Correlation between thermal fatigue and thermal anisotropy in pure Sn and a Pb-free solder alloy

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September 2005
MT05.15

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Summary

During service solder joints are subjected to thermal effects due to power cycling and environmental temperatures changes. The anisotropy in thermal expansion and elastic properties of Sn may induce significant stresses at Sn-grain boundaries. For this research project, the role of intrinsic anisotropy of Sn on the thermal fatigue damage is studied both experimentally and numerically.

Mechanically unconstrained bulk Sn-rich (Sn-3.8Ag-0.7Cu) and pure Sn samples were thermally cycled within the temperature range 20 to 80 °C or -20 to 80 °C for a 1,000 cycles. The damage evolution was studied by optical micrography. Micrographs of the as-solidified bulk samples were compared with micrographs taken after thermal cycling. Fatigue damage was shown to occur in bulk SAC and pure Sn specimens under thermal cycling. The damage was localized mainly along high angle grain boundaries. The amount of fatigue damage increased with increasing temperature range.

Crystal orientations were measured employing orientation imaging microscopy (OIM). The OIM data was also directly used to construct FE models from which the stress distribution resulting from the thermal anisotropy of Sn was calculated. Finite element calculations showed stress concentrations along those boundaries. The location of the fatigue cracks was found to strongly correlate with the regions where the largest stresses encountered due to the thermal anisotropy of Sn.

OIM analysis confirmed that the grain shape and orientation remained unchanged after thermal cycling for SAC specimens. The pure Sn specimen thermally cycled between -20 and 80 °C showed sign of re-crystallization.

Intrinsic thermal anisotropy of Sn is the determining factor in stress build-up which initiates damage during in thermal cycling of mechanically unconstrained SAC and pure Sn specimens.
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Chapter 1

Introduction

Solder interconnections are used in the surface mount technology (SMT) to connect components to the printed circuit board. The interconnections serve both a mechanical and an electrical purpose.

The Sn-Pb solder alloy has been used in interconnection technology for a long time. The growing concern about the effect the toxicity of lead on the environment and health asks for the replacement of the Pb-solders.

During service the solder joints are subjected to thermal effects due to power cycling and environmental temperature changes. The thermal cycling induces mechanical loads on the solder alloy. The mechanical loads on the solder alloy originate on a macroscopic scale from the thermal expansion (CTEs) mismatch between e.g., a chip and a printed circuit board and on a microscopic scale due to differences in CTEs between the various phases in the solder itself [7].

Recently, industry has focused its interest on eutectic Sn-3.8Ag-0.7Cu (SAC) because of its comparatively low melting temperature, the competitive price, and good mechanical properties [2].

The Sn crystals have a body-centered tetragonal (bct) structure with lattice parameters $a = b = 0.632 \text{ nm}$ and $c = 0.3182 \text{ nm}$ at 25 °C, in which $c/a$ ratio equals 0.546 [6]. In relation to the crystal anisotropy of the Sn, the mechanical properties of Sn e.g., the thermal expansion and elastic behavior, are also anisotropic.

This anisotropy in thermal expansion and elastic properties of Sn may induce significant stresses at Sn-grain boundaries during thermal cycling. Damage may initiate at high stressed grain boundaries.

For this research project, the role of intrinsic anisotropy of Sn on the thermal fatigue damage is studied both experimentally and numerically. Damage evolution due to thermal cycling and crystallographic orientation was studied on SAC and pure Sn specimens by polarization light microscopy (PLM) and orientation imaging microscopy (OIM). The OIM results were used as input for the FEM simulations to calculate the stress concentrations resulting from the thermal anisotropy of Sn.
Chapter 2

Experimental techniques

Various experimental techniques used are described in this chapter. The purpose of these techniques was to collect crystallographic data which were used for the numerical part of the research. The experiments also serve to capture micrographs to study the damage evolution due to thermal cycling and to compare with numerical results.

2.1 The specimens

The Sn-3.8Ag-0.7Cu (SAC) solder alloy is the main focus of this research. This eutectic alloy has two phases, the Sn-rich dendrites and the eutectic phase, as shown in Fig. 2.1. The microstructure is characterized by a Sn-matrix, called $\beta$-tin. This is the normal form of the Sn element above 13 $^\circ$C. Below this temperature the crystal structure of Sn changes to the diamond structure called $\alpha$-tin. The structural change is accompanied by an increase of density by 26%. This allotrophic transition is quite slow and the presence of trace amounts of impurity metals (such as aluminium, zinc, antimony or bismuth) can retard or even inhibit the change [9].

Figure 2.1: BSE micrograph of the microstructure of the eutectic SAC
The eutectic region has a small volume fraction of submicron-size $Cu_6Sn_5$ and $Ag_3Sn$ intermetallic particles that provide strengthening of the Sn-matrix. The Sn-dendrites are long shaped features with dendritical arms. The morphology of the Sn-dendrites depends on the employed cooling rate. The size could reach up to 50 µm in diameter (as shown in Fig. 2.1 by an arrow).

We focus on the effect of anisotropy of the Sn crystal at the scale of grain level to investigate whether this anisotropy is a determining factor in stress build-up and damage initiation in thermal cycling. Therefore, a mechanically unconstrained specimen configuration was used (see Fig. 2.2). The effect of the global CTE mismatch between solder and substrate is no longer present.

The complex microstructure of SAC may influence the grain-scale phenomena and cloud the effect of anisotropy. Therefore pure Sn specimens were also prepared. Pure tin is a polycrystalline material containing only the $\beta_{Sn}$ phase. The effect of thermal expansion mismatch among the various phases thereby has also been eliminated.

The effect of thermal anisotropy was investigated in two SAC specimens ($SAC_1$, $SAC_2$) and two pure Sn specimens ($Sn_1$, $Sn_2$).

**Preparation of the specimen**

![Figure 2.2: The mechanically unconstrained specimen configuration](image)

The eutectic SAC solder specimens were prepared from commercial solder alloy Sn-96L-NS (Balverzinn, Germany). The bulk pure Sn specimens were prepared from 99.999 % purity Sn. The materials were separately sealed in cylindrical quartz ampoules of 1 cm in diameter and 5 cm in length under a vacuum of $10^{-4}$ Pa. The ampoules were superheated to 100 °C above the eutectic temperature in a furnace at a heating rate of 10 °C/min.

To ensure the homogeneity of the alloy the ampoules were carefully shaken before quenching them down with liquid nitrogen (LN2) to -196 °C. The purpose of using LN2 was to obtain specimens with very fine microstructure representative of a solder interconnection.

The samples were sectioned into 1 cm pieces, which were then ground onto silicon carbide
Table 2.1: Sample identification and thermal fatigue test variables

<table>
<thead>
<tr>
<th>Sample</th>
<th>$T_{\text{min}}$ [°C]</th>
<th>$T_{\text{max}}$ [°C]</th>
<th>$t_{\text{min}}$ [s]</th>
<th>$t_{\text{max}}$ [s]</th>
<th>cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAC$_1$</td>
<td>20</td>
<td>80</td>
<td>5</td>
<td>15</td>
<td>1000</td>
</tr>
<tr>
<td>SAC$_2$</td>
<td>-20</td>
<td>80</td>
<td>5</td>
<td>15</td>
<td>1000</td>
</tr>
<tr>
<td>Sn$_1$</td>
<td>20</td>
<td>80</td>
<td>5</td>
<td>15</td>
<td>1000</td>
</tr>
<tr>
<td>Sn$_2$</td>
<td>-20</td>
<td>80</td>
<td>5</td>
<td>15</td>
<td>1000</td>
</tr>
</tbody>
</table>

polishing paper with grit sizes 1000 to 2400, followed by fine polishing with diamond suspensions of 6, 3, and 1 µm. Final mechanical polishing was performed with a solution of 0.05 µm colloidal silica.

2.2 Cyclic thermal loading

![Temperature Profiles](image.png)

Figure 2.3: Temperature profiles “1” and “2” used for thermal cycling

The thermo-mechanical fatigue (TMF) cycling was carried out by subjecting the specimens to thermal excursions between +20 to 80 °C (profile 1) or -20 to 80 °C (profile 2), see Fig. 2.3. The temperature profiles had a ramp rate of ±30 °C/min. Profile 1 does not contain the transition temperature between β-tin and α-tin, whereas profile 2 does contain it. Details on the specimens investigated area shown in Table 2.1. Liquid nitrogen (LN2) was purged into the heating-cooling stage (LINKAM LTS-350) to attain sub-zero temperatures.
2.3 Data collection

Polarizing light microscopy

Polarizing Light Micrographs (PLM) were taken before and after thermal cycling from the entire plane section of the specimens. The micrographs before thermal cycling served as a reference of the microstructure. Comparing this reference with the micrograph taken after thermal cycling the microstructural evolution was evaluated during thermal cycling.

Orientation imaging microscopy

Orientation Imaging Microscopy (TSL OIM detector) was performed to obtain local orientation information by indexing Electron Backscatter Diffraction Patterns (EBSP). A 30 kV beam with a current intensity of about 8 nA was used.

The OIM scan data contains spatial coordinates \((x,y)\), the Euler angles \((\varphi_1, \phi, \varphi_2)\), confidence index \((CI)\), image quality \((IQ)\), etc. The data can be processed by the OIM analysis software. This software includes several cleaning routines, to process and filter the OIM scan data. The nearest-neighbor criterion was used to replace scan points with a low confidence index with those of the nearest neighbor that has the highest degree of confidence in the indexing. Also grain dilation was used to reduce the number of small grains [1].

A step size of 15 \(\mu m\) or 20 \(\mu m\) was used. These step sizes are too coarse to capture the dendrite substructure of SAC, where the typical scale for the tin dendrites is about 25 \(\mu m\) and the \(Ag_3Sn\) particles are even smaller. But these step sizes are sufficient to capture the structure at the scale of interest, the grains.

The OIM technique allowed to simultaneously measure the grain orientation and shape. This data were used for a numerical analysis of the stress that develop due to the anisotropy of Sn.

Backscatter electron Microscopy

Backscattered Electron (BSE) images were taken from selected areas on the plane section of the specimens before and after thermal cycling to evaluate the microscopic deformation mechanisms.
Chapter 3

Computational method

This chapter describes the FE analysis technique. Assumptions made for the FE analyses are described in Section 3.1 of this chapter. In Section 3.2 a description of the FE models were given.

The FE models are based on the crystallographic orientation data obtained by the OIM scans. Two different methods of making the models have been investigated. The first method made use of the grain shape and the average orientation within the grains. This method is described and discussed in Appendix A. The second method used the local orientation data directly and is presented in this chapter.

3.1 Assumptions

3.1.1 Material model and properties

To calculate the stress distribution on a given plane section of a specimen a linear elastic material model is used. Plastic deformation was not accounted for in this type of material model.

For an anisotropic linear elastic material model, Hooke’s law relates the stress tensor \( \sigma \) and the strain tensor \( \varepsilon \) by means of the fourth order stiffness tensor, \( \mathbf{C} \):

\[
\sigma = \mathbf{C} : \varepsilon
\]  

(3.1)

A fourth order stiffness tensor has 81 components. All of these components are not independent. The number of independent elastic constants decreases with increasing structural symmetry. Based on the crystallographic symmetry, a BCT crystal only has six independent elastic constants.

In Table 3.1 three sets of elastic constants are [6, 8, 11]. They all show anisotropy in the elastic behavior of Sn. The set from [6] was used for the FE calculations.

The index notation that is used here is the so-called Voigt notation. This is a notation often used in composite mechanics. It is a way to reduce the fourth order stiffness tensor to a second order \( 6 \times 6 \) matrix. The stress components are numbered 1 to 6 and placed in a column:

\[
\tilde{\sigma} = \begin{bmatrix}
\sigma_{11} & \sigma_{22} & \sigma_{33} & \sigma_{12} & \sigma_{23} & \sigma_{13}
\end{bmatrix}^T
\]  

(3.2)

\[
\tilde{\sigma} = \begin{bmatrix}
\sigma_1 & \sigma_2 & \sigma_3 & \sigma_4 & \sigma_5 & \sigma_6
\end{bmatrix}^T
\]
Table 3.1: Elastic constants of $\beta$$_{Sn}$ in GPa

<table>
<thead>
<tr>
<th>$C_{ij}$</th>
<th>[6]</th>
<th>[8]</th>
<th>[11]</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_{11}$ = $C_{22}$</td>
<td>73.5</td>
<td>83.91</td>
<td>86.0</td>
</tr>
<tr>
<td>$C_{33}$</td>
<td>87.0</td>
<td>96.65</td>
<td>133.0</td>
</tr>
<tr>
<td>$C_{44}$</td>
<td>22.65</td>
<td>7.41</td>
<td>53.0</td>
</tr>
<tr>
<td>$C_{55}$ = $C_{66}$</td>
<td>22.0</td>
<td>17.54</td>
<td>49.0</td>
</tr>
<tr>
<td>$C_{12}$</td>
<td>23.4</td>
<td>48.70</td>
<td>35.0</td>
</tr>
<tr>
<td>$C_{13}$ = $C_{23}$</td>
<td>28.0</td>
<td>28.1</td>
<td>30.0</td>
</tr>
</tbody>
</table>

$\sigma_{11}$, $\sigma_{22}$, and $\sigma_{33}$ are the stresses in the principle directions of the crystal working in a plane perpendicular to that direction, respectively [100], [010], and [001]. The corresponding components of the stiffness tensor are numbered accordingly [10].

The linear thermal expansion due to a temperature difference $\Delta T$ is described with the second order thermal expansion tensor $\alpha$:

$$\varepsilon = \alpha \cdot \Delta T$$  \hspace{1cm} (3.3)

The coefficient of thermal expansion in the principle directions used for the FE analysis are $\alpha_{[100]} = 16.5 \times 10^{-6}$ °C$^{-1}$ and $\alpha_{[001]} = 30.5 \times 10^{-6}$ °C$^{-1}$ [12].

3.1.2 Reference state and loading condition

A simple linear material model was used for the FE analysis. To calculate the stress (and strain) distribution due to a temperature difference ($\Delta T$), 20 °C was chosen to be a stress free reference. A temperature increase to 80 °C was simulated ($\Delta T$=60 °C) for both the temperature profiles.

To prescribe the temperature difference in the FE model state variables were used for both the initial condition and the loading of the model. State variables can prescribe the temperature of elements. The same state variable was assigned to the complete mesh; simulating a homogeneous temperature.

3.1.3 The “third dimension” and Boundary conditions

The inclination of the grain boundary in relation to the specimen surface was unknown. Since this information can only be obtained by a sectioning technique, which will destroy the specimen, it is assumed that the grain boundaries are perpendicular to the surface [4].

To reduce the number of elements in the mesh, a symmetry boundary condition for the $z$-direction was used. This was done by suppressing the $z$-translation of all nodes of the lower surface of the mesh. By this means, the effective thickness of the mesh is twice the real mesh thickness.

With the symmetry boundary condition for the $z$-direction the rigid body translation along that direction was suppressed. The symmetry boundary condition also inhibits rigid body
rotations about the $x$ and $y$.
The rotation around the $z$ axis and translations in both $x$ and $y$ direction are inhibited by fixing one node in the $y$ direction and fixing another node in both the $x$ and $y$ directions.

3.2 The FE model

3.2.1 The FE mesh

The OIM scan points were captured in a rectangular grid ($15 \times 12.99 \, \mu m$ and $20 \times 17.32 \, \mu m$). For each specimen more than $10^5$ scan points were needed to cover a broad strip of the specimen surface.

This rectangular grid OIM scan grid could be used to produce a 2D FE element division. But, representing every scan point by one element in an element layer, would be time consuming during simulation. Therefore every nine OIM scan-points were considered to comprise of one element per element layer. This was done by selection the scanning point with the highest Confidence Index (CI) out of every block of $3 \times 3$ OIM scan points. (CI is a characteristic value ranging from 0 to 1; indicating the degree of confidence that the orientation calculated is correct).

This 2-dimensional element grid, $(x,y)$-plane, was transformed to a 3-dimensional mesh by expanding and duplicating it. Two layers of elements, each having a thickness of $2 \, \text{mm}$, were generated across the thickness. The element used were 3D solid hexagonal elements having 20 nodes.

To satisfy the assumption that the ‘orientation’ boundaries are perpendicular to the $(x,y)$-plane, the same orientation (see Paragraph 3.2.2) was assigned to the elements with the same spatial coordinates, $x$ and $y$.

3.2.2 Orientation of material properties

![Figure 3.1: The system of reference of (a) the sample $\epsilon^{sample}$ and (b) FEM $\epsilon^{FEM}$](image)

The orientation of the anisotropic material properties is the most important ingredient of the FE analysis. The crystallographic orientation needs to be specified within the FEM system of reference.
Crystallographic orientation data for Sn-crystals are collected with respect to the sample frame of reference, $\xi^\text{sample}_i$. This reference frame is defined as the rolling direction (RD) axis pointing downward, the transversal direction (TD) axis to the right and normal direction (ND) axis pointing out of the plane, see Fig. 3.1(a).

Crystal orientation is defined by three Euler angles which bring the axes of the sample into coincidence with the crystal axes, $\xi^{\otimes}_i$, following Bunge’s passive right-hand (positive) convention. Following this convention, three consecutive rotations are performed sequentially as follows. The first rotation $\varphi_1$ is about sample ND axis, depicted in Fig. 3.2(c). The next rotation is $\phi$ around the [100] axis, shown in Fig. 3.2(d), followed by the third rotation $\varphi_2$ around the [001] axis, Fig. 3.2(e). These rotations are respectively expressed by the rotation matrices $R_{\varphi_1}$, $R_\phi$, and $R_{\varphi_2}$,

$$
\xi^\text{sample}_i = R_{\varphi_1} \cdot \xi^{\otimes}_i'' = R_{\phi} \cdot \xi^{\otimes}_i' = R_{\varphi_2} \cdot \xi^{\otimes}_i \quad \text{(3.4)}
$$
with,

\[ \mathbf{R}_{\varphi_1} = \begin{pmatrix} \cos \varphi_1 & -\sin \varphi_1 & 0 \\ \sin \varphi_1 & \cos \varphi_1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad \mathbf{R}_\phi = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos \phi & -\sin \phi \\ 0 & \sin \phi & \cos \phi \end{pmatrix} \]

\[ \mathbf{R}_{\varphi_2} = \begin{pmatrix} \cos \varphi_2 & -\sin \varphi_2 & 0 \\ \sin \varphi_2 & \cos \varphi_2 & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad \mathbf{R}_{FEM} = \begin{pmatrix} 0 & 1 & 0 \\ -1 & 0 & 0 \\ 0 & 0 & 1 \end{pmatrix} \]

The FEM frame of reference, \( \mathbf{e}^{FEM} \), is defined as \( x \) pointing to the right, \( y \) upwards, and \( z \) pointing out of the plane, see Fig 3.1(b). To convert from the FEM to the laboratory frame of reference a rotation of 90° clockwise around the \( z \) axis is necessary, see Fig. 3.2(b). This is expressed by the rotation matrix \( \mathbf{R}_{FEM} \):

\[ \mathbf{e}^{FEM}_i = \mathbf{R}_{FEM} \cdot \mathbf{e}_i^{sample} \quad (3.5) \]

Combining equations 3.4 and 3.5 gives the rotation matrix between the FEM and the crystal frame of reference:

\[ \mathbf{e}^{FEM}_i = \mathbf{R}_{FEM} \cdot \mathbf{R}_{\varphi_1} \cdot \mathbf{R}_\phi \cdot \mathbf{R}_{\varphi_2} \cdot \mathbf{e}_i \quad (3.6) \]

Appendix B contains an example of this orientation definition.
Chapter 4

Results and discussion

In this chapter the results from the experiments as well as the simulations are presented and discussed. The polarization micrographs are mostly combined micrographs to represent the entire specimen surface. Also the inverse pole figures (IPF) map contain combined data from several OIM scans.

4.1 The SAC specimens

4.1.1 Sample SAC$_1$:

![Image of micrographs](a) before and (b) after thermal cycling

Figure 4.1: Sample SAC$_1$: optical micrographs (a) before and (b) after thermal cycling

Fig. 4.1 shows polarization micrograph sample SAC$_1$ of the as-solidified condition, and the same sample after the treatment described in Section 2.2. A number of grains with different crystallographic orientations were observed as depicted in Fig. 4.1(a).
After thermal cycling, microstructural changes were noticed which were localized along a grain boundary (indicated by arrow “A” in Fig. 4.1(b)) or near grain boundaries (indicated by arrow “B” in Fig. 4.1(b)). Polarization micrographs obtained from the areas marked by arrows “A” and “B” are shown in Fig. 4.2 where grains have been numbered and boundaries are clearly visible as contrast differences. Within the area “A” cracks followed the grain boundary (between grains “38” and “43”). A region from the boundary between “50” and “43” did not show such microcracks. The region “B” near the triple junction 50-46-41 shows cracks along boundary and slip bands in some dendrites. The boundary between grain “46” and “41” showed similar features as observed the region “A”.

The crystallography of the sample was studied in more detail with orientation imaging microscopy (OIM). Fig. 4.3(a) shows a [001] inverse pole figure (IPF) map for a part of the specimen (marked by white marked in Fig.4.1(a)) before thermal cycling. OIM scans were taken of approximately the same part after thermal cycling, as shown in Fig 4.3(b). The grain structure remained the same after thermal cycling. The IPF maps correspond well with the optical micrographs.

A clean-up routine was applied to OIM data, incorporating lower confidence index points with higher confidence index points (using a nearest neighbor correlation technique) and removing sub-micron size $Ag_3Sn$ and $Cu_6Sn_5$ particles.

From the OIM data the angles between the [001] directions belonging to all pairs of adjacent measurements, were calculated. Fig. 4.4(b) shows a map of the calculated misorientation angles, that range from 0° to 900°. Locations of the grain boundaries can clearly be distinguished.

It is concluded that the microcracks depicted in Fig. 4.1(b) are preferentially located near the boundaries shown in Fig. 4.4(b).

This observation is the basis of more detailed investigations by Finite Element (FE) analysis.

FE analysis were performed to test the hypothesis that the occurrence of damage near grain
Figure 4.3: Sample SAC1: IPF intensity maps (a) before thermal cycling and (b) after thermal cycling; (c) corresponding IPF for $\beta_{Sn}$.

Figure 4.4: Sample SAC1: (a) Von Mises stresses from FE simulations at $\Delta T = 60 \, ^{\circ}C$ (b) Misorientation angles between adjacent pairs of data points with respect to [001] axis.
boundaries during thermal fatigue loading is related to stresses induced by thermal cycling and the thermal anisotropy of Sn. The FEM model was set up as described in chapter 3. The OIM data used for this FE model was captured after thermal cycling and covered the entire specimen surface. The element size of the 2D FE mesh is 60×51.96 µm and each of the layers contained 20,481 element.

The Von Mises stress distribution obtained from simulations is presented in Fig. 4.4(a). The stresses are localized in a small volume around grain boundaries and triple points. A very good correlation exists between the stresses and the local value of the [001] misorientation angle Fig. 4.4(b). This is in accordance with the thermal anisotropy of Sn that should lead to maximum stresses for a misorienation angle of 90°. The yield strengths of pure Sn (at strain rate of 2×10⁻⁴/min) and Sn-3.5Ag alloy (at strain rate of 5×10⁻³/s) at 296 K are 11.0 MPa and 42 MPa, respectively [5, 3]. Therefore, the calculated Von Mises stress is found to be a significant fraction of the yield strength for both pure Sn and Sn-3.5Ag alloy.

![Figure 4.5: BSE micrograph depicting heterogeneous deformation or damage at grain boundaries and within grains](image)

Backscattered electron microscopy (BSE) was performed to investigate the damage in more detail, on a number of locations that showed large differences in stress level in the model calculation.
The first observation is that areas without stress concentrations consistently do not show fatigue damage. As an example, a BSE micrograph obtained inside grain 38 (Fig. 4.5(g)) does not show any microcracks. BSE micrographs were also taken from the centers of grains “20” and “25”. No detectable damage was observed as shown in Fig. 4.5(d) and (f); however, some structural evolution seemed to occur by sub-grain formation within dendrites.

The second observation is that fatigue damage is indeed associated with areas of high stress concentrations. Fig. 4.5(a) shows the microstructure after thermal cycling along part of the grain boundary between grain “38” and “43”. The micrograph shows failure along the grain boundary by sliding.

The boundary region between grains “46”, “41” and “50” is depicted in Fig. 4.5(b) which shows cracks near the grain boundary and a similar observation can be made from Fig. 6(c) showing the grain boundary between grains “36” and “20”.

Another BSE micrograph as shown in Fig. 4.5(e) was taken capturing the triple point between grains “14”, “20” and “6”. Cracks followed the grain boundaries encompassing the triple point and at the triple point more microcracks were encountered.
4.1.2 Sample $SAC_2$:

Figure 4.6: Sample $SAC_2$: optical micrographs (a) before and (b) after thermal cycling.

Fig. 4.6 shows polarization micrographs before (a) and after thermal cycling (b) of sample $SAC_2$. Similar to sample $SAC_1$ a number of grains with different crystallographic orientations are distinguished by contrast differences. The grain structure is very irregular in some regions marked by black outlining in Fig. 4.6(a). This irregular structure is caused by high densities of Sn-dendrites within the plane section. After thermal cycling, microcracks were localized mainly in grain boundary regions. The magnitude of the microcracks increased compared to the sample $SAC_1$.

Figure 4.7: Optical micrographs within marked areas “A” (a) and “B” (b) of Figure 4.6(b)

Fig. 4.7(a) shows a polarization micrograph obtained from the area marked by “A” in Fig.
4.6(b). Arrow “1” marks a crack opening, which nucleated at the grain boundary and followed it for some distance. Then the crack continued downward, leaving the boundary and growing into the grain. Arrow “2” points to a region within a grain were the Sn dendrites show slip bands. But also this damage seems grain boundary related. The optical micrograph in Fig. 4.7(b) shows the marked area “B” in Fig. 4.6(b). The grain structure is very irregular within this area, and the damage is more diffused. At position “3” the a microcrack follows a boundary perfectly. In the area marked by arrow “4”, the damage is more diffuse, but still within the boundary region.

![Image](a) ![Image](b)

Figure 4.8: Sample SAC₂: IPF intensity maps (a) before thermal cycling and (b) after thermal cycling

The [001] IPF map before thermal cycling of a part of the specimen is shown figure 4.8(a). The area captured by the OIM scans is marked between the white lines in the optical micrograph in Fig. 4.6(a). The marked area in 4.8(a) is re-scanned after thermal cycling, the [001] IPF map in shown in Fig. 4.8(b). Dark points are related to microcracks, crystallographic distortions within grains, and grain boundaries.

Similar as for the sample SAC₁, the grain structure in of specimen SAC₂ remained unchanged after thermal cycling.

![Image](a) ![Image](b)

Figure 4.9: Sample SAC₂: (a) Von Mises stresses from FE simulations (b) Misorientation map with respect to [001] axis

Fig. 4.9(b) shows the [001] misorientation map based on the OIM data depicted in Fig. 4.8(a).
It very well shows the irregular grain structure within in the marked areas of Fig. 4.6(a). The Von Mises stress distributed obtained from FE calculations based on the OIM data captured before thermal cycling is depicted in Fig. 4.9(a). The element size is $45 \times 38.97 \, \mu m$ and each layer contained 11,640 elements. The fatigue damage shown in Fig. 4.6(b) again shows a strong correlation to the stress concentrations calculated due to the Sn anisotropy.
4.2 The pure Sn specimens

4.2.1 Sample $Sn_1$:

![Figure 4.10: Sample $Sn_1$: optical micrographs of Sn specimen $Sn_1$ (a) before and (b) after thermal cycling](image)

Fig. 4.10(a) shows the polarization micrograph of the sample $Sn_2$. Compared to the SAC specimens the grain structure of this Sn specimen is much more regular. There is only one phase and the number of grains is less.

The polarization micrograph of the same sample after thermal cycling is depicted in Fig. 4.10(b). It shows fatigue damage which is concentrated along grain boundaries and within small grains. Arrow “A” shows a microcrack which follow a grain boundary. The region indicated by the arrows “E” also shows fatigue damage. The arrow “C” is pointing to areas where the polarization micrographs did not capture any grain. Note that cross polarization microscopy does not exhibit all orientations by contrast differences.

The grains indicated by the arrows “D” are no longer visible after thermal cycling. This could be caused by recrystallization, where small grains merge with larger neighboring grains.

OIM scans were taken from the marked area in Fig. 4.10(a) before thermal cycling. The IPF map is shown in Fig. 4.11(c). It shows more details in the grain structure compared to that revealed by the polarization micrographs. Also the grains marked by the arrows “C” in Fig. 4.10(b) are clearly visible now.

Fig. 4.11(b) shows the calculated misorientation angles [001] based on the OIM scan data. Two types of grain boundary angles can be distinguished. The high angle grain boundaries, which have a misorientation angle of approximately 63$^\circ$. This corresponds to the twinning of the (301) plane of the $\beta$Sn crystal [9].

The other type of grain boundary is the low angle grain boundary, where misorientation angle is less then 10$^\circ$.

Shown in Fig. 4.11(a) is the Von Mises stress distribution obtained from the FE simulations. The FE model is based on OIM scans taken before thermal cycling. $13,206 \times 2$ elements were considered for FEM modeling.
Figure 4.11: Sample $Sn_1$: (a) Von Mises stress distributed from FEM calculations (b) misorientation map with respect to the [001] crystal axis (c) IPF map before thermal cycling

The stresses are related to the high angle grain boundaries. The fatigue damage shown in Fig. 4.10(b) has a strong correlation with these high angle grain boundaries. The region indicated by the arrow “B” in Fig. 4.10(b) does not seem to show any cracks. However, the FE results show high stress concentration at this boundary. The inclination (with respect to the sample surface) of this grain boundary may be differ significantly from the other boundaries on this specimens surface due to its very irregular morphology. This could be the reason that this boundary did not show any.

4.2.2 Sample $Sn_2$

Figure 4.12: Sample $Sn_2$: IPF intensity maps (a) before thermal cycling and (b) after thermal cycling

The sample $Sn_2$ was not as thoroughly examined as the other three specimens.
The IPF maps captured before thermal cycling is shown in Fig. 4.12(a). The marked area was re-scanned after thermal cycling, see Fig. 4.12(b). It shows that the grain structure was changed after thermal cycling. The smaller grains seem to have merged with larger neighboring grains. Also some re-orientation of the crystal indicated by the red spots appeared in Fig. 4.12(b).
Fatigue damage was shown to occur in bulk SAC and pure Sn specimens under thermal cycling. For the SAC specimens the amount of fatigue damage increased with increasing range of the cyclic thermal load.

The damage was localized mainly along high angle grain boundaries. A combination of experiments (OIM) and calculations (FEM) indicated was used to interpret these findings. Finite element calculations showed stress concentrations along those boundaries. The location of the fatigue cracks was found to strongly correlate with the regions where the largest stresses encountered due to the thermal anisotropy of Sn.

OIM analysis confirmed that the grain shape and orientation remained unchanged after thermal cycling for SAC specimens. But the pure Sn specimen thermally cycled between -20 and 80 °C showed sign of re-crystallization.

Intrinsic thermal anisotropy of Sn is the determining factor in stress build-up and damage initiation in thermal cycling of mechanically unconstrained SAC and pure Sn specimens.
Appendix A

Alternative FE modeling: method 2

The method 2 described in this appendix is based on the grain shape and average orientation within the grains, as calculated by the OIM analysis software. Automatizing this method was not very easy and creating FE models was very time consuming. Therefore, the FE calculations (method 1) presented in Chapter 4 are not based on this method. However, in spite of this disadvantage this alternative modeling approach has some advantages over the other.

Procedure

The grain geometry obtained from the OIM analysis is imported in MARC/MENTAT as polyline curves, see Fig. A.1(a). The curves represent grain boundaries and the circled areas represent areas (grains) which have the same crystal orientation. The grain geometry is converted into a 2D FE mesh by “filling” each grain by the auto-mesh routine. The element size within a grain can be varied, resulting in a more coarse mesh within a grain compared to the FE mesh along grain boundaries, see Fig. A.1(b). Next the 2D FE mesh is expanded to a 3D mesh, the same way as described in Paragraph 3.2.1. The same assumptions regarding the “third dimension”, material properties, and loading condition as described in Section 3.1 were also used for this method. The main difference between the two methods is the way the crystal orientation is assigned. In method 1 each element within a grain was assigned with an unique orientation. In this alternative FE modeling approach, an averaged orientation is assigned to all the elements comprising one grain.

Results

Fig. 4.4(a) shows the FE results obtained from method 1. The element size used for the mesh is 60×51.96 µm.

Fig. A.1(c) shows the FEM results from method 2 based on OIM scans taken before thermal cycling of the specimen SAC1 shown in Fig. 4.3(a). A clean-up routine was applied to the OIM data. First the nearest-neighbor criterion was used to replace scanning points with a low confidence index (<0.1). Next grain dilation was
Figure A.1: (a) Grain geometry represented by polyline curves  (b) mesh (c) FEM results of SAC1 generated by the alternative FE modeling method

used with a minimum grain diameter of 300 µm. This was done to reduce the number of grains. Because otherwise the FE modeling would be too time consuming. The characteristic element size used in the grain boundary areas was 30 µm. The element size in the low interest areas was approximately 100 µm.

The number of element used in one element layer of the mesh for the common part modeled, for both methods is roughly the same (12,000). The FEM results from both methods correspond each other very well.
Appendix B

Example of orientations

This appendix shows how the OIM crystal orientation data can be modeled in Marc/Mentat with the aid of the orientation option. This option specifies the frame of reference of the anisotropic material properties.

The OIM data sheets contain the Euler angles of the indexed scan points in radians. In the IPF map of Fig. B.1, two points are highlighted by the local crystal lattice. The direction perpendicular to the square surface of the tetragonal is the [001] direction of the crystal. The normals of the two rectangular surfaces represent the crystal directions [100] and [010].

The Euler angles of point 1 are $\varphi_1 = 1.2892$, $\phi = 1.3561$, and $\varphi_2 = 4.9009$. The angles of point 2 are $\varphi_1 = 3.447$, $\phi = 1.3090$, and $\varphi_2 = 2.0612$.

Equation 3.6 gives the rotation matrix between the crystal principle direction, $\mathbf{e}_i^\text{S}$, and the global reference system of Marc/Mentat, $\mathbf{e}_i^{FEM}$. The rotation matrix of point 2 is:

$$
Q = R_{FEM} \cdot R^{\varphi_1} \cdot R^\phi \cdot R^{\varphi_2} = \begin{pmatrix}
-0.0761 & 0.3815 & 0.9212 \\
-0.5179 & -0.8046 & 0.2905 \\
0.8521 & -0.4550 & 0.2588
\end{pmatrix}
$$

Figure B.1: IPF map with two highlighted oriented lattices

The Euler angles of point 1 are $\varphi_1 = 1.2892$, $\phi = 1.3561$, and $\varphi_2 = 4.9009$. The angles of point 2 are $\varphi_1 = 3.447$, $\phi = 1.3090$, and $\varphi_2 = 2.0612$.

Equation 3.6 gives the rotation matrix between the crystal principle direction, $\mathbf{e}_i^\text{S}$, and the global reference system of Marc/Mentat, $\mathbf{e}_i^{FEM}$. The rotation matrix of point 2 is:

$$
Q = R_{FEM} \cdot R^{\varphi_1} \cdot R^\phi \cdot R^{\varphi_2} = \begin{pmatrix}
-0.0761 & 0.3815 & 0.9212 \\
-0.5179 & -0.8046 & 0.2905 \\
0.8521 & -0.4550 & 0.2588
\end{pmatrix}
$$

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Fig. B.2 shows left a screenshot of the orientation menu, which can be found in the material properties menu. There are several ways to specify the reference system of the material properties. The 3D anisotropic options was used. This options gives the possibility to specify the [100] and [010] principle directions of the crystal, respectively called vector 1 and 2. The third crystal principle direction [001] is automatically chosen perpendicular to the first two vectors. The vectors 1 and 2 correspond to the first two columns of the rotation matrix.

The model window of the screenshot of Fig. B.2 shows three elements with three different orientation. The red arrows represent the first crystal principle direction [100], the green arrows represents the second direction [010], and the blue arrows [001].

In the element “Lab. reference” crystal principle directions are aligned with the system of reference of the specimen or laboratory, $\mathbf{z}^{lab}$. The other two elements, “point 1” and “point 2”, show the principle direction of the crystal of the two highlighted points in Fig. B.1.
Appendix C

FE modeling: part of specimen surface

Before thermal cycling only a parts of the plane sections of the specimens was scanned by orientation imaging microscopy. This OIM data was used for the FE simulations of the samples $SAC_2$, $Sn_1$, and $Sn_2$. Only that part of the plane section from which there was crystallographic information available, could be modeled.

OIM scans of the complete plane section of the specimen $SAC_1$ were made after thermal cycling. Based on this crystallographic data a FE model was constructed, from which the results are shown in Fig. C.1(a).

A copy of this model was used to construct the FE model which simulates a part of sample surface. From the whole surface model a strip of elements on the upper and lower part was removed. Leaving a FE mesh similar shaped as a mesh that will be created from OIM scan from part of a sample surface. The Von Mises stress distribution resulting from this FE model are shown in Fig. C.1(b).
The Von Mises stress distribution calculated from both models looks very alike. Only in the free edge regions of model (b), some extra stress concentrations arise. From this it can be concluded, that the modelling of a part of a sample surface, does not effect the overall calculation results. The influence is restricted to a relative small area near the free edges.
Bibliography


