 grams. Due to the hand support the force actually applied was in the range of 0.196-0.245N.



Figure 11. Jig used for polishing specimens showing a specimen with copper pieces used as backing to avoid bending and twisting of the joint. Middle piece is not moved because it supports the solder joint during polishing.

Preliminary OIM studies carried out on polished specimens indicated that no additional electropolishing was necessary to enhance the sharpness of the EBSP's, since they were sharp enough to determine the crystal orientation for 60-90%^{3,4,5} of the scanned positions. The remaining 40-10% of the non-indexed positions were attributed to porosity, grain or phase boundaries, second phases, oxidation or locally highly deformed regions (for specimens after deformation). The solder joint specimen was then cleaned in methanol or acetone in an ultrasonic cleaner and then dried in compressed air and stored in a refrigerator until future testing. The single shear lap joints made with eutectic tin silver (3.5 wt. %Ag) lead free solder had a solder area of about 1 mm² and a thickness ranging from 100-180µm.

2.2 THERMAL AND MECHANICAL TESTING

OIM scans were carried out in the joint configuration on all specimens. The solder joints used in this study along with their thermal and mechanical histories are given in Table IV.

Aging was accomplished by placing the solder joint in a furnace maintained at a steady temperature of 85° C for 100 hours. Creep test was carried out in a special apparatus devised by Guo² and Choi⁶, which facilitated the documentation of the microstructural changes during creep. The load was applied to the joint through a cable and pulley system. The entire loading assembly along with the solder joint was placed on an optical microscope fitted with an attached closed circuit digital (CCD) camera. The camera was previously setup in such a manner, so that the solder joint edge remained in focus throughout the test. The computer controlled CCD camera took pictures at specified time intervals so that the amount of local strain experienced by the solder joint could be easily studied and documented. Specimens for elevated temperature creep tests were heated by wrapping a heating element around the copper portion of one of the copper dog bones and heat was transferred to the solder joint by conduction, see Figure 12 for details. A constant load of 2.5 lb was applied to the specimen and for high temperature creep; the temperature was controlled at 85°C with ±1°C of accuracy using a variac. Thermocouple wires were wrapped around the dog bones at a distance of about 2 mm from the solder joint (not shown in Figure 12). Figure 12 shows the creep setup used.

	Solder Composition	Joint History	Purpose of Investigation
1	Sn-3.5Ag	As fabricated	Characterize as-cast microstructure and microtexture as a baseline for comparison
2	Sn-3.5Ag	Crept at room temperature (23° C) to γ~0.03	Determine how microstructure and microtexture were changed due to room temperature creep deformation
3	Sn-3.5Ag	Crept at high temperature (85° C) to γ~0.06	Determine how microstructure and microtexture were changed due to high temperature creep deformation
4	Sn-3.5Ag	Aged at 85° C for 100 hours	Analyze same region before and after aging to characterize change in a particular region of microstructure and microtexture due to thermal history, minimal strain history
5	Sn-3.5Ag	TMF 370 cycles between -15° C and 150° C	Determine how TMF changed the microstructure and microtexture

Table IV. Solder joint specimens used to characterize microstructural evolution.



Figure 12. Setup for dead weight loading miniature creep testing frame.

The thermomechanical fatigue (TMF) cycling was carried out by subjecting the solder joints to thermal excursions between -15°C and 150°C with a heating ramp rate of 25°C/min and 20 min dwell time for the heating segment, and a cooling ramp time of 7°C/min and 300 min dwell time for the cooling segment⁷. This was carried out an automated setup developed at Michigan State University⁷. The temperature profile obtained with this setup provided 4 cycles per day using the cycle shown in Figure 13. The TMF cycling was carried out for several cycles on a single solder joint, that were removed periodically to assess the deformation and damage at intermediate cycling stages by OIM analysis, after which they were put back into the cycling mode. The TMF cycles were carried out for 370 cycles in the present study. The deformation generated by TMF was due to the coefficient of thermal expansion (CTE) mismatch between the copper substrates and the solder, and due to the intrinsic anisotropy of tin⁸.



Figure 13. Temperature profile for thermomechanical fatigue cycling $(-15^{\circ}C to 150^{\circ}C)^{7}$.

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2.3 CRYSTALLOGRAPHIC DATA COLLECTION AND TEXTURE MEASUREMENT

The OIM data was collected on a CAMSCAN 44FE SEM using hkl Channel Acquisition software version 4.2 (HKL Technology, Inc., Blaakildevej, 17k, Hobro, DK-9500, Denmark) run on a PC at Michigan State University. The flattened surface (polished with 600µm paper) of the specimen was attached to the flat surface of a standard Aluminum stub using a carbon tape so that the polished edge of the specimen could be seen when observed from the top. The specimen was oriented and placed into the microscope the same way every time. Repeated mounting of the same specimen on separate days resulted in crystal orientations in the same position that differed by less than 2° (however, up to 5° difference occurred in the euler angle for rotations about the normal axis associated with mounting the specimen holder on the microscope stage). Coupled with the intrinsic 1° uncertainty in EBSP pattern indexing the absolute angular orientation in a given specimen is known within $\pm 3^{\circ}$. The specimen was then tilted 70° about the specimen x-axis so that one could view from the top to the bottom of the screen, a solder joint sandwiched between two copper dog bones. A 25kV beam with a specimen current of about 2.3mA was used to generate EBSP's. A probe current greater than 5µA insured good scan conditions. However, with progressive high temperature aging, the detection of EBSPs was degraded. A light polish with 0.05µm silica in an alkaline suspension improved pattern sharpness, probably due to the removal of any oxide layer.

Most OIM scans were overnight scans, collecting around 12,000 pixels of data with 1 μ m step size over a 120x100 μ m² area of the solder joint. Several regions of the same specimen were scanned, to assess the similarities and differences in the microstructure over the entire joint. The most representative maps for each type of specimen are described in the results. The crystal orientation data was collected by rastering the beam over the specimen in an automated manner. Crystal orientation information obtained by these scans was processed to obtain an OIM map. Datasets from the hkl scans were processed and converted to TSL datasets which could be processed by the TexSEM software version 3.0 (TSL Laboratories, Draper, UT), which provided more mapping and analytical tools than version 4.2 of the hkl software. To achieve consistency in the x-y-z coordinate system between the hkl and TexSEM software, the datasets obtained were rotated by $+90^{\circ}$ about the [001] direction (Normal Direction, ND) in the TexSEM software version 3.0 (and this same effect is achieved by rotation of -90° about the [001] direction (Normal Direction, ND) in the TexSEM software alpha version 3.0). Datasets were "cleaned-up" using the software using a nearest neighbor criterion, so as to replace isolated unindexed pixels with the orientation of the nearest neighbor pixel that had the highest degree of confidence in the crystal orientation. Orientation maps, pole figures and misorientation plots were computed using PC version 3.0 of the TexSEM analysis software and version 4.2 of the hkl software.

Since the easiest slip directions in tin are in the c-direction [001] on $\{100\}$ and $\{110\}$ planes, crystals with the c-direction aligned with the solder joint x-axis will be most easily sheared by mechanical or CTE driven stresses in the single shear lap joints. This easy mode of deformation was used to identify a reference crystal orientation scheme. The crystal orientations with the c-axis aligned with the specimen x-axis were chosen as the reference direction and are displayed as red in the orientation maps. The

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P it iŋ bl CO Ĵ eŀ Pe 0ľ h maps are based on the inverse pole figure color key shown in Figure 14. Increased c-axis misorientation from the reference direction [001] by tilting towards the [110] crystal direction is represented by adding blue and removing red, such that a red and a blue grain are misoriented $\sim 90^{\circ}$ by a tilt about a <110> axis. Similarly tilting the [001] reference direction towards the [100] crystal direction is represented by adding green and removing red, such that a red and a green grain are misoriented $\sim 90^{\circ}$ by a tilt about a <100> axis. Further, a [100] (green) crystal direction tilted towards a [110] crystal direction is represented by adding blue and removing green, such that a green and a blue grain are misoriented $\sim 45^{\circ}$ by a tilt about a <110> crystal direction. Any crystal rotation about the specimen x-axis would give the same color. In a different perspective, when the dot product of x- and c-axis unit vectors is equal to unity the representation is red, and when it is equal to zero; the representation could be blue or green or blue-green. Orientations in-between these two are represented by orange, yellow, and various combinations of red, blue and green as seen in Figure 14. Black is used to represent regions where patterns could not be indexed. The colors on these OIM maps do not completely define the crystal orientation (in three dimension), to present a complete three dimensional representation of crystal orientations, 2 such OIM maps are needed, e.g. with different specimen perspectives separated by 90°. Thus the actual crystal orientation is shown in some places using the tetragonal prism (tin has a body centered tetragonal structure), which provides a better understanding to the reader.

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Figure 14. Inverse pole figure color key for an inverse pole figure orientation maps in the [010] direction with sample crystal orientations for grains having a particular color.

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RESULTS AND DISCUSSION

OIM provides a technique that can be used to study the microstructure and its evolution with time after deformation and heat treatment processes. This study is the first of its type with respect to solder, and this technique has tremendous potential to identify the solder joint microstructure evolution. The OIM maps are interpreted with the help of pole figures, misorientation density functions (MODF) and misorientation distribution histograms. The most representative data sets are presented and described here. The results follow the same order as in Table IV on page 65 for a logical presentation and interpretation of the changes that occur with different microstructural evolution paths.

As-fabricated eutectic Sn-Ag specimens were used to characterize the as-cast microstructure and microtexture to establish a baseline for comparison. A specimen was crept at room temperature (23° C) to a global shear strain (γ) of ~0.03. This determined how microstructure and microtexture changed due to room temperature creep deformation. A second specimen with an as-fabricated OIM scan was then crept at high temperature (85° C) to global shear strain γ ~0.06, which determined how microstructure and microtexture creep deformation. A third specimen was used to analyze the same region before and after aging (aged at 85° C for 100 hours) to characterize the change in a particular region of microstructure and microtexture due to thermal history without any externally imposed strain. Another specimen was used to determine how TMF (TMFed for 370 cycles between -15° C and 150° C) changed the microstructure and microtexture. Hence, differences in the behavior of the solder after deformation (creep) or isothermal aging alone were studied, and compared to the role of TMF (thermal and mechanical stresses acting together).

3.1 CREEP

Room Temperature Creep of Eutectic Sn-Ag Solder Joints

Before creep

The SEM and OIM scans of as-fabricated unaged eutectic Sn-Ag solder joints are shown in Figure 15 and 16 respectively. Figure 15(a) is from an area near the center of the joint and Figure 15(b) shows the SEM image of a region 170µm to the right of Figure 15(a). Figure 16(a and b) are the OIM scans corresponding to the boxed regions in Figure 1, which show one dominant orientation in Figure 16(a) and two secondary orientations. The same dominant and secondary orientations are present in Figure 16(b). This dominant orientation has its c-axis tilted from the specimen normal by about 55° towards specimen negative x-axis, whereas the secondary orientation shown by dark orange color has its c-axis tilted from the specimen normal by about 65° towards the positive x-axis. For both these orientations the c-axis is close to the specimen x-axis (which can also be seen from the pole figures in Figure 17(a and b)) and tilted away by about 15°. This orientation is expected to deform easily in shear, since the c-axis vector is a Burgers vector. The secondary orientation shown by purple color has its c-axis tilted from the specimen normal by about 55° towards the negative y-axis. The purple color bands have an orientation with the c-axis pointing nearly out of the page, i.e., the c-axis for this orientation is aligned to resist shear along the specimen x-direction. Thus there appears to be three orientations that are twin related that could be generated by rotating the unit cell about the specimen y-axis by about 60°, however, the region with bluishpurple coloration has the weakest texture intensity (also seen from the pole figure). The regions, which have the red color, have their c-axis, oriented along the x-direction, which aligns the c-axis Burgers vector along the specimen shear direction. The dominant orientation shown by a light orange color in Figure 16(a) and a '+' on the pole figure in Figure 17(a) has a texture intensity peak of 41.9 times that of a random texture^{*}, whereas the dominant orientation in Figure 17(b) has a texture intensity of 110.8 x random.

The secondary (dark orange and purple) orientations are represented by a 'o' symbol in Figure 17(a). The secondary orientation represented by the dark orange color is related to the dominant orientation by a rotation of about 120° (or 60°) about the specimen y-direction.

Low angle and high angle grain boundaries are evident in the map. High angle grain boundaries are easily evident in places where there is a discontinuity in color. There are more high angle grain boundaries representing a range of 55-65° grain misorientations than any other ones. These boundaries are indicated by white lines. These boundaries have been identified as twins¹. Many high angle grain boundaries are linked to low angle grain boundaries (thin for 3-5°, thick black lines for 5-15°) in several locations of the OIM map.

Figure 16(a and b) reveals peculiar texture bands starting from the top right and ending at the bottom left. The secondary orientations, which are the banded texture components visible in the OIM scan, appear to be solidification twins. The dominant orientation throughout this joint had a lighter shade of orange, whereas the minority orientation bands have either a darker orange shade or a purple color. However, within

^{*} A sample with truly random texture would have the same intensity of 1 x random in all orientations on a pole figure. Hence 111 x random is a strong texture that implies a highly preferred crystal orientation. Texture intensities were computed from discrete orientations by representing each orientation with a Gaussian peak and then summing and normalizing using the default setting in the hkl software.

the bands of dark orange there are some regions, which show the lighter shade for the circled region (top middle) in Figure 16(a). The orientations of the crystals in the dark orange bands in Figure 16(b) (lower middle region) are somewhat similar to each other (closely consistent within the band) and are misoriented by 55-65° from the dominant orientation in the joint. The OIM map shown in Figure 16(b), has lesser number of bands as compared to the OIM map in Figure 16(a) (which shows distinct bands).



Figure 15. SEM micrographs of as-fabricated eutectic Sn-Ag solder joint. Figure 15(b) is $170\mu m$ to right of Figure 15(a) used for room temperature creep study.



Figure 16. OIM maps of as-fabricated eutectic Sn-Ag solder. Figure 16(b) is 170 µm to the right of Figure 16(b). White boundaries 55-65°, are twin boundaries; thin and thick black lines correspond to 3-5° and 5-15° misorientations respectively.

The pole figures^{*} in Figure 17 corresponding to the two regions in Figure 16 also show a single dominant orientation along with 2-3 secondary misorientations. The c-axis of the dominant orientation is about 55° from the sample normal. This dominant orientation corresponds to the majority of the matrix, which has a lighter shade of orange. The secondary orientations correspond to c-axis being oriented at 65 and 55° from the sample normal.

Figure 18 shows the misorientation distribution function (MODF) for the asfabricated specimen. The MODF indicates that there is large number (~298 x random) of 62° rotation about the [100] crystal direction. Additional peaks found for other grain boundary angle / axis pairs for this as-fabricated specimen are listed in Table V.

^{*} Pole figures were calculated by TSL software using the "discrete binning" method with 5° bin size.

Angle in degrees	Axis	Intensity (x random)
20°	[110]	1.2
	[100]	8.6
45°	[110] and [111]	4.1 and 7.3
62°	[100]	298
70°	[110]	50.5
77.5°	[829]	7.7
87.5°	[001]	24.4

Table V. Angle / axis pairs as seen in MODF for the eutectic Sn-Ag as-fabricated specimen (Figure 18).



Figure 17. [0 0 1] and [0 1 0] equal area pole figures of as-fabricated Sn-Ag eutectic specimens. 17(a and b) correspond to Figures 16(a and b) respectively.



Figure 18. Misorientation distribution function for as-fabricated specimen with 2.5° bin size.

For each dataset, the number of boundary pixels with a particular misorientation angle between two grains, (using a 1° bin size for misorientations larger than 3°) are plotted as a misorientation histogram. The histogram is represented as a line plot to allow easy comparison between similar misorientation histograms plotted for different specimens (e.g. Figure 19(a and b) that compares histograms before creep and after creep described later). Figure 19(a and b) shows the grain boundary misorientation distribution corresponding to the OIM maps in Figure 16(a and b) respectively for the as-fabricated specimen before creep. There are a large number of pixel pairs (~750) having low angle boundaries <3° and one can see notable peaks at 43, 62 and around 70° misorientations. Maximum number of 62° misorientation is seen throughout the joint, followed by the 70 and 43° peaks respectively. Other peaks corresponding to 30 and 75° are also present but are less intense.

As with misorientation histograms, the area of grains with a particular size^{*} is compared in the specimens before and after creep in Figure 20. For region (a) corresponding to Figure 16(a), one sees that there are a large number of 6-10, 15 and 25 μ m grains. The region (b) corresponding to Figure 16(b) (which has less number of solidification bands) has a large number of 6 and 60 μ m grains. Both regions show a large number of small grains <10 μ m.

[•] The software does not count unbounded grains, whose size is not measurable. Consequently, area is presented in actual area units for bounded grains within the scanned area (which was the same for each scan). Thus changes can be reliably compared.



Figure 19(a). Misorientation profile for as-fabricated and room temperature crept specimens corresponding to Figure 16(a).



Figure 19(b). Misorientation profile for as-fabricated and room temperature crept specimens corresponding to Figure 16(b).



Figure 20. Grain size distribution for as-fabricated and room temperature crept specimens.

After Room Temperature Creep

Figure 21(a and b) are the SEM micrographs of the same regions of the same joint as Figure 15(a and b) after undergoing a global room temperature creep to a shear strain of about 0.03 (The scratch in Figure 21(a) was intentionally put on the specimen after the initial OIM scan on the as-fabricated specimen to assist in the measurement of the amount of creep the specimen had undergone and is not a result of deformation. All OIM scans have been appropriately adjusted and cropped so that the scratch does not affect the interpretation). OIM scans corresponding to the regions shown in SEM micrographs of Figure 21(a and b) are shown in Figure 22(a and b) respectively. On comparison of Figure 16(a and b) and Figure 22(a and b) one sees that room temperature creep does not change the crystal orientation and texture of the solder joint after a global creep shear strain of 0.03. One still sees one dominant orientation and two secondary orientations in both OIM maps. The texture intensity in Figure 23(a) is $35.5 \times random$ which is a slight decrease compared to 41.9 for the as-fabricated specimen. The texture intensity in Figure 23(b) is 115.8 x random which is a slight increase but not a drastic one compared to 110.8 x random in Figure 17(a) for the as-fabricated specimen. These small changes imply that room creep deformation did not cause drastic changes in texture.

However there are subtle changes in the position of both low and high angle grain boundaries. The overall texture remained unchanged and the high and low angle grain boundaries persisted and no major changes in grain shape were observed. There are also minor changes in the texture intensity as indicated in Figures 23(a and b). There was a slight decrease in the texture intensity in the region 'a' (the region with more polycrystalline texture bands) and a slight increase in the texture intensity in region 'b',

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the region with fewer texture bands. The increase in texture intensity of region 'b' can be attributed to the decrease in the purple colored orientation, especially the band of purple in the lower part in Figure 16(b). There have also been slight changes in the grain boundary misorientation distribution as illustrated in Figure 19(a and b). In region 'a', the boundaries with 60° misorientation seem to have decreased while increasing the number of boundaries with a higher 70° misorientation angle. No such features were observed in region 'b'. Also, from comparison of pole figures in Figure 17 and 23, there is a slight rotation of the dominant orientation by about 5-10°. This change is also evident in the prisms in the lower right corner of region b in Figures 16 and 22, by a rotation about an axis close to the specimen normal in the direction of the shear deformation. This observation (lattice rotation and slight changes in maximum texture intensity) shows that the preferred orientation is spread out slightly with deformation².

Figure 24 shows the MODF for the specimen after room temperature creep. The MODF has a maximum of 300 x random for the 62° rotations about the [100] direction.

There has been some grain coarsening of 2-10 μ m grains in the region scanned. The 0.03 global shear strain room temperature creep deformation in region 'a' (region with more bands) increased the number of 2-3 μ m grains and caused the coarsening of 5-7 μ m grains to 10 μ m. Similarly, in region (b) there was a coarsening of 5-7 μ m grains to 10 μ m, increase in the number of 20 μ m grains and a emergence of 45 μ m grains with a corresponding decrease in the 60 μ m grains. However these changes are subtle and this implies that no *significant* grain boundary motion that would cause a change in the misorientation between crystals (such as subgrain coalescence or recrystallization) occurred during room temperature creep deformation to a shear strain of ~0.03. With room temperature and global shear strains of 0.03, there is not enough thermal energy or local deformation, which would cause significant grain boundary motion. The dominant and secondary orientations in the specimen are twin related, and they exhibit some degree of symmetry with respect to the sample coordinate system. Thus room temperature creep deformation occurs by a dislocation creep mechanism, which is consistent with the interpretation from conventional creep experiments³ with a stress exponent greater than 5. The strong texture implies that small solder joints are multicrystals rather than polycrystals, and this accounts for the greater variability of creep resistance in our specimens consisting of single solder joints, as compared to the studies with specimens containing multiple solder joints^{3,4}.

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Figure 21. SEM micrographs of as-fabricated eutectic Sn-Ag solder joint after a global shear creep strain of 0.03. (a) Center and (b) Right of the joint. The scratch in Figure 21(a) was intentionally put on the specimen to assist in the measurement of the amount of creep the specimen had undergone and is not a result of deformation.







Figure 23. [0 0 1] and [0 1 0] equal area pole figures of Sn-Ag eutectic specimens after room temperature global creep to 0.03 shear strain. Figures 23(a and b) correspond to Figures 22(a and b) respectively.



Figure 24. Misorientation distribution function with 2.5° bin size for specimen after global creep to 0.32 shear strain.

High Temperature Creep of Eutectic Sn-Ag Solder Joints

Before Creep

Figure 25(a and b) shows the SEM micrographs before high temperature creep for an as-fabricated eutectic Sn-Ag solder joint. Figure 26 shows the corresponding OIM scans of the two regions shown in these SEM micrographs. Figure 26(a) is from the center of the joint and Figure 26(b) is 323µm to the left. Figure 26(a) shows a number of high angle grain boundaries with a few isolated grains with high angle boundaries. One can also see low angle grain boundaries traversing through the large grains shaded in pink, with mostly the pink color on either side of the low angle (5°) grain boundary. An isolated region shown by a green color has its c-axis pointing about 20° from the specimen normal whereas the majority of the matrix (pink color) has its c-axis about 65° from the specimen x-direction (Figure 26(a)). The purple orientation in the upper region of Figure 26(a) has the c-axis about 80-85° from the positive x-axis and the majority of the blue orientation in Figure 26(b) has its c-axis pointing 90-95° from the positive xaxis. These preferred orientations are readily seen in the pole figures in Figure 27. The region shown in Figure 26(b) encompasses a much larger region as compared to Figure 26(a) and shows predominantly three to four shades of blue separated by low angle grain boundaries. The pole figures shown in Figure 27(a and b) show that in both the scans there is only one dominant orientation (corresponding to the pink and blue colors in the solder matrix of Figures 26(a and b) respectively) with 2 other orientations very close to the dominant one (misoriented by about 10-20°), forming a cluster. These two other orientations are common to both regions and are represented by blue and purple colors in the OIM maps. The pink and blue colors represent orientations, which are very similar,

but they are misoriented by a twin rotation of about 62° . In Figure 26(a), the pink orientation is related to the blue color, by a twin rotation of about 62° about the [100] crystal direction and to the purple (top part of Figure 26(a)) orientation by a rotation of about 40° about an axis near [111] as inferred from the MODF shown later in Figure 28. The OIM map in Figure 26(b) shows blue as the dominant orientation and secondary orientations are different shades of blue. Comparison of the pole figures in Figure 27(a and b) show these relationships, where the dominant pink orientation in Figure 26(a) and 27(a) is about 60° from the dominant blue orientation in Figure 27(b) (that is also present in Figure 26(b)). The texture intensity has a value of 89.8 x random in the scan shown in Figure 27(a) whereas the one in Figure 27(b) has an intensity of 52.9 x random, a lower value due to the clustered blue peaks, which tend to distribute the intensity.

Figure 28 shows the MODF for the as-fabricated specimen. The MODF has a maximum of 198. It shows prominent rotations of about 62° about the [100] direction and a second strong rotation of about 70° about the [110] crystal axis. These peaks are very similar to those observed with the room temperature creep specimen.

Figure 29(a and b) show the misorientation plots corresponding to Figure 26(a and b) respectively. As seen in Figure 29(a and b), there are notable peaks at 43, around 60, around 70, 75 and 80°, with an additional peak at 90° in Figure 29(b).

The grain size plots shown in Figure 30 are similar to Figure 20, showing a bimodal population of very small grains of $\sim 2\mu m$ and grains larger than 10 μm .



Figure 25. SEM micrographs of as-fabricated eutectic Sn-Ag solder joint used for 85° creep study.



Figure 26. OIM maps of as-fabricated eutectic Sn-Ag solder joint used for 85° creep study. Figure 25(b) is $324\mu m$ to the left of Figure 25(a).



Figure 27. $[0\ 0\ 1]$ and $[1\ 0\ 0]$ pole figures of as-fabricated Sn-Ag solder joint specimen used for 85° creep study.



Figure 28. Misorientation distribution before high temperature creep.



Figure 29(a). Misorientation profile for as-fabricated and high temperature crept specimen corresponding to Figure 26(a).



Figure 29(b). Misorientation profile for as-fabricated and high temperature crept specimen corresponding to Figure 26(b).



Figure 30. Grain size distribution for as-fabricated and high temperature crept specimen.

After High Temperature Creep

Figure 31(a and b) shows the SEM micrographs of the same regions as in Figure 25(a and b) after high temperature creep at 85°C with a global shear strain of about 0.06. Figure 32(a and b) shows the OIM maps of the corresponding regions. The scans after creep look similar to the ones before creep with respect to the color representation of texture, however one can easily see the effect of creep by comparing the before and after maps. There is still one dominant orientation with 2 other orientations close to the dominant one, similar to the as-fabricated specimen and shown by the pole figure in Figure 33(a and b). The maximum intensity in the pole figures decreased slightly and all dominant peaks have rotated systematically toward the negative y-direction by about 10°. Also the sharpest peak in 33(b) has the orientation of the secondary peak in Figure 27(b), and vice-versa.

High temperature processes cause grain boundary motion. After high temperature creep (T/T_m = 0.72) to a global shear strain of 0.06, the specimen does show changes with respect to grain boundary locations, particularly in Figure 26(a), but there is not a significant change in the mesotexture. There are changes in the positions of high and low angle grain boundaries which implies that they have moved due to creep that are easily seen by comparing Figures 26(a) and 32(a). The high angle grain boundaries have moved during shear and the isolated minority orientations (shown by circles in Figure 26(a and b)) have been consumed by the larger grains to reduce the number of small grains. The gross texture components remain unchanged, though there is a systematic rotation of all components by about 10° about the x-axis. Also in Figure 32(a), many low angle grain boundaries have become more prominent and small grains have disappeared.

The motion of low angle boundaries implies that recovery has led to dissolution of other low angle grain boundaries to incorporate smaller subgrains, which are separated only by a few degrees, into the larger misorientation. This can be seen in the top right of Figure 32(a) where the low angle grain boundary no longer exists in comparison to Figure 26(a) (arrow shown top right of the OIM map). This shows that the initial dominant orientation grows at the expense of other orientations due to the high temperature. Also, in Figure 32(a) the vertical high angle grain boundary separating the pink and the blue orientations has 'smoothened out' and 'straightened up'. This is also indicative of grain growth indicative of mass movement, which might be temperature assisted or shear strain assisted during creep deformation or a combination of the two.

Figure 34 shows the MODF after high temperature creep. It is similar to the one before creep. However there is a noted addition of 20° rotation about the [110] crystal direction. The maximum intensity after high temperature creep is about 219 x random.

Figures 29(a and b) show the corresponding plots for the misorientation angle. In Figure 29(a) one sees a slight decrease in the 43, 60, 70, 75 and 80°. However, in Figure 29(b) there is a more systematic decrease in all the peaks with minimal retention of the 20, 43 and 70° peaks. In Figure 29(b) one can see an increase in the 10 and 20° peaks. The reduction in peaks implies that grain boundary length decreased, such that the grain boundary energy has been reduced more by high temperature creep than at room temperature (Figures 16 and 22). This means that there is some amount of grain growth accompanying high temperature creep at 85°C.

Cracks develop in high angle boundaries that are not twin boundaries. On examination of the SEM micrograph in Figure 31(a) of the creep specimen after high temperature deformation a discontinuous crack traversing horizontally in the upper region is observed. This crack is located at high angle grain boundaries in Figure 32(a). Figures 35 and 36 show the lattice from the two regions on either side of this high angle grain boundary before and after high temperature creep respectively (the orientations before creep are the corresponding regions from Figure 26(a) on either side of the high angle grain boundary which developed into a crack seen in Figures 31(a) and 32(a)). The (110) plane for both lattices has a misorientation of about 35-40° about the [110] crystal direction. A possible glide system for Sn-crystals is (110) [111]. The [111] crystal directions are aligned along the direction of applied shear strain for the purple and pink orientations in Figure 35. The plane normals are not close to the plane of shear, so these are relatively hard orientations. In Figure 36, the purple orientation has its plane normal nearly perpendicular to the plane of shear, while the pink orientation has slip planes for (110)[111] as well as other c-axis slip systems that could operate to allow a more arbitrary shape change to occur. Thus the pink orientation is soft, and it is adjacent to the purple hard orientation that resists deformation. Such heterogeneity in deformation may account for the formation of the crack.

Crystal rotations of 8-12° developed during high temperature exposure. This must be due to recovery processes and grain boundary motion since small strains cannot cause such large crystal rotations. Figures 35 and 36 illustrate such rotations about the specimen x-axis. Systematic crystal rotation is indicative of slip deformation. Since the solder joint does not deform heterogeneously in all regions during deformation, the observed rotations are rather large for region with local shear strain of 0.06 (seen in the central region of Figure 31(a) and also the lower right corner of Figure 31(b)), which

implies a homogeneous rotation of only 3.4° for e.g. the maximum homogeneous translation to the right of a microstructural feature (region circled in Figure 31(b)) in the lower half of the specimen due to shear is about 9µm (based upon a 150µm thickness), which would result in a local shear strain of 0.06. Portions of the vertical grain boundary between the pink and blue grains moved as much as 20μ m (by comparing Figures 25(a) and 31(a)), with a shear strain value of 0.3 which implies there was localized shear in this region. Since it is unlikely that the specimen could have been mounted at a different orientation by more than a couple degrees (see Appendices), the 10° rotation and the large amount of boundary motion implies that there must have been more microstructural change due to recovery/annealing effects rather than by shear strain alone.

Modest growth of moderate sized grains occurs with creep at higher temperatures. Figure 30 shows the grain size changes after creep at 85° , in comparison to the asfabricated specimens. There is a conversion of grains into larger 20-30µm grains in region 32(a) and a similar trend is seen in region 32(b) with a large number of 33µm and 50µm grains and a decrease in the 20µm grains. The growth indicates motion or elimination of some of the grain boundaries, which removed some of the smaller grains, but did not result in a significant change of the misorientation relationships between crystals during high temperature creep deformation.

These observations show that the single shear lap specimens are multicrystals rather than polycrystals, and hence creep behavior may be made more repeatable by causing a finer grains size with a random texture which would homogenize the plastic deformation properties of the solder interconnect. The substantial motion of grain boundaries as well as crystal rotations support the general view that high temperature creep deformation occurs by a dislocation climb mechanism assisted by glide, which is consistent with the interpretation from conventional creep experiments².



Figure 31. SEM micrographs of the same regions as shown in Figure 25(a and b), after global creep of 0.06 shear strain at 85°C.



global shear strain of 0.06.



Figure 33. Pole figures of eutectic Sn-Ag solder joint after high temperature creep with a global shear strain of 0.06. Figures 33(a and b) correspond to Figures 32(a and b) respectively.



Figure 34. Misorientation distribution after high temperature creep with a global shear strain of 0.06.



Figure 35. Lattice orientations for as-fabricated specimen. The lattice show orientations corresponding to Figure 26(a) before the crack development seen after high temperature creep.



Figure 36. Lattice orientations corresponding to various colors on either side of crack in Figure 32(a) that developed after high temperature creep. The lattice are taken from the Sarne region as in Figure 35.

3.2 AGING

Isothermal Aging of Eutectic Tin-Silver Solder

Eutectic tin-silver, with its low melting point undergoes aging at room temperature since this temperature is above 0.5T_m. Aging at a temperature of 85°C allows us to carry out experimentation possible in reasonable length of time. Bv obtaining OIM maps of the same region of a solder joint before and after aging at 85°C, as illustrated in Figure 37(a and b), microstructural evolution due to aging in a particular location can be examined. Figure 37(a) shows the OIM map of the as-fabricated joint overlaid on the SEM image, and Figure 37(b) shows the OIM map of the same region after aging at 85°C for 100 hours. SEM observations reveal coarsening of the Ag₃Sn particles, evident in the regions adjacent to the OIM scans, and discussed in more detail in Ref. 1. Prior to aging, the OIM scan indicated a single dominant orientation in the joint with fragments of low angle boundaries scattered throughout (essentially a single crystal in the region scanned). Within this orientation, there were many small grains with misorientation angles greater than 15° represented by the white boundaries. Only one continuous low angle boundary was observed (a twist boundary about the c-axis near the bottom in Figure 37(a)).

After aging, (Figure 37(c)) the dominant single crystal orientation was about the same, but the number of small grains with high misorientation angle decreased, and a number of 5-15° low angle boundaries (thicker black lines) developed. The one continuous low angle boundary present near the bottom of the micrograph of the as-fabricated specimen was no longer in the same position after aging.

The pole figures shown in Figure 37(b and d) for as-fabricated and aged specimens are very similar. Subtle changes did occur that reflect substantial motion of low angle grain boundaries. Point plots of the pole figures of both specimens are inserted in-between Figures 37(a and b) with as-fabricated orientations shown in black and the aged orientations in gray (each point represents one or more pixels of the scan). The orientations of the aged specimen in the (001) pole figure show 3 or 4 groups of orientations clustered near that for the unaged specimens. These groups show that the majority orientation is spread out over a larger angular space, such that the intensity of this composite peak is reduced, even though the minority orientations have largely disappeared. These disappearing minority orientations marked with a "+" (near (110)) or a "*" on the pole figures in Figure 37(a) are diminished in Figure 37(b).

The misorientation distributions of the as-fabricated and aged specimen given in Figure 38 shows an increase in the number of low angle misorientations of around 6-10°, and a decrease in the number of high angle misorientations of 43, 60 and 70°. The increase in the number and misorientation of low angle boundaries (thick black lines in Figure 37(a and b)), and the fact that the (001) peak is split into 3-4 distinct sub-peaks rotated by about 15° from the as-fabricated orientation, indicate that low angle boundaries have swept through the microstructure one or more times. The decrease in the number of high angle misorientations is probably due to the energy advantage that large grains have to consume small grains with highly misoriented grain boundaries^{2.5}.

The high angle misorientation peaks in Figure 24 are similar to those in Figure 19(a and b) and 29(a and b). Such misorientation distribution for the as-fabricated specimens used in creep and aging studies, suggest that certain misorientations are

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energetically favored during the solidification process. These misorientations correspond to special boundaries and twins¹: A coincident site lattice is seen when there is a 22.3° rotation about a [110] axis, or with a 43 or 71° rotation about a [010] axis. A 59 or 62° rotation about [010] axis gives a (011)[$\overline{011}$] or (031)[$\overline{013}$] twin, respectively, and both of these twins are evident as overlapping peaks in Figures 19 and 29 (misorientation histogram of as-fabricated creep specimens) and Figure 38.

A few of the highly misoriented small grains did not disappear, particularly if they were part of a cluster with other highly misoriented grains, such as the circled clusters in Figure 37(a and b). These clusters appear to have existed in a smaller size in almost the same location prior to aging in Figure 37(a). A careful investigation of these clusters indicated a higher incidence of misorientations near 60°, than any other misorientation. Twins are known to persist even with long annealing times due to the low energy of their boundary structure⁶ and Figure 38 shows that 60° twins were retained more than the other special boundary misorientations.

A similar OIM scan in a slightly overlapping region to the right of Figure 37(a and b) showed the same type of crystal orientations and changes due to aging, with differences only in the finest details.

A comparison of the MODF before and after aging (Figures 39 and 40 respectively) reveals a decrease in the intensity of the 43° misorientations and an increase in the 0-15° misorientations (blue color) after aging, which is consistent with the result seen from the misorientation distribution histogram in Figure 38. The 5-15° rnisorientations are concentrated preferentially about the [100] crystal direction, though O-5° misorientations have no preferred rotation axis (see Figure 40, 0° triangle). As

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dislocations are absorbed into low angle grain boundaries to increase the misorientation, some boundaries are more stable than others.

Figure 41 presents the grain size distribution for the as-fabricated and aged specimen. The area of the small grains appears to have decreased as peaks in the distribution have shifted from $5\mu m$ to $8\mu m$.^{*} Since large grains are unbounded in the OIM map, the OIM software was not able to characterize grains larger than 10 μm so they are not shown in the grain size distribution.

Low angle grain boundaries of about $8-12^{\circ}$ developed due to high temperature exposure, without stress. In Figure 42, the change in orientation from the bottom location is plotted along a trace as one moves to the top along the vertical red line in Figures 37(a and b). Whereas the *majority* crystal orientation changes monotonically from bottom to top in the as-fabricated joint (spikes are the small highly misoriented grains), the orientation after aging is nearly the same at the top and bottom, but the center of the joint is misoriented from the top and bottom by about 10-15°. Two steps (see arrows) are correlated with the vertical red line crossing a black low angle boundary.

The observed changes in the microstructure such as the elimination of small grains and the polygonization resulting in low angle boundaries are consistent with general physical metallurgy principles. Thus isothermal aging caused strongly preferred orientations to persist, so that the joint maintained a nearly single crystal or multicrystal microstructure. Further aging causes growth and continuance of such multicrystals and

Since all plots are made using the same bin definitions, small differences are statistically meaningful for comparison.

may cause coalescence of smaller crystals into the most dominant of the multicrystals present in the joint.



Figure 37. Effect of isothermal aging on as-fabricated eutectic Sn-Ag joint made from paste solder. (a) As-fabricated condition and with overlay of OIM map on SEM micrograph and (b) corresponding pole figures to (a), (c) aged condition with overlay of OIM map on SEM micrograph and (d) corresponding pole figures to (c). Small highly misoriented grains with a white boundary disappeared (near [110], marked with a "+" on the pole figure in (b), is diminished in (d)). Point plots of the pole figures of both specimens are inserted in-between (b and d) with as-fabricated orientations shown in black and the aged orientations in gray (each point represents one or more pixels of the scan). The aged orientations in the (001) pole figure show 3 or 4 clusters of orientations.



Figure 38. Misorientation angle histograms before and after isothermal aging. The number of misorientations at 43, 60 and 70° decrease after aging, and the number of misorientations in the $6-10^{\circ}$ range increased.



Figure 39. Misorientation distribution function for as-fabricated specimen showing 43° misorientations rotated about a [101] crystal direction and 60° misorientations rotated about a [101] crystal direction.



Figure 40. Misorientation distribution function after aging showing decrease in the intensity of the 43° misorientations and an increase in the $<15^{\circ}$ misorientations.



Figure 41. Grain size distribution for as-fabricated and aged specimen corresponding to the OIM maps in Figure 37. Aging causes an increase in the size of the smaller grains.



Figure 42. Effect of isothermal aging on relative misorientations starting from the bottom to the top of the red line shown in Figure 37(a,b). Aged specimens had a misorientation of about 10° from the as-fabricated orientation using the orientation at the bottom as a reference orientation.

3.3 THERMOMECHANICAL FATIGUE

TMF of Eutectic Sn-Ag Solder Joints

Figure 43 shows the SEM micrographs after 370 TMF cycles for eutectic Sn-Ag solder joint. After obtaining the OIM scans for the as-fabricated Sn-Ag eutectic solder joint in three regions of the same specimen, the OIM scans of the same regions after TMF treatments of 150 and 370 TMF cycles were also obtained. OIM of region 1 is shown in Figure 44(a-c), region 2 is shown in Figure 49(a-c) and region 3 in Figure 53(a-c). Region 1 is close to the center of the joint, whereas region 2 is 362µm to the left of region 1 and region 3 is 229µm to the right of region 1.

Region 1: The OIM scan from region 1 of the as-fabricated joints is shown in Figure 44(a). It reveals a dominant orientation (light pink) with a secondary orientation (lilac). These 2 major orientations present in the joint are spread throughout the joint intermingled with one other. The dominant orientation (light pink) is related to the secondary orientation (lilac) by a rotation of 60° about the [100] crystal direction. The secondary orientation gives a banded appearance to the OIM scan, from the top left toward the bottom right, consistent with the heat flow direction through the copper – solder – copper assembly during solidification. A schematic showing the positioning of the copper strips during the joint fabrication that can result in such a heat flow pattern is shown in Figure 43(d). The banded appearance of the texture is less apparent in this joint due to the fact that these two orientations have similar colors with respect to the sample coordinate systems.
Other than these two orientations, there are a large number of speckled orientations amongst the primary and secondary orientations with a red or a green color. The red and green colored regions are separated from the matrix by high angle grain boundaries (not shown in the figure due to large number of high angle grain boundaries present in the scan). One can also see very few low angle grain boundaries (black lines) in most regions of the joint. The majority (light pink) orientation has the c-axis aligned 45° from the negative x-direction towards positive y-axis, whereas in the secondary orientations (lilac) the c-axes are aligned 45° with respect to the positive x-direction and towards negative y-direction. Note that these two orientations have prisms that appear to have the same orientation, but they do not - this visual illusion is due to the perspective of the unit cell drawing with respect to the normal direction, which can be confirmed with reference to the pole figures in Figure 45. The red and green colored regions, which are scattered amongst the texture bands, have their c-axis aligned along the specimen x-axis and z-axis respectively. The reason for the presence of such small highly misoriented grains in the as-fabricated joint is not clear at present. The primary and secondary orientations are evident on the pole figures in Figure 45.

The OIM scans for region 1 after 150 and 370 TMF cycles are shown in Figure 44(b and c). There is no major change with respect to crystal orientation after 150 TMF cycles, and the bands have persisted and grown after 370 TMF cycles, as seen in Figure 44(c). There are more interconnected low angle grain boundaries after 370 TMF cycles as compared to the as-fabricated specimen or the one that has completed 150 TMF cycles. There are fewer red and green colored orientations after 150 TMF cycles, as is quite evident from looking at the OIM scan in Figure 44(b), and they are further reduced

after 370 TMF cycles. This result may be due to the effect of isothermal aging of 50 hours at 150°C during the first 150 TMF cycles, and another 70 hours at 150°C for the next 220 TMF cycles. The heat input during isothermal aging at such high temperature causes the stress to be reduced by recovery processes, and grain growth. These processes consume small grains seen as speckled green and red regions as seen prominently in the OIM scans of the specimen after 370 TMF cycles. The secondary orientations became more distinct in shape, and larger in size with a significant decrease in the small red and green colors at the same time.

The pole figures for region 1 are provided in Figure 45. The pole figure for the as-fabricated specimen showed a dominant orientation and a secondary orientation. The pole figures showed no change in the overall texture of the joint after 150 and 370 cycles. However after 150 TMF cycles there is a new lighter shade of gray (shown by an arrow) in Figure 45(b) which disappears after 370 TMF cycles as in Figure 45(c). The maximum texture intensity for the as-fabricated specimen was 72.5 x random, which decreased to 49.1 x random for the 150 TMF cycles specimen, but increased to 89.3 after 370 TMF cycles.



(c) Figure 43. SEM micrographs after 370 TMF cycles. (a) Region 1 is near the center of the joint, (b) region 2 is 362µm to the left of (a), (c) region 3 is 229µm to the right of (a) and (d) joint configuration.



Figure 44. Effect of TMF on as-fabricated eutectic Sn-Ag joint made from paste solder from region 1 in Figure 43(a). (a) OIM map of as-fabricated condition (b) OIM map of same region after 150 TMF cycles, and (c) OIM map of same region after 370 TMF cycles.



Figure 45. (a) Pole figures corresponding to OIM map of as-fabricated specimen in Figure 44(a), (b) pole figure after 150 TMF cycles, and (c) after 370 TMF cycles. The dominant orientation has persisted after 370 cycles, which is evident on comparison of the (001) pole figures.

Figure 46 provides the misorientation histogram for region 1 with peaks at 20, 43, 60, 70, 75 and 80° for the as-fabricated specimen. After 150 TMF cycles one sees a reduction in the 60° peak and an increase in the 43 and 70° peak. After 370 TMF cycles, there is a decrease in the number of the 43, 60, 70 and >75° peaks. However, the 60° peak is still persistent and there is no change in the 20° peak. Figure 47(a) shows the corresponding MODF for the as-fabricated specimen. The MODF reveals that the 60° misorientations are rotations about the [100] crystal direction, 70° misorientations are rotations about the [100] crystal direction. After 370 TMF cycles, one sees a larger intensity corresponding to <15° misorientations as seen in Figure 47(c) (see 0-15° triangles in Figure 47(c)). This is consistent with misorientation distribution given in Figure 46 and implies that the grain boundaries corresponding to the misorientation angles less than 15° increase.

Figure 48 shows the grain size distribution for Region 1. At the end of 370 TMF cycles there was an increase in the grain size of the 20 μ m grains and a decrease in the 2 μ m grains. The grain size distribution shows a bimodal distribution similar to the as-fabricated specimens used for creep studies (see footnote on p. 86). The data corresponding to the larger grains are not included in this plot since the software could not consider unbounded grains for this analysis.



Figure 46. Misorientation angle histogram corresponding to region 1 in degrees for asfabricated, 150 and 370 TMF cycled specimen. The number of misorientations at 43, 60, 70 and >75° diminish after 370 TMF cycles and there is an increase in the number of <10° misorientations and no change in the 20° misorientations after 370 TMF cycles.



Figure 47(a). Misorientation distribution function for as-fabricated specimen showing 60° misorientations rotated about a [100] crystal direction. Misorientations >75° are rotated about an axis close to a [313] crystal direction.



Figure 47(b). Misorientation distribution function after 150 TMF cycles showing decrease in the $>75^{\circ}$ misorientations and a slight increase in the $<15^{\circ}$ misorientations as compared to Figure 47(a).



Figure 47(c). Misorientation distribution function after 370 TMF cycles showing an increase in the intensity of $<\!15^\circ$ misorientations.



Figure 48. Grain size distribution corresponding to region 1 for as-fabricated, 150 and 370 TMF cycled specimen and OIM maps shown in Figure 44. TMF caused an increase in the grain size from $10\mu m$ to $20\mu m$ and a decrease in $2\mu m$ grains.

There has been no obvious microcracking on the surface of the SEM micrographs, after 370 TMF cycles. OIM scans show some degree of grain growth for 10-20µm grains, similar to observations for elevated temperature creep. In contrast, the number of smaller grains has remained relatively constant in TMF, unlike in the crept and isothermally aged conditions (Figures 30, 41, 48, 52, and 56). Thus it appears that TMF up to this stage retains small grains, while allowing larger grains to grow in a manner similar to high temperature creep and isothermal aging conditions.

Up to 370 cycles this joint can be considered to undergo stabilization by grain growth probably due to reduction of the number of high interfacial energy grain boundaries present in the as-fabricated specimen (not the 22, 60 and 70° which are considered as low energy boundaries¹). The heat input at the high temperature aids this stabilization and after 370 cycles the joint has spent enough time at the high temperature extreme to substantially reduce the higher energy grain boundary area. This is apparent in the histogram in Figures 46, 51 and 55 (described later), which shows a reduction in the number of non-special boundaries, and an increase in the number of special boundaries. The decrease in grain boundary area implies that grain growth occurred, to reduce the total energy of the grain boundaries⁷

The SEM micrographs in Figure 43(a-c) show coarsening of the Ag₃Sn particles, with no other significant changes. Though the microstructure from the SEM micrographs does not show changes, the mesotexture obtained from the OIM shows changes.

Region 2 showed similar features and changes as seen in Region 1. The OIM maps, pole figures, misorientation histograms and grain size distribution are shown in Figures 49-52 respectively. Observations corresponding to region 3 are provided in Figures 53-57.



Figure 49. Effect of TMF on as-fabricated eutectic Sn-Ag joint made from paste solder from region 2 in Figure 43(b). (a) OIM map of as-fabricated condition (b) OIM map of same region after 150 TMF cycles, and (c) OIM map of same region after 370 TMF cycles.



Figure 50. (a) Pole figures corresponding to OIM map of as-fabricated specimen in Figure 49(a), (b) pole figure after 150 TMF cycles, and (c) after 370 TMF cycles. The dominant orientation has persisted after 370 cycles, which is evident on comparison of the (001) pole figures.



Figure 51. Misorientation angle histogram corresponding to region 2 in degrees for asfabricated, 150 and 370 TMF cycled specimen. The number of misorientations at 43, 70, 75 and 80° diminish after 370 TMF cycles and there is an increase in the number of <10 and 60° misorientations after 370 TMF cycles.



Figure 52. Grain size distribution corresponding to region 2 for as-fabricated, 150, and 370 TMF cycled specimen and OIM maps shown in Figure 49. TMF caused an increase in the grain size from 10 to 20μ m) and decrease in the 2μ m grains.



Figure 53. Effect of TMF on as-fabricated eutectic Sn-Ag joint made from paste solder from region 3 in Figure 43(c). (a) OIM map of as-fabricated condition (b) OIM map of same region after 150 TMF cycles, and (c) OIM map of same region after 370 TMF cycles. The primary orientation in the as-fabricated specimen was consumed by the secondary orientation after 370 TMF cycles.



Figure 54. (a) Pole figures corresponding to OIM map of as-fabricated specimen in Figure 53(a), (b) pole figure after 150 TMF cycles and (c) after 370 TMF cycles. The dominant orientation switched places after 370 cycles, which is evident on comparison of the (001) pole figures.



Figure 55. Misorientation angle histogram corresponding to region 3 in degrees for asfabricated, 150, and 370 TMF cycled specimen. The number of misorientations $<10^{\circ}$ and at 43° diminish after 370 TMF cycles and there is an increase in the number of 60,70 and 75° misorientations after 370 TMF cycles.



Figure 56. Grain size distribution corresponding to region 3 for as-fabricated, 150, and 370 TMF cycled specimen and OIM maps shown in Figure 52. TMF caused an increase in the grain size from 10 to $20\mu m$ grains and a decrease in the $2\mu m$ grains.

Tables VI, VII and VIII compare the microtextural development (Table VI), distribution of misorientation angles between grains, and grain size distribution as a function of TMF cycles in the three regions of the same specimen studied.

Table VI indicates that regions 1 and 2 are similar; both have one dominant and a secondary orientation and large number of speckled orientations in the as-fabricated condition. Region 3 has one predominant orientation and few speckled orientations. Progressively after 150 and 370 TMF cycles, regions 1 and 2 show a decrease in the number of speckled orientations, and the banded texture becomes more distinct. Region 3 shows emergence of a secondary orientation after 150 TMF, which grows and dominates at 370 TMF cycles.

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As can be seen in Table VII, after 370 TMF cycles in regions 1 and 2 there is a tendency for increase in the low angle grain boundary misorientations ($<10^\circ$). Region 3 does not exhibit this trend. All regions show a decrease in the special grain boundary misorientation angles (45, 70 and $>70^\circ$) after 370 TMF cycles. However the 60° peak decreases for region 1 (central region) and increases for regions 2 and 3 (edge regions). It should also be noted that the trend presented for the specimen that has experienced 370 TMF cycles is not consistent with the observations on the same specimen during the course of 150 TMF cycles. He reason for this behavior is not clear at this time.

Table VIII indicates a significant increase in the area of small grains (<10 μ m) after 150 cycles. After 370 TMF cycles there is a decrease in the 2 μ m grains and an increase in the area of larger grains (25 μ m for regions 1 and 3, and 18 μ m grains for region 2). With increasing number of TMF cycles one can envision that some amount of grain growth occurs at the end of 370 TMF cycles. Small grains with larger

misorientations are consumed with reduction is the grain boundary length, and the energetically favored twin-boundaries have persisted at the end of 370 TMF cycles. However, it is not clear why there is an opposite trend in the grain size distribution at the end of 150 TMF cycles in contrast to 370 TMF cycles.

Deformation in TMF is a complicated process and is different from isothermal aging and creep. In addition the highly inhomogeneous behavior of the heterogeneous solder joint complicates the overall behavior.

Table VI.Comparison of Microtextural Features for the Three Regions afterSeveral TMF Cycles.

Region	Feature	As-fabricated	150 TMF cycles	370 TMF cycles
_	Dominant Orientation	Present (Whitish- Pink)	Present (Pink)	Present (Pink) and more distinct
1 (Middle Region)	1 (iddle Secondary Orientation Present (Lilac) (gion)		Present (Lilac)	Present (Lilac) and more distinct
Large numberSpeckledscattered throughoutOrientation(red and green colored)		Reduced in number	Almost absent	
2	Dominant Orientation Present (Pink)		Present (Pink)	Present (Pink) and more distinct
(Edge	Secondary Orientation	Present (Whitish- Green)	Present (Lilac)	Present (Lilac) and more distinct
Region)	Speckled Orientation	Large number scattered throughout (red and green colored)	Reduced in number but not significantly	Almost absent
3 (Edge	Dominant Orientation	Present (Yellow)	Decrease (Yellow-Orange)	Reduced further and becomes secondary orientation (Yellow- Orange)
Region)	Secondary Very small amount Orientation (Purple)		Increases significantly (Purple)	Dominates (Purple)
	Speckled Orientation	Seen scattered throughout (red and green colored)	Reduced in number	Almost absent

Region	Misorientation Angle	tion Number of Grain Boundary Pixels				
	(deg)	As-fabricated	150 TMF cycles	370 TMF cycles		
	<10	200	200	300		
	20	200	200	200		
1	30	60	80	40		
(Middle	45	270	280	200		
Region)	60-62	400	300	350		
	70	325	520	280		
	>70	~200	~200	~50		
	<10	120	80	280		
	20	300	100	250		
2	30	60	100	40		
(Edge	45	170	200	100		
Region)	60-62	350	210	500		
	70	400	400	150		
	>70	~200	~200	~100		
	<10	250	280	120		
2	20	70	50	50		
5	30	50	50	30		
(Edge	45	210	250	120		
Region)	60-62	240	280	490		
	70	320	250	380		
	>70	~220	~180	~180		

Table VII.Comparison of Misorientation Angles for the Three Regions afterSeveral TMF Cycles.

Region	Grain Size		Area (square μm)	
	(μm)	As-fabricated	150 TMF cycles	370 TMF cycles
	2	600	1000	400
1	3-8	200	250	80
(Middle	10	250	400	100
Region)	18-22	400	550	200
	25	-	-	400
	2	650	1600	450
2	3-8	200	225	225
(Edge	10	100	-	200
Region)	18	-	200	400
	25	400	-	-
3	2	550	850	550
Ædge	3-8	100	225	200
Region)	10-12	80	400	125
	20	-	-	300

Table VIII. Comparison of Grain Size for the Three regions after Several TMFCycles.

3.4 REFERENCES

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SUMMARY

The results obtained by OIM provide significant insight into the solder microtexture, which could not be observed by ordinary microscopic techniques. Almost all of the as-fabricated specimens showed texture bands, however the orientation of the dominant crystals in the joint varied from joint to joint. There was typically a dominant and secondary orientation in as-solidified specimens that were twin related, which often exhibited some degree of symmetry with respect to the sample coordinate system. Most specimens tend to have the c-axis not close to the length direction of solder/substrate interface. The strong texture implied that small solder joints are multicrystals instead of polycrystals. Certain misorientations are energetically favored during the solidification process such as the 60° twins and 70° misorientations (Σ boundaries).

The gross texture components remain unchanged after creep, aging, or TMF up to 370 cycles. High temperature processes like creep and aging cause grain boundary motion. Low angle grain boundaries of about 8-12° developed due to high temperature exposure, with or without stress. Also, crystal rotations of 8-12° developed during high temperature exposure, with or without stress. Creep studies have shown that no major crystallographic changes have occurred after room temperature creep of 0.03 global shear strain, whereas high temperature creep with 0.06 global shear strain does show some changes in the crystal orientation but with no major change in the texture of the solder joint. Also, during high temperature creep, cracks nucleated along the high angle grain boundaries (that are not twin type grain

boundaries) aligned in the direction of shear. Twin boundaries persist more than other boundary types after high temperature exposure, with or without stress. Aging studies revealed grain coarsening with an increase in the number of low angle grain boundaries and decrease in the number of high angle grain boundaries. Up to 370 TMF cycles the joint reveals grain growth.

Suggestions for future work:

- Is localized deformation caused by generation of small grains that decrease the creep resistance locally? Do the boundaries provide more interfacial locations for recovery processes?
- 2. Do twin boundaries cause near neighbor boundaries to be more likely to crack? Are twin boundaries resistant to absorbing dislocations so that neighboring boundaries have to absorb more, and hence are more highly strained? Are \sum boundaries less likely to slide, and hence more likely to crack?
- 3. Do non-twin misorientation peaks change in the opposite sense (or differently) as compared to the other ∑ boundaries?
- 4. With increasing number of thermal cycles, is there more nucleation of small grains in grain boundaries or development of cracks? Which types of boundaries cause cracking vs. nucleation of small grains?
- 5. Does damage nucleation depend on the dominant crystal orientation from solidification and their misorientations with neighboring grains and how is damage nucleation related to ultimate failure and breakdown of the interconnect?

APPENDIX

Specimen Mounting Error Calculation: The tables below show euler angle data recorded after mounting the same specimen over and over again. It is found that a maximum of 3.5° is seen about euler angle 1. All euler angles are in degrees.

Specimen: High Temperature Creep Region A

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 1	161	80	58	163	80	58
	162	81	57	163	81	56
	162	81	57	164	80	57
	162	80	57	163	80	58
Average	161.75	80.5	57.25	163.25	80.25	57.25

Difference					
Euler 1	Euler 2	Euler 3			
-1.5	0.25	0			

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 2	150	80	25	151	80	24
	151	80	24	152	80	24
	150	80	25	151	81	25
	148	81	25	150	81	26
Average	149.75	80.25	24.75	151	80.5	24.75

Difference					
Euler 1	Euler 2	Euler 3			
-1.25	-0.25	0			

Specimen: High Temperature Creep Region B

SET 1: First Iteration:

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 1	102	110	46	102	110	46
	101	110	47	103	110	46
	102	110	46	103	110	46
	101	110	46	102	110	46
Average	101.5	110	46.25	102.5	110	46

Difference				
Euler 1 Euler 2 Euler 3				
1	0.5	0.5		

Second Iteration:

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 1	102	110	46	105	112	46
	101	110	47	105	111	46
	102	110	46	105	112	46
	101	110	46	105	111	46
Average	101.5	110	46.25	105	111.5	46

Difference					
Euler 1	Euler 1 Euler 2 Euler 3				
-3.5	-1.5	0.25			

SET 2: First Iteration:

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 2	264	93	55	267	93	55
	265	93	55	266	93	54
	265	94	54	266	93	54
Average	265	93	55	267	93	55

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Difference				
Euler 1	Euler 2	Euler 3		
-1.75	0.25	0.25		

Second Iteration:

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 2	264	93	55	268	91	55
	265	93	55	268	91	54
	265	94	54	268	91	54
Average	265	93	55	268	91	55

Difference				
Euler 1	Euler 2	Euler 3		
-3.25	2.25	0.25		

Specimen: High Temperature Creep Region C

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 1	249	101	51	248	100	50
	249	100	51	249	101	50
	250	101	51	248	99	51
	252	79	54	251	79	54
Average	250	95.25	51.75	249	94.75	51.25

Difference				
Euler 1	Euler 2	Euler 3		
1	0.5	0.5		

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 2	236	115	45	234	114	46
	236	116	52	235	115	51
	236	116	51	234	116	50
	236	116	51	234	115	50
	237	116	52	235	115	52
	234	116	46	234	115	47
Average	235.83	115.83	49.5	234.33	115	49.33

Difference				
Euler 1	Euler 2	Euler 3		
1.5	0.83	0.17		

Specimen: Thermomechanical Fatigue

	Before			After		
	Euler 1	Euler 2	Euler 3	Euler 1	Euler 2	Euler 3
Set 1	135	115	84	135	115	84
	134	115	86	136	115	85
Average	134.5	115	85	135.5	115	84.5

Difference				
Euler 1	Euler 2	Euler 3		
-1	0	0.5		

