Microstructure, Texture, and Hardness Gradients in Aluminum Diffusion-bonded to Aluminum Oxide

by

Ching-Te Lin

Submitted to the Department of Materials Science and Engineering in partial fulfillment of the requirements for the degree of Doctor of Philosophy in Materials Engineering at the MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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Abstract

Experimental results on the evolution of grain structure, texture, and mechanical properties in solid-state diffusion-bonded Al–Al₂O₃ multi-layered composites with various Al thicknesses are studied. The results showed the existence of gradients in grain size and shape, texture, and mechanical properties as a result of the interface constraints on the mobility of grains close to the interface. Grains near the interface are observed to be elongated and slanted and are about 20% bigger than the equiaxed grains that are near the surface. As a result of the rotation of grains, although the regular cube texture is present in the regions away from the interface, the rotated cube texture is present in the regions near the interface. Mechanical properties in those composites were investigated by means of the microhardness test using Vickers microhardness indentation. It is shown that grains near the interface have a hardness of about 10% lower than grains near the surface. This observation is seen to compare favorably with the grain size and texture measurements. A finite-element model using rate-dependent polycrystalline plasticity constitutive relations was adapted to simulate the evolution of grain structure and texture near the interface. Although discrepancies exist between the predicted and experimentally observed texture as a result of the limitation on the current two-dimensional model, the general predicted results on the shape and size of the grains showed in reasonable agreement with the experimental observations.

Thesis Supervisor: Subra Suresh
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Chapter 1

Introduction

1.1 Motivation

Metal–ceramic multi–layered composites are of considerable scientific interest because of their wide variety of practical applications. Layered structures comprising metals, ceramics, and polymers are designed to obtain desirable functions in such applications as electronic, optical, and magnetic devices, thermal barrier coatings and structural components. For example, surface coatings, such as thermal barrier coatings, high-temperature corrosion resistant coatings, and diffusion barrier coatings, are deposited on substrates to achieve improvements in mechanical, chemical, and tribological performance. However, in any type of multi–layered structures, thermal residual stresses are inevitably generated due to the mismatch of the thermal expansion coefficients between dissimilar materials during temperature fluctuations. Since a high magnitude of residual stresses is regularly observed in the vicinity of sharp interfaces [1,2], a knowledge of the microstructure and mechanical properties in the interface region is essential for ensuring the structural integrity of the layered composites during fabrication and in service.

Microstructure evolution, especially texture evolution, in metals has received attention as a result of its important effect on the overall response of materials. Available
information on texture [3–40], however, pertains to homogeneous metal sheets [3–7] or thin films on substrates [34–40]. To the author’s knowledge, microstructure evolution in multi-layered structures comprising ceramics and metals with thicknesses significantly larger than characteristic microstructural dimension (e.g., grain size) has hitherto not been reported in the open literature. In these systems, gradients of microstructure and mechanical properties are likely to exist due to the presence of the metal–ceramic interface.

1.2 Objective

The objective of the present work is to study the evolution of grain structure, texture, and mechanical properties in a pure aluminum layered with ceramics after solid-state diffusion bonding. This investigation includes processing, experiments on microstructures and texture characterization and local mechanical properties analysis, and detailed computational simulations. The specific objectives of the work include:

- To quantify experimentally the grain structure gradient, texture gradient, and mechanical properties gradient in aluminum for metal–ceramic bilayer and tri-layer composites.
- To investigate the effect of the thickness of the metal layers on the gradients in grain size and shape, texture, and local mechanical properties (primarily indentation hardness) in layered materials.
- To highlight the difference in the evolution of the grain structure, texture, and mechanical properties between metal–ceramic layered composites and monolithic metals.
- To conduct detailed computational analyses of crystal plasticity using finite element method to identify the mechanisms responsible for the gradients in grain geometry, texture, and hardness of aluminum diffusion bonded to polycrystalline alumina.
In all of the experiments carried out, the material system chosen as a model is an Al—
\(\text{Al}_2\text{O}_3\) layered composite. This choice was motivated by a number of considerations:
(i) Both Al and \(\text{Al}_2\text{O}_3\) are widely used in engineering structural components; (ii) Their
thermomechanical properties as functions of temperature are well established; (iii) The microstructure, texture, and mechanical properties of monolithic aluminum and
its alloys have been systematically studied and are well documented; (iv) Aluminum
has a low melting point (933°K), and therefore, critical experimental conditions are
easily obtained.

1.3 Brief review of texture in F.C.C. metal sheets

Rolling texture and recrystallization texture are two texture phenomena that are of-
ten adopted to describe specific grain orientations in metal sheets. The textures of
the sheet are described by choosing the ideal orientations that comprise the crys-
tallographic plane \((hkl)\) and the crystallographic direction \([uvw]\) that are parallel to
the sheet surface and the rolling direction, respectively. Rolling texture evolves in
metal sheets when they are subjected to the rolling process. During plastic deforma-
tion, grains in a polycrystal tend to rotate, and each grain slips and rotates in a way
that is determined by the imposed forces and by the slip and rotation of adjoining
grains. Recrystallization texture occurs when cold—worked metals are annealed at
temperatures above the recrystallization temperature.

1.3.1 Rolling texture in F.C.C. metal sheets

In F.C.C. metals, two rolling textures predominate [12]. The \{110\}<112> texture
\((\alpha\text{—brass—type or alloy—type texture})\) develops during initial deformation and the
\{112\}<111> texture (copper—type or pure—metal—type texture) develops during ex-
tensive plastic deformation. Dillamore and Roberts [13] have established full sets
of pole figures covering the development of rolling texture in aluminum and silver.
These figures suggest that the \(\alpha\text{—brass—type texture}, \{110\}<112>, \) which is typical for
F.C.C. materials that possess low stacking fault energy, develops after small thickness reductions, and the pure-metal-type texture, which is typical for F.C.C. materials that possess intermediate and high stacking fault energy, takes over when the amount of thickness reduction is significant. A similar observation [14] was also made on two other F.C.C. metals: copper behaves similarly to aluminum, and 70/30 brass behaves similarly to silver. In all F.C.C. metals, the pattern, alloy-type → pure-metal → \{112\}<111>, is suggested for rolling texture with increasing reduction in thickness.

1.3.2 Recrystallization textures of F.C.C. metal sheets

The most common recrystallization texture is the “cube texture, i.e., \{100\}<001>” which has been observed in many F.C.C. metals such as Cu, Al, Au, and Ni. In this texture, a cube plane, (100), and a cube edge, [001], lie parallel to the plane and the rolling direction of the sheet, respectively. In commercial aluminum, a cube texture is usually accompanied by the “retained rolling texture” [16,17] or “S texture (\{123\}<634>)” [18,19].

1.4 Theories of Recrystallization Texture of F.C.C. Metal Sheets

Two main theories [20], the oriented nucleation theory and the oriented growth theory, have been proposed to explain the formation of recrystallization texture. In the oriented nucleation theory, the nucleation process is the critical factor that determines the nuclei that are able to contribute to the recrystallization texture. However, in the oriented growth theory, only grains having the best orientation relationship to the deformed matrix can grow and form the recrystallization texture [3].

Dillamore and Katoh [21] proposed a specific mechanism of oriented nucleation in which they claimed that the highly misoriented transition bands formed during deformation were the nucleation sites. Hjeien and Nes [22] found high concentra-
tions of cube-oriented nuclei and Goss (i.e., $\{110\}<001>$) nuclei in the transition bands between different variants of the copper (i.e., $\{112\}<111>$) and the brass (i.e., $\{011\}<211>$) deformation texture components in their aluminum study.

Various researchers [23–27] have proposed theories of recrystallization texture in polycrystalline metals based on oriented growth. Beck and Hu [23] and Stüwe [27] showed that the recrystallization texture of aluminum could be explained in terms of $<111>$ rotation relationships [28]. A compromise theory, the viz., oriented-nucleation/growth-selection theory, was also accepted by a number of researchers [29–32]. This theory suggests that the nucleation process governs the range of available orientations and also suggests that there is further selection from among these due to the orientation-dependence of growth rate [28].

1.5 Organization

The thesis is arranged in the following sequence.

- Chapter 2 describes the materials investigated in the current study along with the experimental procedure, including the key parameters for solid state diffusion bonding, materials treatments prior to bonding, and methodology of grain size and hardness measurements.

- Chapter 3 comprises the procedure for X-ray raw data correction and a brief introduction to two X-ray interpreting methods, pole figure (PF) and orientation distribution function (ODF), used in the current study.

- Chapters 4 and 5 present the experimental results, which include observations of gradients of grain structure, texture, and mechanical properties in Al in Al–$\text{Al}_2\text{O}_3$ bilayer and $\text{Al}_2\text{O}_3$–Al–$\text{Al}_2\text{O}_3$ trilayer composites as a function of distance from the interface with $\text{Al}_2\text{O}_3$ (with various Al thicknesses).
• Chapter 6 begins with a concise review of a number of crystal plasticity models, followed by an introduction of the model adopted in this work, and concludes with the results of simulations.

• The thesis concludes in chapter 7 with a summary of the findings of the current study and suggestions for future work.
Chapter 2

Materials and experimental procedure

2.1 Materials

The material investigated in the current study was a 6.25 mm thick commercial purity 1100–F aluminum (Al) sheet. This material was received from PIERCE ALUMINUM CO. (Canton, MA), and the compositions is given in Table 2.1.

To reveal the grain structure, optical microscopy was performed using electrolytic technique by immersing specimens into acid solution (10ml HBF$_4$ + 200ml H$_2$O) for 2 to 3 minutes under constant voltage (30 V. dc) [41,42]. Due to different orientations of grains, the resulting anodic films gave different color contrast when viewed under a plane–polarized optical microscope, and therefore, grains can be distinguished by different colors. Figure 2-1 shows the grain structure of the Al sheet as received. This picture was taken on the through thickness plane that is perpendicular to the trans-

| Table 2.1: Compositions of 1100–F Al |
|------------------|---|---|---|---|---|---|---|
| material | Al | Si+Fe | Mn | Mg | Zn | Ti | others |
| composition (%) | > 99.0 | < 1.0 | < 0.03 | < 0.03 | < 0.05 | < 0.03 | < 0.15 |

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verse direction (TD). As defined here and in all the subsequent figures, RD, TD, and ND stand for the rolling, transverse (in-plane), and normal directions, respectively. It can be seen that the initial Al sheet consists of long ribbons of grains (more than 250 μm long in the RD) of about 5 μm thickness in the ND.

After the initial investigation on the raw material, a number of strips were cut from the Al sheet, and they were cold rolled to have a thickness reduction of 84% (strip A), 90% (strip B), and 97% (strip C), individually, with the corresponding true thickness reduction \( \varepsilon = 1.8, 2.3, \) and 3.4. Figures 2-2(a) and 2-2(b) show the grain structure of strips A and B, respectively. Both pictures were also taken on the through thickness plane that is perpendicular to the transverse direction (TD). As can be seen in both figures, strips A and B also comprise long ribbon grains but with less thickness in the ND.

The crystallographic texture of strips A and B were measured on the normal plane by diffraction techniques using Rigaku RU200 (Rigaku Denki Corp., Tokyo, Japan) X-ray diffractometer with a copper target operating at 60 kv and 150 mA. After corrections of the pole figure raw data (see chapter 3 for details), pole figures (PFs)
Figure 2-2: Grain structure of 1100-F aluminum after having undergone (a) 84% and (b) 90% thickness reduction. The photos were taken on the cross sectional plane whose normals are perpendicular to the TD.
Figure 2-3: The four incomplete (111) pole figures of strip A at the corresponding depth of (a) 0 µm, (b) 250 µm, (c) 500 µm, and (d) 750 µm below the sheet surface. were replotted using the Tecplot graphic software (Amtec Engineering, Inc., Bellevue, WA).

Figures 2-3(a) to 2-3(d) and 2-4(a) to 2-4(c) show the incomplete (111) pole figures of strip A (1 mm in thickness) and B (600 µm in thickness), respectively, at the corresponding depth of 0 µm, 250 µm, 500 µm, and 750 µm in strip A and 0 µm, 250 µm, and 500 µm in strip B below the sheet surface, respectively. It can be seen that after 84% and 90% thickness reduction, the texture in strips A and B were mainly composed of the typical orientations for FCC metals after cold rolling. This specific texture often refers to the so-called P fibre that comprises brass (B), \{110\}<112>, copper (C), \{112\}<111>, and S, \{123\}<634>. Table 2.2 lists the relation between Miller indices and Euler angles (see chapter 3 for detailed definitions) of a number of important orientations discussed in the present study.

The orientation distribution functions (ODFs) were computed from the three pole figures \{111\}, \{200\}, and \{220\} using the series expansion method with \( l_{\text{max}} = 22 \)}
Figure 2-4: The three incomplete (111) pole figures of strip B at the corresponding depth of (a) 0 μm, (b) 250 μm, and (c) 500 μm below the sheet surface.

[43]. The software used to calculate the ODF was PopLa (Los Alamos National Laboratory, Los Alamos, New Mexico). Figures 2-5(a) to 2-5(d) and 2-6(a) to 2-6(c) show the ODFs of strips A and B, respectively, at the depth of 0 μm, 250 μm, 500 μm, and 750 μm (for strip A only) below the sheet surface, respectively. It can be seen that the main textures in all the three figures are B, C, and S.

The ceramic to be bonded to Al in the current study was a 1 mm thick fully dense

Table 2.2: Miller indices and Euler angles of a number of important orientations used in the present study

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Miller indices</th>
<th>Euler angles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube</td>
<td>{001}&lt;100&gt;</td>
<td>0° 0° 0°/90°</td>
</tr>
<tr>
<td>B</td>
<td>{011}&lt;211&gt;</td>
<td>35° 45° 0°/90°</td>
</tr>
<tr>
<td>S</td>
<td>{123}&lt;634&gt;</td>
<td>59° 34° 65°</td>
</tr>
<tr>
<td>C</td>
<td>{112}&lt;111&gt;</td>
<td>90° 30° 45°</td>
</tr>
</tbody>
</table>
aluminum oxide (Al₂O₃, 99.6%), obtained from ACCUMET (Hudson, MA.). In order to provide a favorable surface condition for mechanical attachments during diffusion bonding, the Al₂O₃ surfaces were lapped to have a roughness of about 10 μm.

2.2 Bonding procedure

Solid state diffusion bonding method was employed to bond the pure aluminum with aluminum oxide. In this process, particular attention should be paid to three variables [44]. They are the bonding temperature, the applied load, and the holding time. The bonding temperature should be such that it can aid the interdiffusion of atoms across the interface of the bonded materials and also assist the surface deformation. It is typically above 0.6 of the absolute melting temperature of the least refractory of the two materials [45]. The applied load should be able to provide a tight surface contact between two materials. The holding time, on the other hand, should be kept to a minimum, at a specified bonding temperature and a specified load, to avoid any dramatic change in physical and mechanical properties.

The Al strips were first cut into a number of 13 mm x 13 mm square pieces and surface-finished prior to diffusion bonding in order to remove asperities. To remove contamination and residue from the surfaces of both Al and Al₂O₃, materials were then cleaned using acetone at room temperature and degassed in a vacuum (10⁻⁴ Pa) chamber at 150°C for one hour. Once the degassing was finished, the temperature in the vacuum chamber was directly brought up to 590°C at a 10°C/min. heating rate, and an uniaxial compressive load of 15 MPa was then applied when the desired temperature was reached. After holding the system at 590°C for 10 hrs., materials were then cooled to room temperature within one hour, and the load was then removed. The thicknesses of strips A, B, and C were reduced from 1 mm, 600 μm, and 200μm, respectively, to about 850 μm, 525 μm, and 175 μm, respectively, after diffusion bonding. To prevent the formation of bonds between Al and the fixtures, graphite powder was used as a lubricant.
Figure 2-5: The complete orientation distribution plots of strip A at the corresponding depth of (a) 0 μm, (b) 250 μm, (c) 500 μm, and (d) 750 μm below the sheet surface.
Figure 2-6: The complete orientation distribution plots of strip B at the corresponding depth of (a) 0 μm, (b) 250 μm, and (c) 500 μm below the sheet surface.
2.3 Methodology for grain size measurement

It is important to recognize that although a number of methods have been proposed to measure the grain size, the estimation of grain size is never a precise measurement. In the current study, the Hilliard single-circle procedure [46,47] was employed based on the consideration of non-equiaxed grain shapes in the regions near the interface. Circular intercept procedures are recommended [48,49] since the ambiguous intersections at ends of test lines can be eliminated, and the departures from equiaxed grain shapes can be compensated automatically, without overweighting any local portion of the field. A reasonable estimate of the grain size can be made using this method if the departure from an equiaxed shape is not excessive (grain aspect ratio ≤ 3).

To measure the grain size, samples were first etched, and standard 9 cm x 12 cm color microscope images were taken under optical microscopes. The 9 cm x 12 cm photos then were enlarged to have a size of 18 cm x 24 cm. Before applying the Hilliard single-circle procedure, individual grains were first outlined manually. In order to obtain a high degree of accuracy, three circles with different diameters were drawn with respect to the same center point. Figure 2-7 schematically illustrates the grain size measurement at different depths to the interface using Hilliard single-circle method. Although only a single circle is shown at each location in Fig. 2-7, in reality, there were three concentric circles that were applied at each location. The fields were chosen to have representative results in grain size distribution. However, since the fields of view were small, a number of fields were investigated at each depth.

The grain size in the current work was calculated as follows. For each field count,

\[
\bar{N} = \frac{N_i}{L/M},
\]

where \(N_i\) is the number of intercepts counted on the field, \(L\) is the total measured line length, \(M\) is the magnification, and \(\bar{N}\) is the average value of the number of
Figure 2-7: Schematic of grain size measurement at different depths from the surface.

intercepts per unit length of the measured line. The grain size is then defined as the mean linear intercept value for each field, \( \bar{l} \), by

\[
\bar{l} = \frac{1}{N} .
\]  

2.4 Methodology for hardness measurement

Mechanical properties within the Al layer in Al–Al₂O₃ diffusion bonded samples were characterized by microhardness tests. Since the yield strength is generally related to the hardness index, indentation results should be able to provide a general understanding of mechanical properties change within the Al layer after diffusion bonding. Due to the limitation of the specimen’s geometry on the mechanical testing, in the current study, indentation offers the only means for quantifying the gradients in local mechanical property arising from the gradients in grain geometry and texture as a function of distance from the interface. In order to obtain valid hardness results
in these regions, the Vickers microhardness indentation method was employed. The indentation tests were performed using a Vickers indentor (pyramid angle = 136°) on a LECO DM–400 (LECO Corp., St. Joseph, Michigan) micro-hardness testing machine. This machine has the capability of performing tests at loads in the range of 10 g to 1000 g. All microhardness tests were carried out at a load of 25 g and with a 20-second dwelling time between the end of the loading step and the beginning of the unloading step. The hardness measurements were made at room temperature. Unless particularly specified, all the tests were performed on the cross-sectional plane whose normal is perpendicular to the RD. The hardness value at each depth was obtained by averaging the measured hardness at different locations along the interface at that specified depth.

Figures 2-8(a) and 2-8(b) schematically illustrate the definitions of a number of important parameters in the Vickers test. In these figures, \( P \) (kg) is the load, \( h \) (mm) is the depth of the indented area, and \( d \) (mm) is the diagonal length of the indented area on the tested plane. For a Vickers indentor with a pyramid angle of 136°, the interrelations between \( P, h, \) and \( d \) can be expressed as follows.

\[
H_v = 1.85 \frac{P}{d^2}, \quad \text{and} \quad (2.3)
\]

\[
h = \frac{d}{l}. \quad \text{(2.4)}
\]
It is recognized that the plastic zone size after the Vickers tests is about twice the size of the indented area. That is, a three-dimensional sphere with a radius of \( d \). By using Eq. 2.3, the value of \( d \) was obtained as 40 \( \mu \text{m} \). Therefore, in the current study, each indented field is separated by about 100 \( \mu \text{m} \), and at least 20 fields were indented at each depth with respect to the interface.
Chapter 3

Texture analysis by X-ray diffraction

X-ray back reflection technique using a monochromatic beam (Cu anode, wavelength 1.5405Å) was adopted in the current work to provide statistical information on the macroscopic level texture analysis in the form of pole figures and orientation distribution functions. The typical area on a sample to be illuminated by X-ray was about 1 mm x 5 mm in size, which encompassed a large number of grains to be analyzed. Figure 3-1 shows the geometry and definitions of angles in the current texture analysis schematically. In each run of the texture measurement for a specific (hkl) plane, the X-ray beam source and the detector were arranged in such a way to have a $\theta$–$2\theta$ relationship with respect to the sample. The angle $\theta$ was predetermined experimentally, and in Al, the theoretical values of $2\theta$ are 38.47°, 44.74°, and 65.13° for the three (hkl) planes (111), (200), and (220), respectively. By Bragg’s law, only grains with the (hkl) planes that are parallel to the R-plane can produce the recorded intensity, where the R–plane is perpendicular to the plane that comprises both the X–ray beam source and the detector path. After setting up the geometry, the sample was moved to a new position by tilting it by an angle $\alpha$ and then rotating it about its normal by an angle $\beta$ during the scanning. By moving a sample in the $\alpha - \beta$ directions simultaneously, grains with their (hkl) planes parallel to the R–plane at the current configuration could then contribute to the recorded intensity. In the current analyses,
both $\alpha$ ($15^\circ$–$90^\circ$) and $\beta$ ($0^\circ$–$360^\circ$) were moved at a $5^\circ$ interval.

In order to increase the amount of the reflected intensity that can be received by the detector, the receiving slit was removed during the texture measurement. Although this may cause a decrease in the angular resolution, for materials with cubic crystal structure, this decrease is usually not important since there is a large angular separation between reflections from different crystal planes, and overlapping of adjacent reflections is unlikely in most cases [50].

All textures were examined by measuring the three incomplete pole figures, 111, 200, and 220, in the range of $\alpha$ from $15^\circ$ to $90^\circ$. After correction of the experimental data with respect to three systematic errors (the background scattering, the absorption effect, and the defocusing error), the two-dimensional pole figures were reconstructed. Since the interpretation of pole figures are sometimes ambiguous due to the overlapping of the same poles from different orientations, the quantitative three-dimensional ODF were also calculated from the three pole figures, 111, 200, and 220.
3.1 X–ray raw data correction

In order to obtain high accuracy in the texture results, a correction of raw data must be performed. In general, three experimental errors must be corrected: background scattering, absorption effect, and defocusing error [51]. A computer program using turbo C language was written to correct these effects automatically (Appendix I).

3.1.1 Background scattering correction

The background scattering results from the X–rays that are not diffracted by the specified (hkl) plane but still reach the detector and therefore are recorded. This error can be corrected by repeating the texture measurement over the entire range of $\alpha$ at an angle that is a few degrees away from the $2\theta$ for the specified (hkl) plane. The background intensity can then be subtracted from the original raw data to obtain the intensity that is diffracted by the (hkl) plane only. In the current study, this correction can be performed by the X–ray diffractometer Rigaku 200 automatically.

3.1.2 Absorption effect correction

The second error that needs to be corrected is the absorption effect. This effect will have no substantial influence on the experimental result, provided that the specimen has a thickness that is much greater than the extinction depth of the X–rays. However, since the texture at a distance less than 200 $\mu$m needs to be measured, a correction to the raw data is required. This absorption correction factor is given by

$$F_{\text{abs}}(\alpha) = \frac{1 - \exp \left( -\frac{2\mu d}{\sin \theta} \right)}{1 - \exp \left( -\frac{2\mu d}{\sin \theta \cos \alpha} \right)}, \tag{3.1}$$

As can be seen in this equation, the absorption correction factor depends on the Bragg angle, $\theta$, the tilt angle, $\alpha$, the depth, $d$, and the absorption coefficient, $\mu$, of the sample. In general, the first three parameters are experimental settings and
therefore are known directly. The last parameter, \( \mu \), on the other hand, can be obtained by fitting \( I_0 \) and several values of \( I(d) \), at specified \( \theta, \alpha, d \) and wavelength of the target used in anode into Eq. (3.2) using linear regression.

\[
\ln \frac{I(d)}{I_0} = -\mu d, \quad (3.2)
\]

where \( I_0 \) is the reference state intensity and \( I(d) \) is the intensity for a specified specimen with the thickness, \( d \). The value of the absorption coefficient, \( \mu \), for aluminum is found to be \( 0.196 \times 10^{-5} \text{Å}^{-1} \) [35].

Figures 3-2(a) to 3-2(d) show the absorption factor \( F_{\text{abs}} \) as a function of the tilt angle, \( \alpha \), for four different specimen thicknesses, 150 \( \mu \text{m} \), 100 \( \mu \text{m} \), 50 \( \mu \text{m} \), and 25 \( \mu \text{m} \), respectively. It can be seen that the attenuation effect of the X-ray intensity increases as the specimen thickness decreases. Moreover, different Bragg angles give different degrees of attenuation.

### 3.1.3 Defocusing error correction

The last error that needs to be corrected is the defocusing error. It arises when the specimen rotates out of the vertical plane (i.e., \( \alpha \neq 90^\circ \)). Due to the fact that when the upper portion moves in front of and the lower portion moves behind the focusing circle, the diffracted beam widens at the receiving slit, the measured intensity therefore decreases as \( \alpha \) departs from \( 90^\circ \). In general, this error can be corrected by either slit adjustment or calculation. In the current study, although the receiving slit was chosen to be optimized (i.e., removed), calculations were still needed in order to correct the strong defocusing error.

To correct the defocusing effect, a thick polycrystalline Al sample with random orientations was made from Al powders (purity \( \geq 99.9\% \), particle size \( \leq 10 \mu \text{m} \)) by mixing those powders with a colloid on a glass slide. A number of texture measurements were then carried out on this sample at three specific (hkl) planes, (111), (200), and (220),...
Figure 3-2: The absorption factor $F_{\text{abs}}$ as a function of the tilt angle, $\alpha$, for four different specimen thicknesses, (a) 150 $\mu$m, (b) 100 $\mu$m, (c) 50 $\mu$m, and (d) 25 $\mu$m, respectively.
through the ranges of $\alpha$ (15° to 90°) and $\beta$ (0° to 360°) at 1° steps. The diffraction intensity at each $\alpha$ was then calculated by averaging the values of the entire $\beta$ range. The defocusing factor, $F_{\text{def}}(\alpha)$, at each $\alpha$ was obtained by taking the value of $\frac{F_{\text{def}}(90)}{F_{\text{def}}(\alpha)}$. Those values were then fitted into polynomials to find the corresponding $F_{\text{def}}(\alpha)$ and $\alpha$ (see Appendix I for polynomials). Equation 3.3 calculates the completely corrected intensity.

$$I_{\text{corr},hkl}(\alpha) = F_{\text{abs},hkl}(\alpha) \cdot F_{\text{def},hkl}(\alpha) \cdot [I_{\text{raw},hkl}(\alpha) - I_{\text{bkg},hkl}(\alpha)], \quad (3.3)$$

Figures 3-3(a) to 3-3(c) show the defocusing factor, $F_{\text{def}}(\alpha)$, as a function of the tilt angle, $\alpha$, for three reflection planes, (111), (200), and (220), respectively. As can be seen in those figures, strong defocusing occurs as $\alpha$ decreases. Figures 3-4(a) and 3-4(b) show the pole figures of the thick Al sample before and after correction, respectively. It can be seen that false information could arise if the correction is not done.

### 3.2 Pole figure representation

Two-dimensional pole figure representation is a very common way to interpret the experimental data from texture measurements. It expresses the orientation distribution of a certain set of crystallographic plane normals in spherical polar coordinates that are determined by the sample coordinates. Figure 3-5 gives a schematic example of a pole figure plot. In this example, each data point with a specific $(\alpha, \beta)$ coordinate is located on the plot by the (hkl) stereographic projection. $\alpha$ and $\beta$ are represented as the magnitudes of a stereographic radius vector and the polar rotation angle of the radius vector, respectively. The intensity distribution can be plotted as a set of isodensity contour lines. For instance, the pole figure for a cubic polycrystalline with strong (100)[001] texture looks similar to what is shown in Fig. 3-5 when stereograph-
Figure 3-3: The defocusing factor $F_{\text{def}}$ as a function of the tilt angle, $\alpha$, for three reflection planes, (a) (111), (b) (200), and (c) (220).
Figure 3-4: The pole figures of the thick random orientated aluminum sample: (a) before and (b) after intensity correction.

ically projected onto the (111) pole figure. The angular difference in the $\alpha$ direction between the poles and the center of the plot is close to $54.7^\circ$. This interplanar angle $\phi$ between any two planes $(h_1,k_1,l_1)$ and $(h_2,k_2,l_2)$ in a cubic crystal can be obtained from the following equation 3.4.

$$\cos \phi = \frac{h_1 h_2 + k_1 k_2 + l_1 l_2}{\sqrt{(h_1^2 + k_1^2 + l_1^2)(h_2^2 + k_2^2 + l_2^2)}}. \quad (3.4)$$

Due to the cubic symmetry, four poles are supposed to be presented on this plot. It should be noted that although they are equi-spaced along the $\beta$ direction, their locations depend on the alignment of the sample during texture scan. That is, $\beta$ does not measure the angle between the planes but rather reflects the symmetry of the material with respect to the reference plane.
The ODF is a quantitative three-dimensional texture analysis. Due to the fact that the pole figures only describe the distribution of a certain plane normal of set \{hkl\} in the sample coordinates, not the distribution of crystal orientation, and the interpretation of pole figures are sometimes ambiguous because of the overlapping of the same poles from different orientations, the quantitative three-dimensional ODF were also calculated from the three pole figures, 111, 200, and 220. The series expansion method [52–54] with $l_{\text{max}} = 22$ was used. The ghost effect during computation was also eliminated. Based on the cubic crystal and orthorhombic sample symmetries, the textures were represented in a reduced Euler angle space ($0^\circ \leq \varphi_1, \varphi, \varphi_2 \leq 90^\circ$) and plotted with the isodensity contour lines in $\varphi_2 = \text{constant}$ sections through the Euler space at the interval of $5^\circ$.

In general, there are Euler angles of the first kind ($\varphi_1 \varphi_2$) and of the second kind.
(Ψ,Φ). The relationship between these two kinds are expressed as \[43\]

\[
\begin{align*}
\varphi_1 &= \Psi + \frac{\pi}{2}, \\
\varphi &= \Theta, \\
\varphi_2 &= \Phi - \frac{\pi}{2}.
\end{align*}
\]

(3.5) (3.6) (3.7)

In the current study, the Euler angles of the first kind were used and the definition is shown in Fig. 3-6. In this figure, one begins with an orientation of the crystal coordinate system \((X',Y',Z')\) in which the axes are parallel to those of the sample coordinate system \((X,Y,Z)\). For all the ODF figures presented in the current work, the \(X, Y,\) and \(Z\) correspond to the material system RD, TD, and ND, respectively. The crystal coordinate system is first rotated about the \(Z'\)-axis through the angle \(\varphi_1\), then about the \(X'\)-axis (in its new orientation) through the angle \(\varphi\) and finally, once again about the \(Z'\)-axis (in its new orientation) through the angle \(\varphi_2\). The rotation is therefore represented by the three Euler angles \((\varphi_1, \varphi, \varphi_2)\). It is customary to represent the three parameters of the rotation as Cartesian coordinates in a three dimensional space, the orientation space or Euler space. Each point of the Euler space then corresponds to a particular rotation, and conversely each rotation or crystal orientation leads to a point in the three dimensional space.

The relation between the Euler angles \((\varphi_1, \varphi, \varphi_2)\) and Miller indices \((hkl)[uvw]\) can be expressed as

\[
\begin{align*}
\varphi_1 &= \arcsin \left[ \frac{w}{\sqrt{u^2 + v^2 + w^2}} \ast \frac{\sqrt{h^2 + k^2 + l^2}}{\sqrt{h^2 + k^2}} \right], \\
\varphi &= \arccos \left( \frac{l}{\sqrt{h^2 + k^2 + l^2}} \right), \\
\varphi_2 &= \arccos \left( \frac{k}{\sqrt{h^2 + k^2}} \right).
\end{align*}
\]

(3.8) (3.9) (3.10)
Figure 3-6: The schematic diagram of the definition of Euler angles ($\phi_1\phi_2\phi_3$) in orientation distribution function. "XYZ" is the material coordinate system, and "X'Y'Z'" is the crystal coordinate system.
Chapter 4

Experimental results for Al–Al$_2$O$_3$ bilayer composites

In this chapter, experimental results that encompass the evolution of grain structure, texture and hardness within the Al layer as a function of position with various thicknesses for Al–Al$_2$O$_3$ bilayer composite after solid state diffusion bonding are presented. Due to the presence of the Al–Al$_2$O$_3$ interface, all cases show gradients within the Al layer in the through-thickness direction (ND). The grain structure change, in the current study, was characterized by grain shape change and grain size measurements. The mechanical properties change, on the other hand, was characterized by microhardness measurements. All the specimens had an in-plane dimension of 13 mm x 13 mm along with various thicknesses. Unless mentioned, all the micrographs were taken within the regions between 2 mm and 4 mm from the edge of the ceramic (in the longitudinal direction)

4.1 Results of grain structure examinations

Investigations of grain structure (shape and size) within the Al layer after diffusion bonding was carried out using the methods described in chapter 2. Results for Al–Al$_2$O$_3$ bilayer composites with various Al thicknesses are presented in this section.
Figure 4-1: The distribution of grain shape within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm from two different perspectives. (a) on the plane whose normal is parallel to the longitudinal direction (RD) and (b) on the plane whose normal is parallel to the transverse direction (TD).
4.1.1 Grain structure in an Al–Al₂O₃ bilayer composite with an Al thickness of 1 mm

Figures 4-1(a) and 4-1(b) show the distribution of grain shape within the Al layer in an Al–Al₂O₃ bilayer specimen where both the Al and Al₂O₃ layers are 1 mm thick, from two different perspectives. Figure 4-1(a) was taken on the plane whose normal is parallel to the longitudinal direction (RD). Figure 4-1(b), on the other hand, was taken on the plane whose normal is parallel to the transverse direction (TD). It can be seen that in both figures, grains in the regions near the interface are elongated and slanted, and they are equiaxed in the regions away from the interface. In order to reconstruct the three-dimensional grain shape, another picture was taken on the plane whose normal is parallel to the ND at a distance of 50 μm away from the interface. The in-plane grain shape within the Al layer is presented in Fig. 4-2. As can be seen in this figure, most of the grains are equiaxed. Figure 4-3 shows the distribution of grain shape along the entire Al–Al₂O₃ interface (along the RD). It is
Figure 4-3: The distribution of grain shape along the entire Al–Al₂O₃ interface (along the RD) within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm.
Figure 4-4: The schematic diagram showing the locations where the grain size measurements were performed.

shown that about two-thirds of the interface is covered with slanted grains.

Figure 4-5 shows the distribution of grain size within the Al layer. Unless particularly mentioned here and in the subsequent figures, the grain size measurements were carried out at locations within zone “a” (Fig. 4-4). In this figure, zone “a” represents the region that is about 2 to 3 mm from the free edge of the specimen. Within this region, grains near the interface are slanted and elongated. Zone “c” represents the region that is around the central part of the specimen, and within it, grains are equiaxed even in the regions close to the interface. Due to the creeping effect at high temperatures during bonding, the 1 mm thick Al layer experienced a 15% to 20% reduction in thickness (final thickness is about 800 μm). In Fig. 4-5, a decreasing trend in grain size is observed with increasing distance from the interface in the through-thickness direction.

Figure 4-6 shows the distribution of grain size around the central regions within the Al layer. Although grains in these regions are all equiaxed, in Fig. 4-5, a decreasing trend in grain size with increasing distance from the interface in the through-thickness direction is still observed.

In order to compare the experimental results between bonded and weakly bonded specimens, the grain structure of a weakly bonded Al–Al₂O₃ specimen was studied.
Figure 4-5: The distribution of grain size within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm.

Figures 4-7(a) and 4-7(b), taken on the planes whose normals are parallel to the RD and TD, respectively, show the result of grain shape evolution after bonding. It can be seen that all grains within the Al layer are equiaxed even in the regions next to the interface. Figure 4-8 presents the result of the grain size distribution in the Al layer for a weakly bonded Al–Al₂O₃ specimen. It can be observed that grains have roughly the same size within the entire Al layer.

4.1.2 Grain structure in an Al–Al₂O₃ bilayer composite with an Al thickness of 600 µm

Figure 4-9 shows the grain shape distribution within the Al layer for a 600 µm thick Al bonded to a 1 mm thick Al₂O₃. This photo was taken on the plane whose normal is parallel to the TD. Since grain shape distributions are similar on cross-sectional planes (as can be seen in Figs. 4-1(a) and 4-1(b)), in this case, only one figure is presented. It is seen that in Fig. 4-9, grains in the regions near the interface are of
Figure 4-6: The distribution of grain size around the central regions within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm.

Figure 4-10 presents the result of the grain size measurement. It is seen that in the Al–Al₂O₃ bilayer specimen with an Al thickness of 600 μm, there also exists a gradient in grain size distribution within the Al layer. The trend of the gradient is similar to the one in the Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm.

4.1.3 Grain structure in an Al–Al₂O₃ bilayer composite with an Al thickness of 200μm

Since the Al layer is very thin (it became even thinner after bonding) in this case, it is not possible to investigate the gradient of grain size distribution within the Al layer. Therefore, in this section, only the result of grain shape distribution is presented (Fig. 4-11). This photo was taken on the plane whose normal is parallel to the TD. It can be seen that the regions in which grains are elongated and slanted cover most of the area in the through-thickness direction.
Figure 4-7: The distribution of grain shape in the regions near the interface within the Al layer in a weakly bonded Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm from two different perspectives. (a) on the plane whose normal is parallel to the longitudinal direction (RD) and (b) on the plane whose normal is parallel to the transverse direction (TD).
Figure 4-8: The distribution of grain size within the Al layer in a weakly bonded Al

Figure 4-9: The distribution of grain shape in the regions near the interface within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 600 \( \mu \)m on the plane whose normal is parallel to the transverse direction (TD).
Figure 4-10: The distribution of grain size within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 600 µm.

4.2 Results of texture measurements

All textures were examined by measuring the three incomplete pole figures, (111), (200), and (220), in the range of α from 15° to 90°. After correction of the experimental data with respect to systematic errors (see chapter 3 for details), the two-dimensional pole figures were reconstructed. Since the interpretation of pole figures is sometimes ambiguous because of the overlapping of the same poles from different orientations, the quantitative three-dimensional ODF were also calculated from the three pole figures, (111), (200), and (220). Based on the cubic crystal structure, the textures were represented in a reduced Euler angle space (0° ≤ φ₁, φ, φ₂ ≤ 90°) and plotted with the isodensity contour lines in φ₂=constant sections through the Euler space at the interval of 5°.

In this section, textures within the Al layer for Al–Al₂O₃ bilayer specimens with various Al thicknesses were investigated. In order to obtain an overall understanding of texture evolution within the entire Al layer, textures at different planar locations
Figure 4-11: The distribution of grain shape within the Al layer in an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 200μm.

(Fig. 4-12) along with various depths from the interface were measured. Figure 4-12 is a schematic in-plane representation showing the specimen preparation prior to texture examination. Each specimen with the in-plane dimension of 13 mm x 13 mm was cut into nine pieces evenly. Texture measurement was then carried out on different pieces (“r”, “c”, and “l”) for the same specimen. Also, texture investigation at different depths from the interface was achieved by removing extra Al layer using mechanical polishing. In order to avoid any texture effect resulting from polishing, the Al layer was carefully polished using mastermet (colloidal 0.05 μm SiO$_2$ suspension, pH 9.8) as the final step.

Due to space limitation, only (111) pole figures and ODF contour plots in $\varphi_2 = 0^\circ$ sections are presented in this chapter.

4.2.1 Texture measurement in an Al–Al$_2$O$_3$ bilayer composite with an Al thickness of 1 mm

Figures 4-13(a) to 4-13(c) present the three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections at three locations “r”, “c”, and “l” (Fig. 4-12), respectively, within the Al layer at a distance of 400 μm from the interface for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 1 mm. It can be seen that at the current depth, textures corresponding to the different locations are similar.
Figure 4-12: The schematic in-plane illustration of the different locations “r”, “c”, and “l” on which the texture measurement were carried out.

For textures at a distance of 300 μm from the interface, Figs. 4-14(a) to 4-14(c) show the three (111) pole figures and the ODF contour plots in $\varphi_2 = 0^\circ$ sections at three locations “r”, “c”, and “l”, respectively. It can be observed that although in Figs. 4-14(b), the texture is still the regular cube texture, in Figs. 4-14(a) and 4-14(c), textures start to rotate with respect to the RD. Moreover, since locations “r” and “l” have 90$^\circ$ angular relationship, in Figs. 4-14(a) and 4-14(c), both the pole figures and ODFs also present 90$^\circ$ rotation.

The degree of rotation in texture becomes stronger as the distance to the interface decreased, as can be seen in Figs. 4-15 and 4-16. Figures 4-15(a) to 4-15(c) and Figs. 4-16(a) to 4-16(c) present the (111) pole figures and ODFs ($\varphi_2 = 0^\circ$) at the corresponding locations “r”, “c”, and “l” at distances of 200 μm and 100 μm from the interface, respectively. It is seen that the regular cube texture is present around the center regions of the specimen, and the rotated cube texture develops at locations “r” and “l”.

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Figure 4-13: The three incomplete (111) pole figures along with the ODF contour plots in $\phi_2 = 0^\circ$ sections at three locations, (a) “r”, (b) “c”, and (c) “l”, respectively, within the Al layer at a distance of 400 $\mu$m from the interface for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 1 mm.
Figure 4-14: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections at three locations, (a) “r”, (b) “c”, and (c) “l”, respectively, within the Al layer at a distance of 300 $\mu$m from the interface for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 1 mm.
Figure 4-15: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections at three locations, (a) “r”, (b) “c”, and (c) “l”, respectively, within the Al layer at a distance of 200 $\mu$m from the interface for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 1 mm.
Figure 4-16: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections at three locations, (a) “r”, (b) “c”, and (c) “l”, respectively, within the Al layer at a distance of 100 $\mu$m from the interface for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 1 mm.
4.2.2 Texture measurement in an Al–Al₂O₃ bilayer composite with an Al thickness of 600 μm

Pole figures and ODFs within the Al layer in the Al–Al₂O₃ bilayer specimen with an Al thickness of 600 μm are presented in this section. Since it has been shown in the previous section that the rotated cube texture exists in all but the center regions of the Al layer, only texture results corresponding to locations “r” and “c” (Fig. 4-12) are presented. Figures 4-17(a) to 4-17(c) show the three (111) pole figures and the corresponding ODFs (φ₂ = 0° sections only) at location “r” (Fig. 4-12) with the depths of 300 μm, 200 μm, and 100 μm from the interface, respectively. As can be seen in these figures, a trend of texture evolution from the regular cube texture to the rotated cube texture appears as the distance to the interface decreases.

Pole figures and ODFs (φ₂ = 0° sections only) at location “c” at a distances of 300 μm, 200 μm, and 100 μm from the interface are shown in Figs. 4-18(a) to 4-18(c), respectively. It is shown that in all the figures, the regular cube texture dominates within the entire Al layer in the regions around the specimen’s center.

4.2.3 Texture measurement in an Al–Al₂O₃ bilayer composite with an Al thickness of 200μm

Since both the cube texture and R texture exist within the Al layer in the Al–Al₂O₃ bilayer specimen with an Al thickness of 200μm, in order to provide a better understanding of the current texture, the (111) pole figures and complete ODFs (φ₂ = 0° to 90°) are presented separately. Figures 4-19(a) and 4-19(b) present the (111) pole figures at location “r” (Fig. 4-12) within the Al layer at distances of 150 μm and 100 μm from the interface, respectively, for an Al–Al₂O₃ bilayer specimen with an Al thickness of 200μm. It can be seen that in this specimen, rotated cube texture along with a small amount of retained deformation texture dominate the entire Al layer. The complete ODFs corresponding to the two different depths are shown in Figs. 4-20(a) and 4-20(b), respectively. It shows that in these two figures, besides
Figure 4-17: The three (111) pole figures and the corresponding ODFs ($\varphi_2 = 0^\circ$ sections only) at location “r” with the depths of (a) 300 $\mu$m, (b) 200 $\mu$m, and (c) 100 $\mu$m to the interface, respectively, for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 600 $\mu$m.
Figure 4-18: The three (111) pole figures and the corresponding ODFs ($\varphi_2 = 0^\circ$ sections only) at location “c” with the depths of (a) 300 $\mu$m, (b) 200 $\mu$m, and (c) 100 $\mu$m to the interface, respectively, for an Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 600 $\mu$m.
the rotated cube texture, a retained deformation texture that was characterized as R texture also exists.

The (111) pole figures at location “c” with the depths of 150 µm and 100 µm from the interface are shown in Figs. 4-21(a) and 4-21(b), respectively, and the corresponding complete ODFs are shown in Figs. 4-22(a) and 4-22(b). All the figures show that around the center regions of the specimen, the regular cube texture and R texture coexist in the current case.

4.3 Results of microhardness tests

Mechanical properties within the Al layer were characterized by microhardness measurements (see chapter 2 for the test method). Since the 200 µm Al layer was too thin to have a valid zone for indentation due to the overlapping of large plastic zones created after each indentation, tests were carried out on Al–Al₂O₃ bilayer specimens with Al thicknesses of 1 mm and 600 µm only.
Figure 4-20: The complete orientation distribution function plots corresponding to the two different depths of (a) 150 µm and (b) 100 µm at location “r” within the Al layer for an Al–Al₂O₃ bilayer specimen with an Al thickness of 200µm.

Figure 4-21: The (111) pole figures at location “c” within the Al layer at distances of (a) 150 µm and (b) 100 µm from the interface, respectively, for an Al–Al₂O₃ bilayer specimen with an Al thickness of 200µm.
Figure 4-22: The complete orientation distribution function plots corresponding to the two different depths of (a) 150 μm and (b) 100 μm at location “c” within the Al layer for an Al–Al₂O₃ bilayer specimen with an Al thickness of 200μm.

4.3.1 Results of the microhardness tests of an Al–Al₂O₃ bilayer composite with an Al thickness of 1 mm

Figure 4-23 presents the Vickers hardness result of the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm. The indentation tests were performed in the through-thickness direction. In this case, hardness tests performed on planes with different depths from the interface were accomplished by removing extra Al layer using mechanical polishing. In order to avoid any residual stress effect resulting from polishing, the Al layer was carefully polished using mastermat (0.05 μm SiO₂ suspension) as the final step. In Fig. 4-23, there is an increasing trend in hardness with increasing distance from the interface. It is seen that the hardness value is about 28 in the regions near the interface and increased to about 31.5 in the regions near the surface.

In order to investigate texture effects on the observed trend in hardness gradient, two additional experiments were carried out (Figs. 4-24(a) and 4-24(b)). Figure 4-24(a)
Figure 4-23: The hardness result of the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm. The indentation tests were performed in the through-thickness direction.

presents the indentation result that was obtained on the cross-sectional plane whose normal is perpendicular to the RD. Figure 4-24(b) shows the results of the test that was performed on the cross-sectional plane whose normal is 60° from the TD. Both figures show similar trends in hardness gradient, as also shown in Fig. 4-23.

Figure 4-25 shows the hardness as a function of the distance in the longitudinal direction. This test was performed on the cross-sectional plane whose normal is perpendicular to the RD at different depths from the interface. To eliminate the edge effect, indentations were not performed in the vicinity of the edges (≈ 2 mm). It can be seen that at the same distance from the interface, there is no hardness gradient across the specimen in the longitudinal direction. However, there is an increase in hardness with increasing distance normal to the interface.
Figure 4-24: The hardness result within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm. The indentation tests were carried out on the cross-sectional plane whose normal is (a) perpendicular to the RD and (b) perpendicular to the RD.
Figure 4-25: The hardness result as a function of the distance in the longitudinal direction within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm. The indentation tests were carried out on the cross-sectional plane whose normal is perpendicular to the RD.

4.3.2 Results of the microhardness test of an Al–Al₂O₃ bilayer composite with an Al thickness of 600 μm

Since indentation tests performed on planes with different perspectives all gave similar results in the previous section, only the result of the test that was performed in the through-thickness direction is presented here. Figure 4-26 shows the result of indentation test of the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 600 μm. It is seen that a strong gradient of hardness exists within the Al layer. In the regions near the interface, the hardness is about 28.5, and the value is about 31 in the regions away from the interface.

4.4 Discussion

In the current study, it is shown that for all the diffusion bonded Al–Al₂O₃ bilayer composites, the elongated and rotated grains exist in the regions near the interface. This may be explained as follows. When intergrain bonds formed between the Al and Al₂O₃ layers during diffusion bonding, the Al grains next to the interface can be
Figure 4-26: The hardness result within the Al layer in an Al–Al₂O₃ bilayer specimen with an Al thickness of 600 μm. The indentation tests were carried out on the plane whose normal is perpendicular to the RD.

treated as being pinned by the Al₂O₃ grains that are next to the interface due to the different longitudinal displacements within each layer. Therefore, when the Al layer crept during bonding, the interface limited the mobility of the Al grains near it and resulted in the longitudinal displacement gradient within the Al layer in the through-thickness direction. This displacement gradient in the through-thickness direction caused any grain close to the interface to experience increased levels of longitudinal displacement at different locations within the grain itself as compared to the grain located farther away from the interface and therefore, resulted in the grain rotations.

This argument can also be justified by looking at the unbonded case (Figs. 4-7(a) and 4-7(b)). In the unbonded case, since the interface imposed no constraints on the motion of the neighboring Al grains, no longitudinal displacement gradient was formed. Therefore, grains in the regions near the interface are similar to grains near the surface. By looking at Figs. 4-1(a), 4-1(b), and 4-2, the three-dimensional grain shape and distribution may be reconstructed. These results suggest that grains near the interface are cylinder-like and symmetrically distributed around specimen’s center.
Although the elongated and slanted grains are within the regions that are within a distance of 200 μm from the interface, a transition zone for grain size and hardness extends far beyond that region. It can be seen that in Figs. 4-5, 4-23, 4-24, 4-10, and 4-26, the transition zone extends to regions that are 500 μm away from the interface. To investigate texture effects on the anisotropic response in mechanical properties, two additional hardness tests were performed. Since materials with the cube texture give anisotropic mechanical response in directions between the RD and 45° from the RD, indentations on the cross-sectional planes with planes’ normal 60° and 0° from the TD were carried out (Figs. 4-24(a) and 4-24(b), respectively). Both of them show a similar trend in the variation of hardness as in Fig. 4-2. The variations of grain size and hardness are about 20% and 10%, respectively, between the regions close to and away from the interface. In the Al–Al₂O₃ bilayer specimen with an Al thickness of 1 mm (Fig. 4-5), grains near the interface are about 33 μm, and they are about 27 μm in the regions near the surface. Also, in Figs. 4-23 and 4-24, hardness Hᵥ in the regions near the interface is about 28, and it is about 31 in the regions close to the surface. Similar variations in grain size and hardness can also be observed in the Al–Al₂O₃ bilayer specimen with an Al thickness of 600 μm. Since the texture in the central regions of the specimen was characterized as the regular cube texture, the results presented in Figs. 4-6 and 4-25 preclude the possibility of texture induced hardness gradient in the through-thickness direction within the Al layer. Therefore, the relationship between grain size and hardness variations may be correlated with the Hall–Petch equation (Eq. 4.1).

\[
\sigma_y = \sigma_0 + k d_y^{-1/2},
\]  

(4.1)

In general,

\[
\sigma_y \propto (\text{hardness index, } Hᵥ) \propto (\text{grain size, } d_y)^{-1/2}
\]

Figure 4-27 shows the hardness as a function of the inverse square root of the grain size. The trend line in this figure represents the Hall–Petch equation. It was assumed that the hardness value varies linearly with the value of the inverse square root of the
Figure 4-27: The hardness result within the Al layer in an Al–Al₂O₃ bilayer specimen as a function of the inverse square root of the grain size (Hall–Petch relation is assumed).

As can be seen in this figure, the measurements of the hardness and grain size are in reasonable agreement.

Two suggestions are provided to explain the existence of bigger grains near the interface. Firstly, due to the gradient of longitudinal displacement in the through-thickness direction within the Al layer, the localization of shear stress exists in the regions near the interface. Since grains may tend to grow faster in certain directions when stress is concentrated in certain directions, in order to eliminate such a stress concentration, grains in these regions may grow faster. Another explanation may be made based on the energy point of view. The interface may have a strong effect on the surface energy of grains near it, and this effect may favor grains near the interface to grow faster.

The effects of interface constraints on the texture evolution were also investigated (Figs. 4-13 to 4-21). A change of texture from the regular cube texture to the rotated
Figure 4-28: The distribution of grain size within the Al layer in an Al–Al2O3 bilayer specimen with an Al thickness of 1 mm. The measurement was performed on the plane whose normal is parallel to the ND at location, “r”, at different depths from the interface.

cube texture in the regions near the interface prevails within the Al layer except in the center regions. The observation of texture gradient is in good agreement with the results of grain shape gradient. Since grains near the interface are rotated, rotations of texture occur in the regions near the interface. This is justified by looking at the central regions of the specimen. In these regions, grains close to the interface did not rotate, and therefore, the regular cube texture does not rotate either.

It should be noted that the gradient of the microhardness is a result of the grain size effect and is not an effect of the texture gradient. In Fig. 4-25, it is seen that at the same distance from the interface, there is no hardness gradient across the specimen in the longitudinal direction. However, there is an increase in hardness with increasing distance normal to the interface. It can also be verified by looking at Fig. 4-28. This figure shows the result of the grain size measurement that was carried out on the plane whose normal is parallel to the ND at location, “r” (Fig. 4-12), at different depths from the interface. An increase in grain size with the decreasing distance to the interface is observed.
Moreover, in the present work, the regions affected by the interface do not depend on the thickness of the Al layer. For example, in both the Al–Al₂O₃ bilayer specimens with Al thicknesses of 1 mm and 600 μm, the rotated cube texture began to dominate within the regions that are located about 200μm from the interface. The trends of grain size and hardness gradients are also very similar.

In Köhler et al’s work [55] on monolithic F.C.C. metals, the rotated cube texture can also be found under different conditions. In their work, the rotated cube texture was found by simulating the recrystallization process in high stacking fault F.C.C. metals with different starting deformation textures. On condition that the orientation distribution of nuclei is random, and the initial deformation texture is pure B, pure Cu, or the mixture of S, B, and Cu, the recrystallization texture that corresponds to the fastest growing “compromise” orientations (i.e., 40°<111> local growth law) will be the rotated cube texture. In the current Al–Al₂O₃ bilayer composites, however, the rotated cube texture purely resulted from the interface constraints.

4.5 Summary of experimental findings

Grain structure, texture, and hardness within the Al layer in Al–Al₂O₃ bilayer composites with various Al thicknesses after diffusion bonding were investigated. Although these effects are small, they are experimentally reproducible, and all were in good agreements with each other. The grain structure change was characterized by grain orientation and grain size measurements, and the mechanical properties change was characterized by microhardness measurements.

A number of conclusions were drawn from the experimental results obtained in the current study.

- Experimental observations show for the first time in this study that the grain structure, texture, and microhardness within the Al layer in Al–Al₂O₃ bilayer
composites are different from these within a monolithic Al layer after having undergone the similar heat treatment.

- Due to the presence of the Al–Al$_2$O$_3$ interface, gradients in grain structure, texture, and hardness were observed to be distributed symmetrically about the specimen’s center within the Al layer.

- The regions affected by the interface do not depend on the thickness of the Al layer.

- In all bonded Al–Al$_2$O$_3$ bilayer composites, within the Al layer, grains near the interface were elongated and slanted. However, grains close to the surface were equiaxed, which is a trend normally observed in recrystallized F.C.C. metals.

- The long, slanted grains near the interface appear to have a cylindrical three-dimensional shape.

- In both Al–Al$_2$O$_3$ bilayer composites with Al thicknesses of 1 mm and 600 μm, trends of decreasing grain size with increasing distance from the interface were observed. Grains near the interface were about 20% larger than grains near the surface.

- The observation that grains near the interface were larger may be attributed to the anisotropic grain growth behavior (i.e., grains grow faster in certain directions in certain planes.)

- In all bonded Al–Al$_2$O$_3$ bilayer composites, the rotated cube texture is present in the regions near the interface within the Al layer, and regular cube texture is present in the regions near the surface.

- In both Al–Al$_2$O$_3$ bilayer composites with Al thicknesses of 1 mm and 600 μm, trends of increasing hardness with increasing distance from the interface were observed. The variation in hardness between the regions near the interface and the surface was about 10%.
- The results of microhardness tests and grain size measurements may be correlated with the Hall–Petch relationship.
Chapter 5

Experimental results for Al$_2$O$_3$–Al–Al$_2$O$_3$ trilayer composites

In this chapter, experimental results of the evolution of grain structure, texture and microhardness within the Al layer with two different thicknesses (1 mm and 200 μm) in Al$_2$O$_3$–Al–Al$_2$O$_3$ trilayer composites after diffusion bonding are presented. The aluminum was bonded to two layers of aluminum oxide that have the same thickness of 1 mm. Due to the presence of the Al–Al$_2$O$_3$ interface, all cases show gradients within the Al layer in the through-thickness directions (ND). Moreover, since the Al layer was symmetrically sandwiched by two layers of Al$_2$O$_3$ with the same thickness, all gradients within the Al layer were symmetrically distributed with respect to the middle normal plane of the specimens. The grain structure change, in the current study, was also characterized by measurements of grain shape change and grain size. The mechanical properties change was also characterized by microhardness measurements, based on the direct relationship between the yield strength and hardness index. All the specimens had an in-plane dimension of 13 mm x 13 mm along with various thicknesses. Discussions in this chapter are omitted due to the similarity of microstructural evolution between the bilayer and trilayer cases (see the appropriate sections of the previous chapter for details).
5.1 Results of grain structure examination

Investigations of grain structure (shape and size) within the Al layer after diffusion bonding was carried out using the methods described in chapter 2. Results for Al₂O₃–Al–Al₂O₃ trilayer composites with two different Al thicknesses, 1 mm and 200 μm are presented in this section.

5.1.1 Grain structure in Al₂O₃–1 mm Al–Al₂O₃ trilayer composite

Since it has been shown in chapter 4 that grain shape was symmetrically distributed with respect to specimen’s center regions, in the current case, only the micrograph that was taken on the plane whose normal is perpendicular to the longitudinal direction (RD) is presented (Fig. 5-1). It can be seen that grains in the regions near the two interfaces are elongated and slanted, and they are equiaxed in the central regions of the Al layer. The sizes of the regions where grains are long and slanted
are approximately the same on the two sides and are comparable with the size in the Al–Al₂O₃ bilayer specimens.

Figure 5-2 shows the distribution of grain size within the Al layer. The final thickness for the 1 mm Al layer was about 850 μm because of the creeping effect at high temperatures during bonding. In Fig. 5-2, symmetric decreasing trends in grain size are observed with increasing distance from the two interfaces towards the center of the Al layer in the through-thickness direction. It is noteworthy that in the current case, the variation of grain size between the central regions (≈ 26 μm) and the near interface regions (≈ 31 μm) is about 20%, which is similar to the grain size variation in the Al–Al₂O₃ bilayer cases.
5.1.2 Grain structure in $\text{Al}_2\text{O}_3-200\ \mu\text{m Al-}\text{Al}_2\text{O}_3$ trilayer composite

Since it is not possible to investigate the gradient of grain size distribution within the Al layer due to the very thin layer of Al that only comprises about six layers of grains in the through–thickness direction, in this section, only the result of grain shape distribution is presented (Fig. 5-3). This micrograph was taken on the plane whose normal is perpendicular to the RD. It can be seen that in the $\text{Al}_2\text{O}_3-200\ \mu\text{m Al-}\text{Al}_2\text{O}_3$ trilayer specimen, the regions with the elongated and slanted grains extended from the two interfaces and meet at the center. In this case, there are no equiaxed grains within the Al layer in the through–thickness direction. This observation is in good agreement with the results in the Al–Al$_2$O$_3$ bilayer specimen with an Al thickness of 200$\mu$m (Fig. 4-11). In the bilayer case, the elongated and slanted grains covered about two-thirds of the regions within the Al layer in the through–thickness direction.
5.2 Results of texture measurements

In Al₂O₃–Al–Al₂O₃ trilayer specimens, the Al layer was bonded to two layers of Al₂O₃ with the same thickness. In order to perform texture measurements on the Al layer, one layer of the Al₂O₃ was removed after diffusion bonding. A diamond wheel with particle size of 120 μm was first used to grind off one Al₂O₃ layer in the trilayer specimen. In order to avoid any texture effect resulting from polishing, the Al layer was carefully polished and mastermet (colloidal 0.05 μm SiO₂ suspension, pH 9.8) was used as the final step.

All textures were examined by measuring the three incomplete pole figures, (111), (200), and (220), in the range of α from 15° to 90°. After correction of the experimental data with respect to systematic errors (see chapter 3 for details), the two-dimensional pole figures were reconstructed. Since the overlapping of the same poles from different orientations sometimes causes ambiguity in the interpretation of pole figures, the quantitative three-dimensional ODF were also calculated from the three pole figures, (111), (200), and (220). Based on the cubic crystal structure, the textures were represented in a reduced Euler angle space (0° ≤ ϕ₁, ϕ, ϕ₂ ≤ 90°) and plotted with the isodensity contour lines in ϕ₂ = constant sections through the Euler space at the interval of 5°.

In this section, only texture within the Al layer for Al₂O₃–1 mm Al–Al₂O₃ trilayer specimen was investigated. As described in chapter 4, in order to obtain an overall understanding of texture evolution within the entire Al layer, textures at two different planar locations, “r” and “c” (Fig. 4-12), along with various depths from the interface were measured.

Only (111) pole figures and ODF contour plots in ϕ₂ = 0° sections are presented here due to the space limitation. Figures 5-4(a) to 5-4(c) present the three (111) pole figures along with the ODF contour plots in ϕ₂ = 0° sections within the Al layer at location, “r”, (Fig. 4-12) at distances of 100 μm, 200 μm, and 300 μm from interface, “1”, respectively, in an Al₂O₃–1 mm Al–Al₂O₃ trilayer specimen. It can be seen
that although the regions that are 300 μm away from interface, “1”, have the regular cube texture, the regions that are within the distance of 200 μm from the interface have the rotated cube texture. The degree of rotation in texture becomes stronger as the distance to the interface decreases. The results are in good agreement with the observations in the Al–Al₂O₃ bilayer cases.

The rotated cube texture is also observed in the regions near interface, “2”, (Figs. 5-5(a) to 5-5(c)). Figures 5-5(a) to 5-5(c) present the texture results at the same location, “r”, with the corresponding distances of 100 μm, 200 μm, and 300 μm from interface, “2”. In the trilayer Al₂O₃–1 mm Al–Al₂O₃ specimen, it is observed that the texture gradients are symmetrically distributed with respect to the middle normal plane. This result is seen to compare favorably with the grain size measurements.

The texture results at location, “c”, with the three different depths, 700 μm, 400 μm, and 100 μm from interface, “1”, are presented in Figs. 5-6(a) to 5-6(c), respectively. It can be seen that the regular cube texture dominates the entire central regions of the Al layer.

5.3 Results of microhardness tests

In the Al₂O₃–1 mm Al–Al₂O₃ trilayer case, mechanical properties within the Al layer were characterized by microhardness measurements (see chapter 2 for methodology details). Since the 200 μm Al layer was too thin to have a valid zone for indentation due to the overlapping of large plastic zones created after each indentation, tests were carried out on the Al₂O₃–1 mm Al–Al₂O₃ trilayer specimen only.

Figure 5-7 presents the hardness result of the Al layer in a Al₂O₃–1 mm Al–Al₂O₃ trilayer specimen. The indentation tests were carried out on the plane whose normal is perpendicular to the TD. In this figure, there are increasing trends in hardness with increasing distance from the interface towards the central regions of the Al layer in the through–thickness direction. It is seen that the hardness value is about 27 in
Figure 5-4: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections within the Al layer at location, "r", at distances of (a) 100 $\mu$m, (b) 200 $\mu$m, and (c) 300 $\mu$m from interface, "1", respectively, in a Al$_2$O$_3$–1 mm Al–Al$_2$O$_3$ trilayer specimen.
Figure 5-5: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections within the Al layer at location, “r”, at distances of (a) 100 $\mu$m, (b) 200 $\mu$m, and (c) 300 $\mu$m from interface, “2”, respectively, in a $\text{Al}_2\text{O}_3$–1 mm Al–$\text{Al}_2\text{O}_3$ trilayer specimen.
Figure 5-6: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections within the Al layer at location, “c”, at distances of (a) 700 $\mu$m, (b) 400 $\mu$m, and (c) 100 $\mu$m from interface, “2”, respectively, in a Al$_2$O$_3$–1 mm Al–Al$_2$O$_3$ trilayer specimen.
Figure 5-7: The hardness result within the Al layer in an Al$_2$O$_3$–1 mm Al–Al$_2$O$_3$ trilayer specimen. The indentation tests were carried out on the cross-sectional plane whose normal is perpendicular to the RD.

The hardness values decreased to around 27 in the regions near the interfaces and increased to about 29 in the regions around the center. The maximum variation, in this case, is about 8% and is comparable with the result in the bilayer case in which the variation is about 10%.

5.4 Conclusions

Grain structure, texture, and microhardness within the Al layer in Al$_2$O$_3$–Al–Al$_2$O$_3$ trilayer composites with two Al thicknesses, 1 mm and 200 µm, after diffusion bonding were investigated. All were in good agreements with the results obtained in Al–Al$_2$O$_3$ bilayer cases.

A number of conclusions were drawn from the experimental results obtained in the current study.

- Experimental observations show that the gradients of grain structure, texture,
and mechanical properties within the Al layer in Al₂O₃–Al–Al₂O₃ trilayer composites are symmetrically distributed with respect to the middle normal plane.

- Symmetric trends of decreasing grain size with increasing distance from the two interfaces were observed in the Al₂O₃–1 mm Al–Al₂O₃ trilayer specimen. Grains near the interfaces were about 20% larger than grains near the central normal plane of the Al layer.

- Symmetric trends of texture gradient within the Al layer at location "r" were observed. The texture changes from regular cube texture to the rotated cube texture as the distances to the interfaces decreases.

- In the Al₂O₃–1 mm Al–Al₂O₃ trilayer composite, symmetric trends of increasing hardness with increasing distance from the two interfaces were observed. The variation between the regions near the interfaces and the middle normal plane of the Al layer is about 8%.

- Results obtained in the Al₂O₃–Al–Al₂O₃ trilayer cases compared favorably with the results obtained in the Al–Al₂O₃ bilayer cases.
Chapter 6

Finite element analysis

In this chapter, detailed computational analyses were performed using a rate–dependent crystal plasticity model along with finite element simulations to develop a mechanistic rationale for the experimental observations.

6.1 A brief review of crystal plasticity constitutive models

In the simulation of crystal deformation, polycrystals are assumed to be deformed by the mechanism of crystallographic slip. One of the difficulties in formulating these crystal plasticity models is to capture the interactions between grains accurately. In these models, specific solutions depend on the shapes and orientations of the grains, and both the shapes and orientations change with strain. Although compatibility and equilibrium between grains are both important fundamental issues to be deliberated in polycrystalline modeling, since the stress and strain are coupled through the material constitutive relation and depend on the specific neighboring grain orientations, they are not independent constraints. Therefore, without detailed formulations, it is not possible to satisfy both compatibility and equilibrium strictly. In this section, a number of crystal plasticity models, which formulate the interactions between grains differently and impose different degrees of requirements on compatibility and
equilibrium between grains, are reviewed briefly to provide a general background understanding among different models.

The Sachs model [56], which is one of the earliest polycrystal models, was developed based on the assumption that equilibrium is satisfied by having the same stress state in each grain with compatibility ignored. Kochendorfer [57] then refined the model by assuming that each grain was subjected to the same stretch. This model has two major drawbacks [58]. Firstly, stress equilibrium could not be established across grain boundaries if each grain is subjected to only simple tension of different amounts. Secondly, compatibility among the grains cannot be maintained when each grain is assumed to deform by single slip.

In Taylor type models [58,59–61], on the other hand, in order to overcome the objections in the Sachs model, compatibility was strictly enforced by imposing the same amount of strain on each grain. It then results in the ignorance of the intergrain equilibrium. This model has been extensively used in analyzing texture in both F.C.C. and B.C.C. metals due to the successful predictions of texture and polycrystal stress-strain response. However, as noted by Taylor [59–62], since the model was developed based on rate independent crystallographic slip, it lacks the uniqueness in determining the active slip systems and the predicted lattice rotations. Therefore, although it is possible for this model to predict texture qualitatively, detailed predictions that unambiguously relate the texture development to material properties such as strain hardening and strain/stress histories are not possible. The nonuniqueness causes more serious problems, when the crystalline slip constitutive theory is applied to the solution of boundary value problems for crystals, because of the nonunique constitutive relation between stress and strain rates that results from the nonunique determination of active slip system [58].

Relaxed constraint models [63–65], which modified Taylor models by selectively satisfying compatibility and ignoring some intergrain equilibrium components, assume that when the grain aspect ratio is large, it is possible to partially relax the strict compatibility requirements, and the strain resulting from compatibility constraints is
accommodated by the nonuniform deformation at the grain boundaries. That is, the grain interior can deform differently than the overall deformation without violating compatibility. These models, however, do not resolve the nonuniqueness problem in determining active slip systems, and neither compatibility nor equilibrium are satisfied exactly.

Self consistent models [66–71] for polycrystal modeling (not Taylor-type based), attempt to satisfy both the compatibility and equilibrium in an average sense and some of them are rate-independent. The average is taken over the entire population of grains by assuming that each grain is embedded in an infinite homogeneous matrix, and therefore, its modulus is the average of all grains. Since these models are still based on the rate–independent formulations, the nonuniqueness problem is yet to be resolved.

Among all the models described above, the two inevitable problems are the nonuniqueness in determining active slip systems and satisfying both compatibility and equilibrium exactly. The nonuniqueness problem can be solved, however, if a rate–dependent model is used. In a rate–dependent model, the slip rate in each slip system always depends on the current stress and hardness properties, and therefore, there is no need to determine active/inactive slip systems. The second problem can also be solved if a numerical method such as finite element method (FEM) is adopted. In FEM, the compatibility and equilibrium are satisfied simultaneously by numerical computation. Based on those considerations, a rate–dependent crystal plasticity model developed by Peirce et al [72] and modified by Becker [73] was used in the current work.

6.2 Finite element model

The rate–dependent crystal plasticity model used in the current study was first developed by Peirce et al [72] and used by Asaro and Needleman [58]. It was then modified by Becker (see [73] for model details) and employed here. The kinematic description was that given by Asaro [74] and Asaro and Rice [75]. In a rate–dependent
formulation, there is no need for determining the active or inactive slip systems. All slip systems always slip at a rate which depends on the current stress and hardness properties. Since the slipping rates are related to the current stress and material states directly and uniquely, the nonuniqueness for determining the slipping rate on each slip system in rate-independent models can therefore be avoided.

Although the current model is similar to that of Peirce et al’s in the kinematics and numerical integration scheme, there are still two major differences. The first difference is the crystal structure. In the current model, the plastic deformation is assumed to result from slip along twelve \(\{111\}<110>\) slip systems that are typical of the F.C.C. materials. In the Peirce et al’ model, on the other hand, there are only two slip systems assumed to exist. The second difference is the slip system constitutive model. In Peirce et al’s model, they used a hyperbolic cosine relation for strain hardening and a power law equation for strain rate sensitivity. However, since the strain hardening is almost zero except at very small strains for high temperature deformation, it is assumed that there is no strain hardening in the current model. Moreover, rather than using a power law equation to describe the strain rate sensitivity, a hyperbolic sine relation is used in the current study.

The model accounts for finite strains, rotations of the crystal lattice with deformation and for the cubic symmetry of the elastic constants. It also assumes that plastic flow occurs by slip through the crystal lattice, leaving the lattice undisturbed. The grain boundaries are modeled as perfectly bonded, and compatibility and equilibrium are satisfied across the grain boundaries by the finite element method. The crystal constitutive relations are implemented as a user subroutine in the finite element code ABAQUS [76,77] with the integration of the constitutive relations following the rate tangent modulus method of Peirce et al [72].

In the crystal plasticity model, the velocity gradient is decomposed into the elastic part, \(L^e\), and the plastic part due to slip on prescribed twelve slip systems (Eq. 6.1) [79].
\[
\frac{\partial \dot{x}}{\partial x} = L^e + \sum_{i=1}^{12} \dot{\gamma}^i s^i m^i .
\] 

(6.1)

In this equation, \( x \) denotes the current position of the material point, and \( \dot{x} \) is the velocity. The superscript \( i \) indicates one of the twelve slip systems for F.C.C. materials. The current slip direction on the slip system \( i \) is denoted by \( s^i \). \( m^i \) is a vector normal to the slip plane \( i \). \( \dot{\gamma}^i \) is the slip rate. The slip vectors are updated by \( \dot{S}^i = L^e S^i \) and \( \dot{m}^i = -m^i L^e \) to account for stretching and rotation of the crystal lattice. The cubic constants used in the current study are \( C_{11} = 108 \) GPa, \( C_{12} = 62 \) GPa, and \( C_{44} = 28.3 \) GPa, which are for the commercially pure aluminum at room temperatures.

The slip rate, \( \dot{\gamma}^i \), in the rate dependent formulation [58,72] is expressed as a function of the current resolved shear stress, \( \tau^i \), on the slip system \( i \) and the slip system hardness, \( q(\gamma) \), which is assumed to be identical on all the slip systems at a material point (i.e., self hardening equals to latent hardening) [80].

\[
\dot{\gamma}^i = F(\tau^i, q(\gamma)) .
\] 

(6.2)

The resolved shear stress, \( \tau^i \), on the slip system is given as a function of the Kirchhoff stress, \( \tau \), and the slip system geometry. The difference between the Cauchy stress, \( \sigma \), and the Kirchhoff stress, \( \tau \), is the elastic volume change that is neglected in the current study as a result of the small difference.

\[
\tau^i = m^i \tau s^i .
\] 

(6.3)

Although this model has been applied in a number of studies for single and polycrystals [73,78–80], it is the first time that the current model is applied on materials comprise metal–ceramic bilayers. In the current study, this model was used to simu-
Figure 6-1: The finite element mesh used in the current simulation. Grains in the regions near the top of the mesh were characterized by the rate–dependent polycrystal plasticity constitutive model, and grains in the remainder of the model regions were simulated as an isotropic material.

late the deformation of grains in the regions near the Al–Al₂O₃ interface.

6.3 Model description

The current grain geometry and crystallographic orientations were obtained from another commercial aluminum alloy (6111–T4) used in a different study [81]. This material also had roughly equiaxed grains and a very strong cube texture. The orientation of individual grains was measured from back–scattered electron patterns on an scanning electron microscope (SEM), and 77 grains were measured. The finite element mesh is shown in Fig. 6-1. The model comprises 4258 elements, and all of them are 4–noded plane strain elements. Equilibrium was satisfied by newton iterations in the commerical finite element code ABAQUS. The residual forces were less than 0.1% of the average force. The time step is limited such that the maximum accumulated slip increment was 0.01 in a time increment. This is small enough to
Figure 6-2: The initial structure of grains in the regions near the interface. The axes “1” and “2” correspond to the specimen’s RD and ND, respectively.

have an accurate integration of the constitutive model. The solution of the element convergence is mesh dependent. Although crystal models tend to produce strain localization that cannot be accurately resolved even the finer mesh is adapted, the current simulation still provides a reasonable prediction of the overall deformation. In Fig. 6-1, the symbol, “+”, which is located near the top central region of the figure, is a sign that indicates the location of the node that controls the motion of the rigid surface.

The slip rate equation (Eq. 6.2) for the colored grains in the crystal model is expressed explicitly as

$$\dot{\gamma} = 490 \sinh \left( \frac{\tau}{10.34} \right)^4.75,$$

where the value, 490, includes the temperature and other constants. The temperature in the simulation is kept at 590°C. The function, $q(\gamma)$, which is similar to a reference flow strength, in Eq. (6.2) is a constant with the value of 10.34 in the current study.

Figure 6-2 shows the initial structure of grains in the regions near the interface. The axes “1” and “2” correspond to the specimen’s RD and ND, respectively. It can be seen that most grains are equiaxed before subjected to any compression. The
colored grains in the regions near the top of the mesh (i.e., near the interface) were characterized by the rate-dependent polycrystal plasticity constitutive model, and the grains in the remainder of the model regions (i.e., the black regions) were simulated as an isotropic material that was characterized by the von Mises flow potential and the normality flow rule. Since only the behavior of the grains near the interface is of great interest, a model that comprises four layers of grains should be sufficient to illustrate the trends qualitatively.

The in-plane dimensions of the current model are 750 μm long and 120 μm thick. The model’s aspect ratio (length/height ≈ 6.3) does not match the real aspect ratio (≈ 13) exactly, and the size of the model differs from the actual dimensions of the experimental specimens (13 mm x 13 mm x 1 mm, 600 μm, 200 μm) significantly. However, the model involves no size scale other than the size of the colored grains, the physical size of the model should not have a substantial effect on the deformation patterns. Due to the two-dimensional plane strain constraint, there is no interaction with grains in the third direction, and any strains involving the third direction are excluded.

The constitutive model proposed by Sample and Lalli [81] for commercially pure aluminum during hot working was adapted in the current study to describe the behavior of the isotropic Al layer. In this model both creep and plasticity over a wide range of temperatures and strain rates are captured by the same relation. This model has been validated for aluminum at strain rates from 10^{-5} sec^{-1} to 10^{1} sec^{-1} at temperatures above 300°C. At temperatures above 450°C, any strain hardening occurs at very small strains and the material response can be characterized adequately by a steady flow response. This steady state response is governed by the equation:

\[ \dot{\varepsilon} = \exp \left[ - \left( \frac{177244}{RT} - 29.777 \right) \right] \sinh \left( \frac{\sigma}{31} \right)^{4.75}, \]

where \( \sigma \) is the stress in MPa, \( R \) is the gas constant, \( T \) is the absolute temperature, and \( \dot{\varepsilon} \) is the strain rate in sec^{-1} (see appendix II for details). In the current study,
Figure 6-3: The grain structure within the Al layer after a 10% thickness reduction.

Eq. (6.5) for the continuum model is expressed as

$$\dot{\epsilon} = 163.3 \sinh \left( \frac{\sigma}{31} \right)^{4.75},$$

(6.6)

where the value, 163.3, includes the temperature and other constants. The value, 31, is a constant that is similar to a reference flow strength, and a constant temperature of 590°C is used in the simulation.

The relationship between the resolved shear stress and slip in the crystal model (Eq.(6.4)) was constructed by converting the von Mises stress, $\sigma$, and effective strain rate, $\dot{\epsilon}$, of Eq. (6.5) into resolved shear stress, $\tau$, and the accumulated slip rate, $\dot{\gamma}$, respectively, using an appropriate Taylor factor, $M = 3$. That is,

$$M \tau = \sigma,$$

(6.7)

and

$$\frac{\dot{\gamma}}{M} = \dot{\epsilon}.$$  

(6.8)

The flow strengths of all 12 slip systems are equal at any integration point, the Taylor hardening assumption. With this characterization, the macroscopic response of the crystal region is similar to that of the isotropic continuum, which is thought to be adequate for the current simulations. In reality, however, the glide, climb and
Figure 6-4: The distributions of (a) logarithmic shear strain, (b) shear stress, and (c) displacement in the “1” direction (longitudinal displacement) within the Al layer after a 10% thickness reduction.
dislocation interactions are significantly more complex.

The Al$_2$O$_3$ was modeled as rigid. The interface bonding was simulated by a friction model which allowed the interface to slip at a specified shear stress. The options for the frictional interface behavior are limited in ABAQUS. The friction can be characterized either by Coulomb friction or Coulomb friction with a limit on the maximum shear stress at the interface. This latter option is more realistic since the friction cannot exceed the shear flow strength of the material. In the current work, the friction limit was set on the shear stress to either 1.5 MPa or 0 MPa to allow different degrees of sliding between the metal and the ceramic. The simulations were isothermal at the 590° bonding temperature, and therefore, the thermal expansion differences across the interface were not considered.

The left edge (“1” = 0) of the model is assumed to be a plane of symmetry. This boundary was constrained from motion in the longitudinal direction (i.e., $u_1 = 0$). The bottom of the model region (“2” = 0) was constrained from motion in the ver-
tical direction (i.e., \( u_2 = 0 \)), and the shear traction on this surface was set to be zero. Loading was applied by vertical displacement of the rigid surface representing the ceramic component, and displacement control was used to simulate a number of different thickness reductions. The model calculations are included to illustrate the potential effects of shear deformation at the interface, not to simulate the process in detail. It is felt that this model is adequate for such a demonstration.

6.4 Results of simulation

Figure 6-3 shows the grain structure after a 10% thickness reduction. Unless particularly mentioned, in the current and the subsequent cases, the friction at the interface was specified such that slip would occur for shear stresses greater than 1.5 MPa. By comparing Fig. 6-3 with Fig. 6-2, it is clearly shown that in Fig. 6-3, grains near the interface became elongated and slanted. Figures 6-4(a)—6-4(c) show the distributions of logarithmic shear strain (true strain), shear stress, and displacement in the “1” direction (longitudinal displacement) within the Al layer after deformation, respectively. As can be seen in Figs. 6-4(a) and 6-4(b), a strong localization of shear strain and stress is present in the regions near the interface, and it is absent at the opposite boundary where the shear tractions are zero. Also, in Fig. 6-4(c), the displacement is not uniformly distributed in the through-thickness direction (marked “2”) except in the regions close to the symmetry plane. Figures 6-5(a) to 6-5(c) show the (111), (200), and (220) pole figures, respectively, in the current case. It is seen that the regular cube texture retains is retained even after \( \approx 10\% \) thickness reduction.

Figures 6-6 and 6-7 show the grain structure after 20% and 40% thickness reductions, respectively. In both cases, the grains are slanted and more elongated, compared with Fig. 6-3, since they have undergone larger deformation. Figures 6-8(a)—6-8(c) and 6-9(a)—6-9(c) show the distributions of logarithmic shear strain, shear stress, and longitudinal displacement, respectively, within the Al layer after 20% and 40%
Figure 6-6: The grain structure within the Al layer after a 20% thickness reduction.

Figure 6-7: The grain structure within the Al layer after a 40% thickness reduction.
Figure 6-8: The distributions of (a) logarithmic shear strain, (b) shear stress, and (c) displacement in the "1" direction (longitudinal displacement) within the Al layer after a 20% thickness reduction.
Figure 6-9: The distributions of (a) logarithmic shear strain, (b) shear stress, and (c) displacement in the “1” direction (longitudinal displacement) within the Al layer after a 40% thickness reduction.
Figure 6-10: The three pole figures (a) (111), (b) (200), and (c) (220) within the Al layer after a 20% thickness reduction.

Figure 6-11: The three pole figures (a) (111), (b) (200), and (c) (220) within the Al layer after a 40% thickness reduction.
Figure 6-12: The grain structure within the Al layer after a 40% thickness reduction without interface friction.

thickness reduction, respectively. In Figs. 6-8(a) and 6-8(b) and 6-9 (a) and 6-9(b), the shear strain and shear stress level is higher, and the localization area is bigger. However, it is still absent at the opposite boundary. In Figs. 6-8(c) and 6-9(c), it can be seen that a strong degree of nonuniformity in the distribution of longitudinal displacement is present. Figures 6-10(a) to 6-10(c) and 6-11(a) to 6-11(c)show the (111), (200), and (220) pole figures, respectively, in cases after 20% and 40% thickness reductions, respectively. It is seen that although in the case of 20% thickness reduction, the regular cube texture is present, in the case of 40% thickness reduction, a slightly upward shifting in the RD of the regular cube texture (Fig. 6-11(b)) along with a small amount of deformation texture are present.

In order to determine the effects of the shear strain localization and the interfacial friction on the evolution of grain structure, a model without the friction at the interface was simulated. This was done by compressing the Al layer against an infinitely long, frictionless surface, and the results represent the grain structure within the Al layer after a 40% thickness reduction, without the interface constraints. Figure 6-12 shows the grain structure. It can be seen that although grains near the interface are elongated due to the compressive deformation, they are not slanted. Figures 6-13(a)—6-13(c) show the distributions of logarithmic shear strain, shear stress, and longitudinal displacement within the Al layer after deformation, respectively. These
Figure 6-13: The distributions of (a) logarithmic shear strain, (b) shear stress, and (c) displacement in the "1" direction (longitudinal displacement) within the Al layer after a 40% thickness reduction without interface friction.
Figure 6-14: The three pole figures (a) (111), (b) (200), and (c) (220) within the Al layer after a 40% thickness reduction in the frictionless case.

three figures show that in the case without interfacial friction, there is no shear strain and shear stress localization (Figs. 6-13(a) and 6-13), and the longitudinal displacement is uniformly distributed in the through–thickness direction (Fig. 6-13(c)). Figures 6-14(a)—6-14(c) show the (111), (200), and (220) pole figures, respectively, in the current case. It is seen that the regular cube texture along with a small amount of deformation texture are present in the frictionless case after a 40% thickness reduction.

### 6.5 Discussion

The Al–Al$_2$O$_3$ interface was modeled with/without friction. As being mentioned in section 6.3, in the current work, the friction limit was set on the shear stress to either 1.5 MPa or 0 MPa to allow different degrees of sliding between the metal and the ceramic. This simulates different levels of bond strength between Al and Al$_2$O$_3$. 
As can be seen in Figs. 6-9 (with friction) and 6-13 (without friction), the levels of the strain and stress localization are affected by the friction limit. The size of the localization may increase in the friction limit.

Figures 6-3 and 6-7 have shown that grains are rotated due to the friction at the interface. This is explained as follows. When bonds formed between Al and Al₂O₃ grains during bonding, the Al grains next to the interface can be treated as being pinned by the Al₂O₃ grains that are next to the interface. This effect was simulated by assuming the existence of a frictional interface in the current model. Therefore, when the Al layer crept during bonding, the interface limited the mobility of the Al grains near it and resulted in the longitudinal displacement gradient (Fig. 6-4(c), 6-8(c)), and 6-9(c)) within the Al layer in the through-thickness direction. This gradient caused any grain close to the interface to experience increased levels of longitudinal displacement farther away from the interface and therefore, resulted in the grain rotations. These rotations then gave rise to the rotations of texture in the regions near the interface. However, grain and texture rotations do not exist in the regions close to the symmetry plane due to the small displacement gradients in these regions.

As can be seen in Figs. 6-4(a), 6-8(a), and 6-9(a), the shear strain was localized in the regions near the interface, which is also explained based on the existence of the longitudinal displacement gradient in the through-thickness direction. Moreover, it is noteworthy that due to the lack of strong displacement gradients in the regions close to the interface, the shear strain localizations do not exist in these regions, which is in good agreement with the experimental results. It can be seen in Fig. 4-3 that although grains are slanted in the near-interface regions that are close to the edges of the specimen, grains are equiaxed in the central near-interface regions.

In order to validate the above arguments, a model with the frictionless interface was also carried out. Under the frictionless condition, any grain within the Al layer can slide freely, and therefore, although grains were elongated due to the compressive deformation, neither the localization of shear strain and shear stress nor the displacement gradient exist (Fig. 6-12, 6-13(a)—6-13(c)). It is seen in Figs. 4-7(a) and
ref{2-1000un(b)} that when the Al and Al$_2$O$_3$ are bonded weakly, grains are equiaxed even in the regions near the interface.

Although the results of the current simulation are in good agreement with the experimental observations in grain structure evolution, there is a discrepancy between the simulation result and experimental observation in texture evolution. For example, in Fig. 4-15, the near-interface texture was experimentally characterized as the rotated cube texture, however, the regular cube texture or a slight rotation of the regular cube texture was predicted by the current simulation. Since the predicted texture was obtained by taking into account all the orientations of individual grains within the entire model, this may be a reason responsible for the current prediction. Moreover, the discrepancy may also be attributed to the failure of the current two-dimensional model in capturing the complete three-dimensional grain evolution, the lack of incorporating with the grain growth mechanism, and the aspect ratio of the specimens.

### 6.6 Conclusions

A rate–dependent crystal plasticity model was used to illustrate the effects of interface constraints on grain structure evolution after materials have undergone 10% and 40% thickness reductions. A number of conclusions can be drawn from the current numerical modeling.

- The work applies a rate–dependent crystal plasticity model on materials comprising metal–ceramic bilayers.

- The results from the computational analyses are in good qualitative agreement with the experimental observations in grain structure. However, the texture simulation is different from what was observed experimentally. It may be attributed to the failure of the current two–dimensional model in capturing the
complete three–dimensional grain evolution, the lack of incorporating with the grain growth mechanism, and the aspect ratio of the specimens.

- In the cases with frictional interface, a strong shear strain and nonuniform displacement distribution in the through–thickness direction are present in the regions close to the interface due to the interface constraints.

- In the cases without interfacial friction, shear strain and nonuniform displacement distribution in the through–thickness direction are not present within the Al layer due to the lack of the interface constraints.

- Due to the large shear strain and the compressive deformation, grains in the regions close to the interface are slanted and elongated.
Chapter 7

Discussions

In the current study, the evolution of grain structure, texture, microhardness in diffusion–bonded Al–Al₂O₃ multi–layered composites with various Al thicknesses were investigated. It was found that the majority of the grains in the rolled Al sheet recrystallized before the temperature reached 590°C. Figure 7-1 shows the grain structure in a monolithic Al specimen that was subjected to a heat treatment similar to the one for bonding Al and Al₂O₃. The Al specimen was removed from the furnace the moment the temperature reached 590°C, and no load was applied during the heat treatment. This figure shows the majority of the grains are equiaxed, an evidence of recrystallization.

The significance of the observation that the majority of the grains recrystallized before the temperature reached 590°C is that the grains near the interface rotated and changed the shape from equiaxed to elongated during the bonding process where the grain growth is taking place. This evolution of grain structure during bonding was caused by the strong localization of shear strain and shear stress (e.g., Fig. 6-4(a) and 6-4(b)) near the interface as predicted in the finite element simulation. When the Al layer crept during bonding, the interface limited the mobility of the Al grains near it and resulted in the longitudinal displacement gradient (e.g., Fig. 6-4(c) and 6-8(c)) within the Al layer in the through–thickness direction. This gradient caused any grain near the interface to have increased levels of longitudinal displacement farther away
Figure 7-1: The grain structure in a monolithic Al specimen after subjected to a heat treatment similar to the one for bonding Al and Al₂O₃. The Al specimen was removed from the furnace the moment the temperature reached 590°C, and no load was during the heat treatment.

from the interface and therefore, resulted in the grain rotations.

Figures 4-7(a) and 4-7(b) provide the evidence for the explanation. If there are no constraints from the interface, grains are expected to be equiaxed. In Figs. 4-7(a) and 4-7(b), when specimens were not bonded or only weakly bonded, the interface was concluded to have no or only a small effect on limiting the motion of the grains near it. Therefore, grains near the interface are expected to be equiaxed as shown in this figure. Also, by modeling the interface without friction (Fig.6-12), it is seen that although grains are elongated due to the severe thickness reduction (40%), they are not slanted. This can also be verified by looking at Fig. 6-13(a). When there is no friction on the interface, the localization of shear strain does not exist.

Without bonding Al to Al₂O₃, Figs. 7-2 and 7-3(a) to 7-3(c) present the grain structure and three incomplete (111) pole figures, respectively, in a monolithic Al specimen that was subjected to a heat treatment similar to the one for bonding Al–Al₂O₃. It
Figure 7-2: The grain structure in a monolithic Al specimen after subjected to a heat treatment similar to the one for bonding Al and Al$_2$O$_3$.

can be seen that in Fig. 7-2, without the interface constraint, grains are all equiaxed. The (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ at the two surfaces and the middle of the Al layer are shown in Figs. 7-3 (a) and (c) and (b), respectively. As can be seen in these figures, the regular cube texture is present throughout the entire Al layer.

On the other hand, it can be seen that a texture gradient exists within the Al layer in Al–Al$_2$O$_3$ multi-layered specimens (e.g., Fig. 4-17). One possible to cause the gradient is the existence of a deformation texture gradient within the Al layer prior to diffusion bonding. However, since the raw Al sheet was carefully flipped over after each rolling pass during cold rolling, this is not likely. Another explanation can be made based on the mechanistic point of view. As a result of the limited mobility of grains near the interface during bonding, a strong localization of shear strain and stress (e.g., Fig. 6-4) is present in the regions near the interface. This localization of the shear strain and stress then caused the grains in these regions not only to be
Figure 7-3: The three incomplete (111) pole figures along with the ODF contour plots in $\varphi_2 = 0^\circ$ sections in a monolithic Al specimen after subjected to a heat treatment similar to the one for bonding Al and Al$_2$O$_3$ at the two surfaces (a), (c) and at the middle of the Al layer (b).
stretched but also to rotate while they were growing. This would certainly cause the texture to change. In the current study, the texture gradient is present in the regions near the interface except in the central regions of the specimens (e.g., Fig. 4-16). It is in good agreements with both the grain structure observation (e.g., Fig. 4-3) and the finite element predictions (e.g., Fig. 6-4). It is seen in Fig. 4-3 that grains around the central regions of the specimen are equiaxed even when the grains are close to the interface. In Fig. 6-4, the high shear strain and stress are not present in the regions near the symmetry plane (i.e., the central region).

It should be noted that in Köhler et al’s work [55] on monolithic F.C.C. metals, the rotated cube texture can also be found under different conditions. In their work, the rotated cube texture was found by simulating the recrystallization process in high stacking fault F.C.C. metals with different starting deformation textures. On condition that the orientation distribution of nuclei is random, and the initial deformation texture is pure B, pure Cu, or the mixture of S, B, and Cu, the recrystallization texture that corresponds to the fastest growing “compromise” orientations (i.e., 40°<111> local growth law) will be the rotated cube texture. In the current Al–Al₂O₃ bilayer composites, however, the rotated cube texture purely resulted from the interface constraints.

It can be justified that the texture effect may not be responsible for the evolution of the hardness gradient within the Al layer. As can be seen in Fig. 4-25, at the same distance from the interface, there is no hardness gradient across the specimen in the longitudinal direction although there is a texture difference in the longitudinal direction in the regions near the interface. In the texture measurement, the results have shown that regular cube texture is present in the central regions of the specimen (corresponding to about 4.5 mm – 7.5 mm in Fig. 4-25), and rotated cube texture is present in all the other regions (corresponding to about 2.5 mm – 4.5 mm and 7.5 mm – 11 mm in Fig. 4-25). On the other hand, there is an monotonic increase in hardness with increasing distance normal to the interface.

The results of the grain size measurement show that grains near the interface are
about 20% larger than grains near the surface, and the results of the microhardness indentation tests show that the hardness decreases by about 10% when the indented region moves from the surface to the interface. The two results can be correlated using the Hall–Petch relation,

\[ \sigma_y = \sigma_0 + k d_g^c, \quad (7.1) \]

where \( c \) is a constant and depends on materials properties. In general,

\[ \sigma_y \propto (\text{hardness index}, H_v) \propto (\text{grain size}, d_g)^c. \]

Figure 7-4 presents the result of the Hall–Petch relation by replotting Fig. 4-27 in a log–log scale. The trend line in this figure has a slope with the value of about -0.87, which is in reasonable agreement with the value that is commonly used for bulk aluminum alloys (i.e., “-0.5”) [82]. It should be noted that the Hall–Petch relation is not always satisfied [83], especially in the case of subgrain strengthening [84] and at large strains [82].

Two possible explanations are provided at the current stage to rationalize the existence of bigger grains near the interface. Firstly, based on the grain–boundary–energy point of view, the interface may have a strong effect on the redistribution of the surface energy of grains near it, and this effect may provide a favorable condition for grains near the interface to grow faster.

Another explanation may be made based on the depletion of secondary elements or impurities on the Al grain boundaries near the interface. In this case, since grain boundaries can migrate faster with less obstacles, grains close to the interface may grow faster and therefore bigger.

It is noteworthy that dynamic recrystallization is unlikely to cause the gradient of grain size in the current study as a result of the rare observation of dynamic recryst-
Figure 7-4: Hardness as a function of grain size in log–log scale.

tallization in pure Al [85,86] and the small strain rate deformation. In the current investigation, the thickness of Al was reduced by about 10% to 15% during 10 hours. That is, the average strain rate was on the order of 10^{-6}/sec. This value is too low to cause dynamic recrystallization in the current material system.

According to the work done by King [87] and Liang and King [88], although slanted and elongated grains can be caused (i.e., variation in grain size) by the mechanism of the so-called diffusion induced grain boundary migration (DIGM), this mechanism cannot give a satisfactory explanation in the current study as a result of the lack of interdiffusion between Al and Al\(_2\)O\(_3\) during the bonding process. In the current study, Al\(_2\)O\(_3\) is known to be very stable at the bonding temperature of 590°C, and therefore, only Al can diffuse into Al\(_2\)O\(_3\). It has also been verified by carrying out the wave dispersive spectrum (WPS) analysis on the cross-sectional plane of the Al–Al\(_2\)O\(_3\) specimen. The result showed that the oxygen concentration was very low within the Al layer (within the error range of machine’s capability in detecting oxygen), and beyond a distance of 10 \(\mu\)m from the interface, there was no significant amount of oxygen within the Al layer can be picked up by the WPS analysis.
Figure 7-5: Deformation mechanisms within the Al layer in a Al–Al₂O₃ bilayer composite, as a function of both the through-thickness location and the temperature, during a cooling process (from 200°C to 20°C). [89]

Moreover, Shen and Suresh [89] have theoretically predicted the steady–state creep response in metal–ceramic multilayers subjected to monotonic and cyclic variations in temperature. In their model systems, the predicted results showed that different dominant deformation mechanisms co–existed at different through–thickness locations within the Al layer for the Al–Al₂O₃ bilayer composite at any instant of time or temperature during either the heating or the cooling process. Figure 7-5 shows the occurrence of different deformation mechanisms within the Al layer in a Al–Al₂O₃ bilayer composite, as a function of both the through-thickness location and the temperature, during a cooling process only (from 200°C to 20°C). In this figure, “DG”, “C”, “P(LT)”, and “P(HT)” stand for the mechanisms of “dislocation glide”, “cobale creep”, “low temperature power-law creep”, and “high temperature power-law creep”, respectively. It can be seen that as a result existence of the stress gradinets within the Al layer during cooling, different dominant deformation mechanisms co–existed at different through–thickness locations within the Al layer. In their work, however, only one dominant deformation mechanism existed within the entire Al layer for the Al₂O₃–Al–Al₂O₃ trilayer system with the equal thickness of Al₂O₃, as a result of the
lack of stress gradient in the through-thickness direction. In the current study, the gradients of grain structure, texture, and microhardness were observed in both the Al–Al₂O₃ bilayer and the Al₂O₃–Al–Al₂O₃ trilayer specimens. Therefore, it is to say that the gradient of the deformation mechanisms within the Al layer during the cooling process may not be responsible for the experimental observations.

It should also be noted that although in reality, the localization of shear strain and stress exists in both the bilayer and trilayer specimens as a result of the non-perfect bonding between the Al and Al₂O₃ interface (i.e., certain degree of sliding), the shear strain and stress localization can only be responsible for the rotation and elongation of grains but not for the grain size gradient. This can be verified by looking at Fig. 4-6. Although this figure represents the grain size distribution on the cross-sectional plane that is around the central region of the specimen (where the localization of shear strain and stress does not exist), an increasing trend in grain size with decreasing distance from the interface is still observed.

Although in the current work, a number of mechanisms have been proposed and discussed to rationalize the existence of grain size gradient within the Al layer, further work such as the effects of the point defects, precipitates, cell boundaries, sub–boundaries, and dislocations on the mobility of grain boundaries, will need to be studied in order to give a clearer picture of the phenomenon.
Chapter 8

Conclusions and suggestions for future work

8.1 Conclusions

On the basis of the current findings, a number of conclusions can be drawn.

- The current work is the first experimental documentation of the grain structure, texture, and microhardness evolution of gradients in a metal which is solid-state diffusion-bonded to a ceramic.

- The grain structure gradient, texture gradient, and hardness gradient have been quantified experimentally for both the Al–Al$_2$O$_3$ bilayer and Al$_2$O$_3$–Al–Al$_2$O$_3$ trilayer composites with various Al thicknesses.

- Texture near the interface was characterized as the so called rotated-cube texture, and it becomes the regular cube texture in the regions away from the interface.

- Grains close to the interface are elongated, slanted, and about 20% larger than the near surface grains, which are equiaxed.
• In all the bilayer and trilayer specimens with various thicknesses, the size of the regions within which grains are slanted and elongated are about the same (within a distance of about 150 μm from the interface).

• This result may be explained by the anisotropic grain growth behavior (i.e., grains grow faster in certain directions in certain planes).

• The existence of gradients resulted from the interface constraints, which limited the mobility of grains and therefore caused strong shear strain localization in the regions close to it.

• Detailed computational analyses of grain structure and texture evolution using finite element method have been carried out to provide a mechanistic justification for the observed experimental trends, and the analyses are in qualitative agreement with the experimental observations.

8.2 Suggestions for future work

Based on the conclusions achieved in the current work, a number of future research directions are suggested.

• More detailed simulations using polycrystalline plasticity models that incorporates both the deformation and grain growth mechanisms can be carried out using the distributions of grain size gradient, texture gradient, and mechanical property gradient.

• Similar studies can be performed on material systems that are manufactured by other processing methods, such as physical vapor deposition (PVD), and plasma spray.

• Other metal–ceramic multi-layered composites, such as Steel/ZrO₂ high temperature corrosion resistant coating and steel/MCrAlY/ZrO₂ thermal barrier
coating (TBC) where MCrAlY serves as an interlayer between the metal and the ceramic, can also be investigated based on the current results.
Appendix A

Programs for processing X-ray raw data files

The following program reads the (111), (200), and (220) X-ray raw data files at one time, corrects, and converts into the PopLa files for calculating the orientation distribution functions.

program #2

```
#include "stdio.h"
#include "math.h"
#include "stdlib.h"
#define pi 3.141592654
#define absrptn_coeff 0.0102

void read_header();
void write_odf();
void polynomial_111_6_order();
void polynomial_200_6_order();
void polynomial_220_6_order();
double absorption_factor();
```
char input_name[15], output_name[15], input_name_odf[15];
char pole_fig_index[3];
int alpha, beta;
int data_points, data[20][80];
float defocusing_factor, thickness, max, n_factor;
float alpha_start, alpha_step, alpha_stop, beta_start, beta_step, beta_stop;
FILE *finput, *foutput;

/* This is the main block of this program */

main(argc, argv)
int argc;
char *argv[];
{
    int i;

    /* Check the command line arguments */
    if (argc != 5)
    {
        printf("Please enter the names of the data files and specimen thickness
(um)! \n");
        exit(0);
    }
    printf("Please enter the normalizing factor n_factor = ");
    scanf("%f", &n_factor);
    printf("\n");

    /* Open a new file for saving the corrected data */
    strcpy(output_name, argv[3]);
    strcat(output_name, ".epf");
    if ((foutput=fopen(output_name, "w")) == NULL)
    {
        printf("Sorry! Cannot open the output file! \n");
        exit(1);
    }
}
thickness=atoi(argv[4]);

/* Open the data file need corrected */
for (i=1; i<=3; i++)
{
    strcpy(inputname,argv[i]);
    strcpy(input_name_odf,argv[i]);
    strcat(inputname,".pfg");
    if ((finput=fopen(input_name,"r")) == NULL)
    {
        printf("Sorry ! Cannot open the input file !\n");
        exit(1);
    }
}

/* Reading the input file */
read_header();

/* Reading and Correcting the raw data */
max=0.;
printf("the index = %d",atoi(polefigindex));
if ( atoi(polefigindex) == 111 )
    corr_111();
else if ( atoi(polefig_index) == 200 )
    corr_200();
else if ( atoi(polefigindex) == 220 )
    corr_220();
else
{
    printf("\nThe pole figure plane is not in this program !\n");
    printf("\n(111) or (200) or (220) ONLY !!!!!\n");
    exit(1);
}

/* Write to the output ODF file */
write_odf();
/ * Close the working files */
         fclose(finput);
    }
    fclose(foutput);
}

/ * Reading header from the input file == The first 40 lines */

void read_header()
{
    int i,j,loop,index;
    char input_data[30],output_data[30],index_temp[4];

    fprintf(foutput, "%8s%8.4f \n",input_name_odf,n_factor);
    /* Start reading header */
    for (loop = 1; loop <= 19; loop++)
    {
        fgets(input_data,30,finput);
    /*
       fputs(input_data,foutput);
    */
    }
    /* read alpha_start, step, stop */
    fscanf(finput,"%f%f%f",&alpha_start,&alpha_step,&alpha_stop);
    /*
       fprintf(foutput, "%8.3f\n%8.3f\n%8.3f\n",alpha_start,alpha_step,alpha_stop);
    */
    /* read beta_start, step, stop */
    fscanf(finput,"%f%f%f",&beta_start,&beta_step,&beta_stop);
    /*
       fprintf(foutput, "%3.0f\n%3.0f\n%3.0f\n",beta_start,beta_step,beta_stop);
    */
    /* read pole figure index */
    for (index = 1; index <= 3; index++)
    {
        fscanf(finput,"%9s",index_temp[index]);
        strcpy(polefig_index,index_temp[1]);
        strcat(polefig_index,index_temp[2]);
        strcat(polefig_index,index_temp[3]);
        fprintf(foutput,"\%ls\%3s\%ls\%5.1f\%5.1f\%5.1f%2d%2d%2d%2d%5d%5d
",...
"(".pole_fig_index, ")", alpha_step, 90.0, beta_step, beta_stop + beta_step,
1, 1, 2, 3, 100, 17);

/**< read the rest of the header */
for (loop = 29; loop <= 43; loop++)
{
    fgets(input_data, 30, finput);
    /*
    fputs(input_data, foutput);
    */
}

/**< Start reading data */
alpha = ((alpha_stop - alpha_start) / alpha_step) + 1;
beta = ((beta_stop - beta_start) / beta_step) + 1;
data_points = alpha * beta;
}
corr_111()
{
    int i, j;
    double alpha_angle;
    float beta_angle, x_beta, data_temp, y_beta, radius;
    float repeat_1st_alpha, repeat_1st_beta, repeat_1st_intensity, alpha_radius;

    for (i = 1; i <= alpha; i++)
    {
        alpha_angle = alpha_start + alpha_step * (i - 1);

        /**< Calculating the defocusing factor */
        if (15 <= alpha_angle < 90)
        
            polynominal_111_6_order(alpha_angle);
        else if (alpha_angle == 90)
        
            defocusing_factor = 1.0;
        else
        {
            printf("Alpha_angle is not in the range (15 to 90)!\n");
            exit(1);
        }
for (j = 1; j <= beta; j++)
{
    fscanf(finput, "%f", &data_temp);
    data[i-1][j-1] = data_temp * defocusing_factor * n_factor;
    if (data[i-1][j-1] > max) max = data[i-1][j-1];
}
}
corr_200()
{
    int i, j;
    double alpha_angle;
    float beta_angle, x_beta, y_beta, data_temp, radius;
    float repeat_lst_alpha, repeat_lst_beta, repeat_lst_intensity, alpha_radius;

    for (i = 1; i <= alpha; i++)
    {
        alpha_angle = alpha_start + alpha_step * (i - 1);
        if (15 <= alpha_angle < 90)
            polynominal_200_6_order(alpha_angle);
        else if (alpha_angle == 90)
            defocusing_factor = 1.0;
        else
        {
            printf("Alpha_angle is not in the range (15 to 90)!\n");
            exit(1);
        }
    }

    for (j = 1; j <= beta; j++)
    {
        fscanf(finput, "%f", &data_temp);
        data[i-1][j-1] = data_temp * defocusing_factor * n_factor;
        if (data[i-1][j-1] > max) max = data[i-1][j-1];
    }
corr_220()
{
    int i,j;
    double alpha_angle;
    float beta_angle,x_beta,y_beta,data_temp,radius;
    float repeat_1st_alpha,repeat_1st_beta,repeat_1st_intensity,alpha_radius;

    for (i = 1; i <= alpha; i++)
    {
        alpha_angle= alpha_start+alpha_step*(i-1);
        if ( 15 <= alpha_angle < 90 )
            polynominal_220_6_order(alpha_angle);
        else if ( alpha_angle == 90 )
            defocusing_factor=1.0;
        else
        {
            printf("Alpha_angle is not in the range (15 to 90)!\n");
            exit(1);
        }

        for (j = 1; j <= beta; j++)
        {
            fscanf(finput,"%f ",&data_temp);
            data[i-1][j-1]=data_temp*defocusing_factor*n_factor;
            if ( data[i-1][j-1] > max ) max=data[i-1][j-1];
        }
    }
}

void write_odfO()
{
    int i,j,k,integer_data;
for (i = 1; i <= 16; i++)
{
    for (j = 1; j <= 4; j++)
    {
        for (k = 1; k <= 18; k++)
        {
            integer_data = data[alpha-i][(j-1)*18+k-1];
            fprintf(foutoutput,"%4d",integer_data);
        }
        fprintf(foutoutput,"\n");
    }
}
for (i = 1; i <= 12; i++)
{
    for (k = 1; k <= 18; k++) fprintf(foutoutput,"%4d",0);
    fprintf(foutoutput,"\n");
}
fprintf(foutoutput,"\n");
printf("\n%7.2f\n",max);

void polynomial_111_6_order(angle)
double angle;
{
    double C0,C1,C2,C3,C4,C5,C6,corr_factor_1,corr_factor_2, theta;
    
    C6=2.1114091376027600E−9;
    C5=−5.5202467297375100E−7;
    C4=5.9406444301960600E−5;
    C3=−3.4727914136567500E−3;
    C2=1.2296287415080100E−1;
    C1=−2.9029180593502100;
    C0=46.742652959129400;
    
    theta=19.235;
void polynomial_200_6_order(angle)

double angle;
{
    double C0,C1,C2,C3,C4,C5,C6,corr_factor_1,corr_factor_2,theta;

    C6=2.0674848144634200E-9;
    C5=-5.2769274794597900E-7;
    C4=5.5371337615739400E-5;
    C3=-3.1664904423252000E-3;
    C2=1.1084818869534300E-1;
    C1=-2.6620976138706300;
    C0=45.302282600982800;

    theta=22.37;
    corr_factor_1=absorption_factor(theta,angle);
    angle=angle-14.0;
    corr_factor_2=C6*pow(angle,6.) +C5*pow(angle,5.) +C4*pow(angle,4.) +
                   C3*pow(angle,3.) +C2*pow(angle,2.) +C1*pow(angle,1.) +C0;

defocusing_factor=corr_factor_1*corr_factor_2;
}

void polynomial_220_6_order(angle)

double angle;
{
    double C0,C1,C2,C3,C4,C5,C6,corr_factor_1,corr_factor_2,theta;

    C6=7.6087599903016300E-10;
    C5=-2.0694733338187000E-7;
    C4=2.4214011073442300E-5;

C3 = -1.6169795520362100E-3;  
C2 = 6.7418545637451600E-2;  
C1 = -1.8929781715451100;  
C0 = 35.514171724553300;  

theta = 32.565;  
corr_factor_1 = absorption_factor(theta, angle);  
angle = angle - 14.0;  
corr_factor_2 = C6 * pow(angle, 6.) + C5 * pow(angle, 5.) + C4 * pow(angle, 4.) +  
C3 * pow(angle, 3.) + C2 * pow(angle, 2.) + C1 * pow(angle, 1.) + C0;  
defocusing_factor = corr_factor_1 * corr_factor_2;  

double absorption factor(temp_theta, temp_angle)  
{  
double temp_numerator, temp_denominator;  

    temp_theta = pi * (temp_theta / 180.0);  
    temp_angle = pi * ((90.0 - temp_angle) / 180.0);  
    temp_numerator = (1.0 - exp(-2.0 * absrptn_coeff * thickness) / sin(temp_theta)));  
    temp_denominator = (1.0 - exp(-2.0 * absrptn_coeff * thickness) / (sin(temp_theta) * cos(temp_angle)));  
    return temp_numerator / temp_denominator;
Appendix B

Definitions and values of the parameters used in finite element simulation

The definitions and values of the parameters used in the unified constitutive equation refeq:unify in chapter 6 are listed below. Those parameters were obtained by Sample and Lalli [6.1] for commercial purity aluminum (PO815) during hot working and used in the current model as material properties. The flow stress $\sigma$ is defined as

$$\sigma = \sigma_{ss} \left[ \tanh \left( \frac{S^*}{S} \right) \right]^{0.2}$$

where

$$\sigma_{ss} = \frac{1}{\alpha} \sinh^{-1} \left( \left( \frac{Z}{A} \right)^{\frac{1}{n}} \right),$$

$$Z = \dot{\epsilon} \exp \left( \frac{Q}{RT} \right),$$

$$S = C(Z) \dot{\epsilon} \left[ 1 - \left( \frac{S}{S^*} \right) \right],$$

$$C(Z) = 15504.6 \exp(0.224Z^{0.051}),$$

$$S^* = 9.887Z^{0.22623},$$

and
\[ \mu_h = 23.94 + 0.727 \ln \left( \frac{S}{S_0} \right)^{1.924}. \]

The values for the above parameters are

\[
\begin{align*}
\alpha &= 0.032238 (Mnm^{-2})^{-1}, \\
 n &= 4.75, \\
\ln(A) &= 29.777 s^{-1}, \\
 Q &= 177244.3 Jmol^{-1}, \\
 R &= 8.3213 Jmol^{-1} K^{-1}, \quad \text{and} \\
 S_0 &= 1024.6 \text{ (complete recrystallization)}. 
\end{align*}
\]

The definitions of the parameters are

\( \sigma \): flow stress,
\( \sigma_{ss} \): steady state flow stress,
\( \alpha \): constant in constitutive equation ((Mnm\(^{-2}\))\(^{-1}\)),
\( \epsilon \): strain,
\( \dot{\epsilon} \): strain rate (/sec),
\( \mu_h \): microhardness (H\(_v\) 100g),
\( A \): reciprocal strain rate factor (sec),
\( n \): stress exponent in hyperbolic sine expression,
\( Q \): activation energy (J/mol),
\( R \): gas constant (J/molK),
\( S \): internal variable (hardness),
\( S_0 \): initial value of \( S \),
\( \dot{S} \): rate of evolution of \( S \),
\( S^* \): steady state value of \( S \),
\( T \): absolute temperature (K),
\( Z \): temperature compensated strain rate (/sec), and
\( C(Z) \): function in evolution equation.
Bibliography


