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Prediction and measurement of active slip in aluminum bicrystals and multicroystals

Yao, Zhicong, Ph.D.
The Ohio State University, 1990

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PREDICTION AND MEASUREMENT OF ACTIVE SLIP IN ALUMINUM BICRYSTALS AND MULTICRYSTALS

Dissertation

Presented in Partial Fulfillment of the Requirements for the Degree Doctor of Philosophy in the Graduate School of the Ohio State University

By
Zhicong Yao, B.S., M.S.

******

The Ohio State University
1990

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PREDICTION AND MEASUREMENT OF ACTIVE SLIP IN AL BICRYSTALS AND MULTICRYSTALS

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The slip behavior of thirteen aluminum bicrystal and multicrystal specimens was investigated after tensile strains of 1–7%. Finite element modeling (FEM), based on anisotropic elasticity, was used to predict the active slip systems and locations. The location and density of {111} slip agrees well with experiment in most cases, although some observed slip systems at external or internal surfaces have very low calculated resolved shear stresses (RSS). Some slip systems with Schmid factors ranking the second have the largest RSS at the grain boundary, based on the FEM calculation. This accounts for the observation of these secondary slip systems in the absence of higher–Schmid factor primary slip. Similar effects are found at gripped regions. The activation of these kinds of slip are predicted well by the FEM. In general, it appears that anisotropic elasticity can predict dislocation nucleation and propagation well at the early stages of deformation. In some cases, unusual slip on \{110\} or \{100\} planes was
observed near grain boundaries, slip bands, or free surfaces. These departures from the bulk behavior are probably related to micro-plastic compatibility and nucleation effects near the interfaces, and therefore could not be predicted by elastic effects.
To My Family
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LIST OF NOTATIONS

U — Upper end or Z=1 end of the specimens (+Z)
L — Lower end or Z=0 end of the specimens (−Z)
+Y — +Y edge or Y=w side of a specimen
−Y — −Y edge or Y=0 side of a specimen
G or GB — Grain boundary
GBab — Grain boundary between grain a and grain b
hdr — High deformation region
sb — Slip bands
E — Edges
C — Corner
RSS — Resolved shear stress
CRSS — Critical resolved shear stress
A_i(k) — Slip system A with ith RSS ranking and has a RSS value k. K is in an arbitrary unit.
Primary slip — Slip system with the largest Schmid factor
Secondary slip — Slip system other than primary slip
1.1 General remarks

Grain boundaries play a very important role in the plastic deformation of polycrystalline metals. The fundamental process of the deformation of polycrystals is the propagation of dislocations within individual grains and across the grain boundaries. Fundamental investigations of polycrystal deformation are usually focused on the structure of grain boundaries, the interactions of the associated dislocations in the vicinity of the grain boundary and the effect of grain boundaries on the behavior of dislocations. The influence of the grain boundary on the plastic deformation of polycrystalline metals is studied on both macroscopic and microscopic levels.

On the macroscopic level, it is often assumed that the grains in a polycrystal are either homogeneously deformed or stressed, and the grain boundaries are considered only as locations where the certain boundary conditions for stresses and strains have to be satisfied. However, the individual grains in a polycrystal are neither subjected to
the macroscopic stress system nor are they deformed like the macro—body. Internal equilibrium and continuity in a polycrystal must be maintained so that the grain boundaries between the deforming crystals remain intact. The constraints imposed by continuity cause considerable differences in the deformation between neighboring grains and within each grain [1]. In other words, since the grains of a polycrystal are neither plastically nor elastically isotropic, so the deformation of the grains produced by external stresses are not generally compatible and therefore additional stresses are induced to match the shape of a strained grain with the neighboring grains [2].

Microscopically, the plastic deformation of a polycrystal is described in terms of lattice dislocations. Lattice dislocations interact with the grain boundaries which are usually oriented unfavorably to the passage of dislocations. The interactions result in an increment of internal local stress. The general relationship between the flow—stress and grain size that was originally proposed by Hall and Petch [3,4] assumes that grain boundaries act as obstacles to the propagation of the dislocations. Because of the presence of grain boundaries, a residual dislocation is left at a grain boundary when a dislocation moves from one grain to another [2]. These residual dislocations form dislocation pile—ups in the boundaries and can further obstruct the passage of lattice dislocations. The dislocations impinging on the grain boundaries increase the stress field locally and may promote the nucleation of the lattice dislocation in neighboring grains.
Because of the constraints imposed by grain boundaries, the slip of dislocation takes place on several slip systems, even at low strains. The slip may occur on non-close-packed planes in the regions near the grain boundaries. Since more slip systems are usually operative in the vicinity of grain boundaries, the dislocation density and hardness will usually be higher near the grain boundaries than in the center of a grain [2].

In order to get more direct explanations for the experimental observations and to achieve a further understanding of the influence of a grain boundary on the plastic deformation of a polycrystalline metal, many investigations have been done with single crystals, bicrystals, tricrystals and polycrystals.

The deformation behavior of single crystals has been studied by many people since the 1920’s [5–105]. For the deformation behavior of bicrystals and tricrystals, many investigators started with flat grain boundaries [106–142]. Almost all the bicrystals used for studies in the literature had the grain boundary plane parallel to the load direction. Although a variety of models for grain boundaries have been established [143–157] and the behavior of grain boundaries in the plastic deformation of polycrystalline metals has been studied for a long time [158–198], some questions, such as how stresses are distributed in crystal grains and near the grain boundaries, or how much strain occurs on each slip
system during the process of non-uniform multiple slip, or how the stress field can be determined quantitatively, still are not fully answered. Some solutions are difficult to achieve experimentally. They have to be acquired with the aid of some theoretical or numerical analysis.

The Finite Element Method (FEM) is a general technique for constructing approximate solutions to boundary value problems. The basic idea of this method is to divide the domain of the solution into a finite number of simple subdomains which are called finite elements and to use variational formulations to seek an approximate solution [199]. FEM is a powerful mathematical tool suitable for the study of the effect of grain boundaries on the plastic deformation of polycrystalline materials since the consideration of mathematical boundary conditions gives rise to many of the observed effects of grain boundaries. Many investigations of stress distribution or grain boundary effects in single crystals, bicrystals and polycrystals have been done with FEM analyses [200—218].

The current study is aimed at understanding the slip behavior of each grain in a polycrystal. In order to reduce computational and experimental complexity, we concentrate mainly on bicrystals and tricrystals. The questions raised for us are how grain boundaries affect dislocation nucleation, propagation and stress distribution, how primary slip and secondary slip occur, where they occur, and so on. A comparison between our elastic continuum FEM simulations and experimental results shows a close match of activated slip systems in
most cases, including some specimen edge effects, grain boundary curvature effects and gripping effect.

1.2 Literature Review

This literature review mainly includes following five aspects: (1) deformation behavior of single crystals; (2) elastic and plastic properties of bicrystals and tricrystals; (3) grain boundary effect in polycrystals; (4) a survey of deformation research on aluminum single crystals, bicrystals and polycrystals; and (5) theoretical and numerical analyses of single crystals, bicrystals and polycrystals.

1.2.1 Single Crystal

Since the structures of polycrystals are more complex than that of a single crystal, and a polycrystal can be considered as an aggregate of single crystals, many investigators centered their attention on single crystals.

The plastic deformation of a single crystal was first studied by Schmid [5]. He introduced the *Schmid's law*, stating that plastic flow starts as the resolved shear stress (RSS) reached a critical value. This important law was verified experimentally, in hexagonal metals by Schmid *et al.* [6–8] and Jillson [9], in face centered cubic materials by Karnop and Sachs [10], Masima and Sachs [11], Sachs and Weerts [12], Osswald
Schmid's law was found to be approximately obeyed except near the boundaries of the elementary triangle where more than one slip system operated simultaneously. In those boundary regions, the resolved shear stress could be higher by a factor of up to 1.75 [16,17].

The orientation effect on the initial stress—strain curve was investigated in silver and aluminum by Rosi [18], Taylor [19], Lucke and Lange [20], and other investigators [21—22]. The results for aluminum are shown in Fig. 1.1. It is evident that the rate of work hardening is dependent on orientation and it is larger near the [100] and [111] corners of the standard triangle.

Schmid et al. summarized the three—stage stress—strain curve for f.c.c. metals [23]. They believed that many f.c.c. metals possessed a parabolic relationship between stress and strain with rapid hardening rate, whereas other types of metals with one strongly preferred glide plane showed a slow increment in stress. The reason for this phenomenon in f.c.c. materials was interpreted as the interaction of primary slip and other slip. These interactions might come from sessile dislocations which acted as obstacles to further glide. The obstacles could be Lomer—Cottrell barriers [24,25], Hirth Locks [26,27], Kocks dipoles [28], Thompson stacking faults [29,30], Nabarro point defects [31], or free surfaces [32]. To overcome these obstacles, more stress is required [29].
Fig. 1.1 Stress-strain curve for pure Al single crystals [22]
were at sub-grain boundaries, which agreed with the increase of etch-pit density near sub-grain boundaries on the cross slip plane, with no increase on the primary glide plane [40,45-46].

In stage I, the stress was observed to increase largely due to twisting [40,47]. And hardening was found to be sensitive to the condition of the surface [32,48-50]. Kramer et al. found that electro-polishing of the specimen caused stress drops [32].

Livingston [51] and Basinski [44] studied stress strain curves in copper and found that stress increased slowly in the transition from stage I to II region. They believed that the long dipoles were cut into short segments. Their etch-pit studies both showed that "forest" dislocations ("forest" dislocations refer to a relatively immobile set of dislocations which serve as obstacles to the mobile dislocations) were almost as numerous as primary dislocation by the end of stage II. And the primary dislocations piled up at sub-grain boundaries, whereas forest dislocations did not. This implied that primary dislocations had a larger mean free path.

Seeger et al. found that the slip bands in copper were broad in stage II [40]. He noticed that many old bands continued to grow if the crystal was polished and strained again. The investigations of Mader [52] and Blewitt et al. [53] indicated that the lengths of the individual slip lines were inversely proportional to the strain in stage II. In
Some studies showed that a large number of dislocation dipoles were at sub-grain boundaries, which agreed with the increase of etch-pit density near sub-grain boundaries on the cross slip plane, with no increase on the primary glide plane [40,45−46].

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and Blewitt et al. [53] indicated that the lengths of the individual slip lines were inversely proportional to the strain in stage II. In Mader's study, the irregularities of kink band did not seem to have a strong effect on stress strain curve [54].

Mitchell and Thornton studied stress strain curves for Al, Cu and Ni [17]. They found that the work hardening rate in stage II was dependent on the initial orientation of the crystal in the same way as in stage I, but this dependence was not affected by cross slip.

The recent study of Higashida et al. on the generation of secondary slip in stage II in Cu single crystal indicated that a sudden occurrence of secondary slip at stage II was directly related to the formation of slip bands [55]. Among the secondary slip, cross slip systems with zero Schmid factor were observed. It was believed that secondary slip was caused by the internal stress induced by primary slip.

Kramer studied surface effects and found that the surface had less influence in stage II than in stage I [56]. The dislocations were less tangled on the surface than in the bulk, and were randomly arranged.

Nabarro pointed out that the stress was larger by a factor of 1.5 to 2 for the crystals with initially symmetrical orientations than for the crystals with initial tensile axes near the middle of the unit triangle.
And the strain at the beginning of stage III was dependent upon orientation in much the same way as the strain at the beginning of stage II.

Mader found that the slip bands, in stage III, were coarse and fragmented with abundant cross slip and individual slip bands [52]. In the investigation of Blewitt et al. [53], the cross slip in stage III was found to be thermally activated. They found that it is possible, if crystals were strained at low temperature, to avoid the occurrence of stage III; twinning on \{111\} would happen when stress reached a high value.

Specimen gripping was believed to be important in the uniaxial tensile deformation of f.c.c. metals [58]. It mainly affected the transition from stage I to stage II. The grip constraint was dependent on the bending stress and the shear stress. Inoko et al. recently studied the grip effect on aluminum single crystal tensile specimens [59]. Their result revealed that the grip had no effect on the formation of the deformation bands, but it influenced the intrinsic properties such as sub-grain boundaries.

For fcc metals, the parameter $\gamma/b\mu$ was used as the principal factor determining the work hardening behaviors [57]. Here $b$ is the slip distance, $\mu$ is the elastic modulus and $\gamma$ is the stacking-fault energy. Thornton et al. found that the total strain would be large in stage I if
the stacking-fault energy $\gamma$ was low [60]. But the work hardening rate in stage II was not affected by the value of parameter $\gamma/b\mu$ [57]. Since the onset of stage III was determined by the cross slip of screw dislocations, therefore, in the metals of high stacking fault energy such as aluminum, the stage III would take place at low temperature and at low stresses.

In aluminum, it was found that macroscopic slip began on the conjugate slip system when the RSS was lower than that on the primary slip system, and the stress on the conjugate system was never more than 1.02 times that on the primary system [61–62].

For stainless steel, the important observations were made by Whelan [63] and by Hirsch [64]. Their studies showed a completely different dislocation pattern from that in high stacking fault energy metals. Dislocations piled up on primary slip planes and Lomer–Cottrell locks were abundant in stage III. Dislocation networks appeared with wide stacking-faults. No cross slip was observed.

Deformation bands were studied by many investigators [65–71]. It was believed that the formation of deformation bands (kink band, shear band) had an important influence on the plastic flow behavior of crystals. Cahn observed deformation kink bands in aluminum single crystals after 1 or 2% strain [66]. The slip was primarily single slip. Honeycombe [65] noticed that the bands did not occur with double
primary conjugate slip from the beginning of plastic flow. Chang and Asaro studied the localized shear band formation in the single crystals of Al—Cu alloy [71]. They mentioned two major differences in the kinematics of macroscopic shear bands and coarse slip bands. The material planes of macroscopic bands were not aligned with either of the two slip systems in the adjacent parts of the crystal and the lattice within the bands was misoriented with respect to the lattice outside. The lattice rotation increased the Schmid factor in the band and the band hardened more. Asaro later introduced some mathematical models for the formation of deformation bands [72].

BCC metals

The plastic properties of b.c.c. metals were found to be extremely sensitive to the presence of carbon, nitrogen and oxygen as interstitial impurities [73]. However, the sensitivity to impurities was found not to be strong in large lattice parameter metals [74]. Kimura and Matsui studied the hydrogen effect on work hardening behavior of high purity iron single crystal, at temperatures between 200K and 296K [75]. The result showed that hydrogen caused softening in stage I and resulted in an expansion of stage I. It was found that hydrogen induced semi brittle fracture.
In b.c.c. metals, the closest packed plane is \{110\}, and direction is \langle111\rangle. In general, \langle111\rangle slip can occur on \{110\}, \{112\} or \{123\} planes. The typical stress—strain curve of a single crystal of b.c.c. metal starts at a stress which is often much higher than that in f.c.c metal [57]. Some studies showed that the initial flow stress almost always increased very rapidly as temperature decreased [17,76—78]. Temperature affected the stress strain curve differently in different stages, for example, molybdenum slipped on \{101\} plane at above 1300°C, and only one stage of hardening was observed [78]. As temperature decreased, the critical resolved shear stress increased rapidly [76], Fig. 1.2. shows the dependency of critical resolved shear stress on temperature of different orientations.

Cottrell proposed that the flow stress in b.c.c. single phase single crystals was generally composed of three parts: an intrinsic resistance to dislocation motion, the stress associated with the dislocation configuration, and a stress arising from the presence of interstitial impurities [79]. For the structure hardening of b.c.c. materials, the basic mechanisms were believed to be similar to those in f.c.c. metals with Lomer—Cottrell interactions. Keh and Weissmann [80] pointed out that both the long—range stress field and the short—range dislocation interaction in cell walls must be effective in hardening. They found that the dislocations in the cell walls formed pile—ups. These pile—ups contributed to the mean stress more than dislocations in dispersed form.
Fig. 1.2 Relationship between CRSS and temperature [76]
Matsuda investigated the work hardening behavior of iron single crystals [81]. He observed three-stage hardening in the stress-strain curves at 195°K and 300°K for shear tests on the (112) plane, whereas a parabolic type hardening showed for the crystals sheared on the (101) plane. The dislocation distribution in iron single crystals during three-stage hardening at high temperatures was very inhomogeneous and this sort of behavior was markedly dependent on the shearing direction. In the crystals in which the applied stress deviated from the slip direction [11I], the dislocation cell structure began to form from stage II. However, the dislocation structure was found to be similar for crystals sheared on both the (112) and (101) planes.

Bolton and Taylor performed an analysis on the anomalous slip in high purity niobium single crystals deformed at 77°K in tension [82]. Their results showed that most of the deformation occurred on the low Schmid factor systems (0T1) [T11] and (0T1) [111] with very little slip on the primary (T01) [111] and conjugate (101) [T11] systems.

Nabarro found that the electron microscopy dislocation patterns of bcc iron deformed to higher strains were similar to those formed in f.c.c. metals of low stacking-fault energy at high temperatures [57]. At 3-5%, strain a well-marked cellular structure was developed from a pattern of jogged screw dislocations accompanied by edge dipoles and loops formed at 1-2% strain. As the strain reached 9%, the cell walls
were densely filled with dislocations, but their interiors were quite clear. It was observed that at lower temperature, the cell structure was formed at higher strains, and the cell size remained constant after about 8% strain around room temperature. It was also found that temperature influenced the cell size. The final cell size increased moderately with increasing temperature up to 300°C, and rapidly thereafter.

HCP metals

Studies on hexagonal metal single crystals were carried out by many investigators. For example, the materials used were zinc [41–42,83–84], cadmium [7], or magnesium [43]. All the results of these investigations showed that the stress strain curve was similar to that of f.c.c. metals, except for much larger extensions in corresponding portions of the curve. Although there was a general agreement about the curves, the h.c.p. materials did not show as much dependency of work hardening rate on crystal orientation as f.c.c metal did [41,42].

Because of only one basal slip plane in h.c.p. metals, it was found that the deformation was extensively implemented by twinning as well as by glide [57]. Seeger and Trauble [42] made a discussion on the work hardening of h.c.p. metals. They indicated that in stage II the deformation in h.c.p. materials was similar to that in f.c.c metals. However, the stresses were generated by isolated dislocations, not by pile-ups. The transition from stage I to stage II occurred as the rate
of generation of vacancies was so high that vacancies were not all absorbed in the climb of edge dislocations. Sessile dislocation loops in the basal planes were built up.

Diamond structure material

The deformation behavior of diamond structure materials such as silicon and germanium was studied by some investigators such as Bardsley [85], Dash [86], Booker and Stickler [87], Thomas [88]. It was believed that dislocation stresses could be relieved by forming a 'crack', and broken bonds could often be eliminated by some complicated rearrangements such as removal of atoms to interstitial sites. In both cases, the motion of dislocations required thermally activated diffusion.

Slip on the secondary systems

The concept of latent hardening is often used to measure the difficulty of activation of secondary slip [89—96 etc.] The latent hardening ratio (LHR) is defined as follows [90]: if a crystal is pre-deformed to a flow-stress $\sigma_1$, the glide system is changed, and the initial flow-stress on the new system is $\sigma_2$, then LHR is $\sigma_2/\sigma_1$.

Basinski and Basinski studied secondary slip, dividing it into two groups: coplanar slip and slip on a slip plane other than the primary slip plane [90]. It was found that LHR was equal or near to 1 for
coplanar slip system, whereas always markedly higher for the intersecting systems. For aluminum, LHR was 1 in stages I and II; for copper, LHR was 1 in stage I, but greater than 1 in stage II. The value of LHR, for copper, could be as high as 2.6 for intersecting systems. The LHR of intersecting secondary slip was found to be very high at the beginning of the deformation, and then to drop rapidly with increasing pre-strain. It was structure sensitive. Havner and Shalaby developed a mathematical model for latent hardening in single crystals, and it was in a qualitative agreement with their experiments [94].

Taylor and Elam first observed that the tensile axis was rotated as deformation continued [97]. They found that the crystal would rotate so that two slip systems would become geometrically equivalent. Elam found that the tensile axis near [110] overshot the symmetric boundary, but it did not move for [211] in copper [35].

Basinski and Basinski indicated in their paper that all the pure metals overshot the symmetric boundary, and the rotation of the tensile axis slowed down progressively and gradually reversed the direction [90]. However, in some studies, no overshoot was found for aluminum material [17,62,98]
Summary of single crystal research

According to the Schmid law, if the shear stress resolved on a slip plane and a slip direction reached a critical value, the single crystal yielded. The Schmid law was found to work very well in almost all the single crystals. However, low Schmid factor slip systems were observed without the existence of high Schmid factor slip systems in single crystals [52,82].

In general, a three stage $\sigma-\epsilon$ curve can be observed in f.c.c. metals and b.c.c. metals at high temperature. The $\sigma-\epsilon$ curve of h.c.p. metals is similar to that of f.c.c. metals, except having much longer extension in each corresponding portion of the curve. The deformation of h.c.p. metals is carried out by twinning and by gliding. For diamond structure materials, their deformation can be implemented by breaking bonds, complicated atom rearrangements or even forming cracks. It was believed that the work hardening rate would be low when single crystal is orientated so that one slip plane is initially favored. A high initial work hardening rate could be observed if two or more slip systems are equally favored.

In stage I, from etch—pits study, it was found that at the region with more etch—pits, the density of the etch—pits increased faster as strain increased [8]. The work hardening rate is strongly dependent on crystal orientation. For fcc metals, stacking fault energy ($\gamma$) plays
an important role in the stress–strain curve. The stage I would be short for a high γ material, such as Al. It was found that electro-polishing could soften the crystal [32,56], whereas, surface coating of the other kind of metal could harden the substrate [105]. For bcc metals, the initial flow stress would increase rapidly with decreasing temperature, Fig. 1.2 [76]. It is possible that only stage I would be observed if the deformation temperature is low [78]. The stress–strain curve is very sensitive to impurity for bcc metals. For hcp metals, strain is much larger than that for fcc metals. At the transition from stage I to II, the hardening effect was caused by cutting long dislocation dipole into short segments and primary slip dislocations pile-up at sub-grain boundaries. It was found that surface oxide would reduce the critical resolved shear stress in stage I for bcc metals [101–104].

At the beginning of stage II, secondary slip starts. It was believed that primary slip may make the secondary slip difficult which is called latent hardening [89–96]. For a high latent hardening ratio (LHR) crystal, high stress would be needed to activate the secondary slip. If a secondary slip with a same slip plane as primary one, the secondary slip would be easy to operate. LHR for this kind of slip system is equal or close to 1 [90]. Whereas, for the secondary slip with slip plane other than primary slip plane, LHR would be very high at the beginning of the deformation, and then decreasing with increasing strain. LHR was found to be structure sensitive. For fcc metals, the work hardening rate in stage II was dependent on initial orientation of
the crystal in the same way as in stage I, and it was not affected by cross-slip [17]. Formation of slip bands is caused by primary slip [55]. The work hardening rate is not affected by stacking fault energy. For bcc metals, dislocation cell structures begin to form at stage II [80,81]. The hardening effects come from the dislocation interactions in cell walls which form pile-ups. These pile-ups contribute to the mean stress more than dislocations in dispersed form.

In stage III, multiple slip occurs. For fcc metals, the strain at the beginning of stage III is dependent upon orientation in much the same way as that in stage II [57]. Slip bands are coarse and fragmented with abundant cross-slip and individual slip bands [52]. It was found possible that if a crystal was strained at low temperature, the occurrence of stage III could be avoided [53]. Twinning on \{111\} planes would happen when the stress reached a critical value. For high stacking fault energy materials, stage III would take place at low temperature and at low stress. For bcc metals, the dislocation patterns formed at high strains were similar to those formed in fcc metals with low stacking fault energy at high temperature [57]. At 3—5% strain, cellular structure is well marked. As strain reached 9%, the cell walls are densely filled with dislocations, but their interiors are quite clear. The lower the deformation temperature, the higher the strain for the cell structure to form. If the deformation temperature is very low, stage II and III could be avoided, and twinning would occur.
Generally, work—hardening comes from dislocation interactions which form obstacles to further motion of the dislocations, especially in stage III. To overcome these obstacles, higher stress is required [29].

1.2.2 Bicrystals and Tricrystals

Incompatibility and heterogeneous slip

Clark and Chalmers studied deformation behavior of aluminum bicrystal tensile specimens [106]. They found that it was possible that the bicrystal specimens began to yield at the same stress as the corresponding single crystals, and the dislocations in regions far from a grain boundary could move at the same stress as in the single crystal. The grain boundary caused dislocations to pile—up and to produce new dislocations to continue the deformation. The grain boundary made the easy glide (stage I) region shorter and a greater resolved shear stress should be applied to operate the latent slip systems. As a result, the work—hardening rate was higher in bicrystals than in corresponding single crystals, and work—hardening rate increased with increasing orientation difference between the crystals.

Livingston and Chalmers [1] analyzed multiple slip in aluminum tensile bicrystals. They observed that macroscopic slip continuity at grain boundary required at least four slip systems in the bicrystal. The
four slip systems can be distributed as two in each crystal, or one in one crystal, three in the other crystal.

Hauser and Chalmers investigated plastic deformation of silver bicrystals in tension [107]. One of the bicrystals was made so that one crystal was inside the other one, and the two crystals were then sintered together. It was found that for this surrounded bicrystal, deformation occurred on six slip systems, while in a similar case, the bicrystal without one crystal totally surrounded by the other one, the deformation occurred only on four slip systems. The totally surrounded bicrystal was harder than other bicrystals and was a better approximation of the polycrystal. It was also found that the slope of shear stress—shear strain curve of this bicrystal was the same as that of the polycrystal. The major effect of incompatibility was to produce multiple slip in the vicinity of the grain boundary, and the effect was mainly on the easy glide region.

To study the grain boundary effect, many investigations were of the most simple bicrystal in which the grain boundary is a flat plane and with plane normal perpendicular to the load axis [108—141]. The typical example is the Fe—3%Si compression bicrystal experiments performed by Hook and Hirth [113—116]. They found that strong elastic and plastic incompatibility effects were caused by grain boundaries. In their studies, two kinds of bicrystals, whose grain boundaries were parallel to the load axis, were subjected to a small amount of plastic
strain by being deformed in compression. One kind was a series of isoaxial bicrystals in which the two crystals had same crystallographic compression axis. The other kind was a series of nonisoaxial bicrystals having different crystallographic load axes in each component. Because of the elastic and plastic incompatibility at the grain boundary, the secondary slip was favored in the vicinity of the grain boundary. For isoaxial bicrystals, secondary slip sometimes even occurred at positions where no primary slip was observed because of the elastic incompatibility (EI type — slip systems which appear only near the grain boundary and which have no one—for—one matching with slip systems across the boundary). And these types of slip were observed to be different at front and back surfaces within each grain. For nonisoaxial bicrystals, no primary slip was observed in the crystal with (001) tensile axis, and the secondary slip activated (S or SA — slip systems observed at the boundary which have high coincidence with primary slip systems across the boundary and usually appear near a free surface) by the primary slip of the other component was different at the front and the back surfaces in the grain. (Simulations for these experimental results were done by Wagoner et al., and will be discussed later). Some secondary slip was observed close to the compression interfaces, and some slip such as second—order slip (SO — slip systems which appear across the boundary from non—primary slip systems) caused by the plastic incompatibility at grain boundary was observed, too. These second—order slip systems did not have high Schmid factors like other observed slip systems.
The stress fields of dislocation arrays at interfaces in bicrystals were calculated by Hirth et al. [117]. In their study, a physical interpretation of the elastic fields giving rise to the single dislocation traction for isotropic elastic case and the long-range elastic fields of dislocation walls being removed by uniform surface traction leading to uniform shear and rotations of the component crystals were performed. Also a proof that the long-range elastic fields could be removed in the general anisotropic elastic case was presented.

Dislocation spacings in pile-ups at grain boundaries were calculated by Wagoner [118] using the nine bicrystal orientations examined by Hook and Hirth [114—116]. It is believed that the dislocation pile-ups can be affected by crystal anisotropy and bicrystal orientation relationship in several ways, and the strength of dislocation interactions (and therefore, spacing), stress distributions and the number of dislocations required to reach a critical shear stress can be affected by the crystal elastic properties on both sides of the obstacle boundary.

A special kind of bicrystal containing α—β interface (Ti—13Mn) was studied by Lee and Margolin [119]. The bicrystals were composed of α and β single crystals with flat boundary plane parallel to the load axis. It was observed that the α and β components behaved differently from those of α and β single crystals because of the grain boundary effect. Similar to the experimental results of Hook and Hirth, the slip systems operating in α and β components at front and back surfaces
were different. The behavior of $\alpha$ and $\beta$ components in the bicrystals was different from that of the corresponding single crystals because of the stress induced at grain boundary and the compatibility requirements. Their calculation showed that the stress at the interface deformation zone was about twice as the average bicrystal flow stress. It was not clear why the slip system with high Schmid factor was not activated.

The deformation of tungsten bicrystals with flat twist grain boundary planes and the plane normal parallel to the load axis was investigated by Fekete et al. [120]. Because tungsten is an isotropic material, the secondary slip activated at grain boundary was caused by plastic incompatibility. An interesting thing was that the secondary slip was found to be the slip system with zero Schmid factors. The origin of the effect was believed to be grain rotation. As plastic deformation increased, the two crystal components in the bicrystal tried to twist around the load axis. Secondary slip was observed to increase if the misorientation angle (twist angle) between the two crystal components increased.

Rey and Zaoui found that the heterogeneity of plastic glide occurred at the beginning of plastic deformation could vanish in certain orientations of the specimen as deformation increased [121,122,134]. They assumed that the initiation of plastic deformation was governed by a geometrical process. A new method derived from the continuous dislocation theory was applied to examine the internal stress. The
results fit the experimental observations, and showed that the incompatible copper bicrystals exhibited stronger latent hardening phenomena than the aluminum bicrystals.

Maruyama et al. studied the effect of plastic strain incompatibility on zinc bicrystals [135]. They used isoaxial zinc bicrystal specimens, compatible and incompatible ones, whose grain boundaries were parallel to the tensile axis. It was found that the secondary slip lines observed near the grain boundary in the incompatible bicrystals at an early stage of plastic deformation did not show up in the compatible ones. The density of the secondary slip lines increased with the incompatibility between primary slip systems of component crystals. The secondary slip systems releasing the incompatibility were operative in the incompatible bicrystals. The slip systems irrelevant to incompatibility were proposed to be activated to cancel the bending moment caused by a shift of the tensile accompanying slip on the primary slip system. Regardless of misorientation angles between the component crystals, the 0.1% proof stress was found for the compatible bicrystals nearly equal to, and for incompatible ones larger than, those of the single crystals of the same orientation. It was assumed that the increase in the stress was caused by multiple slip near the grain boundary, induced to release the incompatibility.
Deformation zone model

A so-called grain boundary deformation zone, in which the flow stress at grain boundary region was assumed to be different from that in the bulk away from grain boundary, was introduced by Margolin et al. [123—125]. Lee and Margolin studied the stress—strain behavior of β—brass single crystals, bicrystals and tricrystals for a series of isoaxial specimens in which the bicrystals and tricrystals were incompatible in shear along the grain boundaries [124]. The bicrystals had a considerably higher flow stress than single crystals did. However, the strain hardening of the bicrystal was only slightly lower than that of the tricrystals. It was found that the grain boundary deformation zone was larger in the tricrystals than in the bicrystals. This evidence implied that the stresses of secondary slip extended further from the grain boundaries in the tricrystals than in the bicrystals, and the average grain boundary flow stress for the tricrystals was smaller than the corresponding stress for the bicrystals, despite the increased constraint. Chuang and Margolin performed an investigation on the stress—strain relations of β—brass bicrystal [123]. They believed that the grain boundary deformation produced strengthening by the interaction of multiple slip systems as well as by blocking of slip in one grain by an adjacent grain. The effectiveness of the blocking slip was assumed to be dependent upon the orientation of the adjacent crystals. From the comparison of their experimental results of β—brass isoaxial and nonisoaxial bicrystals, they found that the nonisoaxial bicrystal whose
grain boundaries were parallel to the stress axis produced much more strengthening than isoaxial bicrystals did. This strengthening by the grain boundary of nonisoaxial bicrystals led to support for a large average grain boundary stress in the hard crystal. The isoaxial β—brass bicrystals were elastically and plastically incompatible in the same way in shear, and this incompatibility led to the formation of a clearly defined grain boundary deformation zone. Outside the grain boundary deformation zone, each component crystal of the bicrystal was found to behave as a single crystal. A similar model was proposed by Trefilov et al. [127].

Slip in low angle grain boundaries

In studying the deformation behavior of germanium bicrystals, Bailllin and Pelissier, using high voltage electron microscopy, found that their bicrystal containing \{122\} Σ=9 grain boundary was a strong obstacle to intergranular dislocation motion [128]. The stress concentration appeared as dislocations piled up at the grain boundary. An investigation on the micro deformation of Σ=25 [001] tilt bicrystals of silicon was performed by Martinez—Hernandez et al. [129]. They found that the grain boundary strongly opposed the dislocation motion in silicon and the transmission of the blocked dislocations across the grain boundary required large stress concentrations arising ahead of pile—ups or deformation bands. Lin and Wu performed an analysis of plastic deformation of niobium single, bicrystal and polycrystal [131].
bicrystals used in their study were made with three different low tilt angles respectively. Their result showed that the misorientation angle had a significant effect on the stress on both monotonic and cyclic deformations. They explained that the effect of the grain boundary in the bicrystals was in the activation of the extra slip systems to satisfy various compatibility requirements because, under a uniform axial elastic strain, the bicrystal exhibited an additional effect of elastic incompatibility which was raised by elastic anisotropy. This incompatibility resulted in additional stress at the grain boundary.

The deformation behavior of low sigma coincidence twin or twist boundary bicrystals recently had some special attention [2,128—133]. For instance, the plastic deformation of symmetrical bicrystals of Fe—5.8%Si with Σ=3 coincidence twin grain boundary were studied by Paidar et al. [2]. The experimental result showed that the central part of the bicrystal was deformed by primary slip on the slip planes identical with those of single crystals having the orientation of corresponding component grains, and the macroscopic changes of the grain boundary plane induced by the accumulation of residual grain boundary dislocations were in a good agreement with the calculations based on the independent deformation of component crystals. It was found that the end or grip effect was related to the restrictions imposed on the grain rotation which could be eliminated in a single crystal.
Summary of bicrystals and tricrystals

The deformation of bicrystals and tricrystals has been investigated by many investigators [106—142]. Because of the compatibility requirement imposed at grain boundary, elastic and plastic incompatibilities increased the local stress at the grain boundary plane which induced secondary slip at the grain boundary. These slip systems were not usually expected in the individual single crystal component. Chalmers et al. [106,107] did an investigation on fcc metals and found that grain boundary hardening increased with increasing degrees of incompatibilities and the deformation behavior of the bicrystal with one grain totally surrounded by the other one was similar to that of polycrystal. Hook and Hirth [114—116] observed the elastic incompatible slip (EI) and secondary activated slip (S or SA) in Fe—3%Si compression bicrystals. Because of the elastic incompatibility, this secondary slip was different at front and back free surfaces in each single crystal component. The Schmid factors of the second order slip in the two single crystal components caused by plastic incompatibility were found to be quite low. In the bicrystal composed of α and β single crystals, the observed slip systems in each component at front and back surfaces were also found to be different [119]. Secondary slip with low Schmid factors activated to release the incompatibility at grain boundary [119,120].
Heterogeneous plastic deformation was observed by many investigators [114—116, 119—126, 134—135]. The results showed that the flow stress at grain boundary region was usually higher than that in the bulk material because more dislocation were induced by grain boundaries. At the region away from the grain boundaries, the deformation behavior can be treated as a single crystal. For different materials, the degree of heterogeneity was different. Copper was believed to have a stronger latent hardening effect than aluminum.

Some people believed that a low angle boundary forms a strong obstacle to the motion of dislocation [128—133]. For example, in Si bicrystals [129], grain boundaries were found to oppose strongly dislocation transmission from one grain to the others; in niobium bicrystal with low tilt angle boundary, extra slip systems must be operated to satisfy the various compatibility requirements [131]; in Σ=3 twin boundary Paidar discovered that the central part of bicrystals was deformed like a single crystal [2], and at the ends of the specimen, because of the grip effect, grain boundary plane could be changed by residual grain boundary dislocation and hence induce other slip systems.

The deformation behaviors of aluminum bicrystals will be discussed in detail in section 1.2.4.
1.2.3 Polycrystals

For polycrystalline metals, grain size refinement is one of the ways to increase the strength of materials. Because of the existence of the grain boundaries, complicated stress fields can be induced by incompatibilities. To release the internal stress, more slip systems would be operated. This strengthening mechanism is dislocations interacting with each other and dislocation interacting with the grain boundaries.

Nucleation of dislocation

It was generally believed that there was microplastic yield in polycrystalline materials before the flow stress reached upper yield point. In the study of the plastic microstrain in silicon—iron polycrystal specimens, Suits and Chalmers [162] found that dislocation etch—pits were irregularly nucleated and were piled—up after the specimen was stressed to 90% of the yield stress. The spacing between etch—pits in a given slip line was not uniform. In some regions, yield propagated from one grain to an adjacent grain. Slip continuity across the grain boundary was observed. They believed that the formation of slip bands was governed by the presence of randomly distributed stress concentrations in the matrix. A similar experiment was done on silicon iron by Worthington and Smith [163—164]. They found that slip was often coming out from one side of a grain boundary because not all the grains had sources in their boundaries. They also found that more than one
slip system appeared before slip started in the adjacent grain.

Carrington and McLean studied the dislocation nucleation in the commercial pure iron—silicon alloys [166]. The specimens were strained below and into the Luders' extension. It was found that slip nucleation at grain boundaries was greatly associated with the grain orientation. If a grain was in special orientation, yield would not occur until the stress at the adjacent grain rose to the critical value at the end of the slip bands, that could promote nucleating slip in perfect crystal.

On the contrary to the above observations, McLean found [167] that sources for the pre—yield microstrain slip bands were all at the grain boundaries. Margolin et al. observed the similar thing; that the slip began at or near grain boundaries, then proceeded inward with increasing strain [168]. The slip appeared as a single set of parallel bands referred to as the primary slip. The secondary slip started at grain boundaries but was much less evident. A study, on the contrary, showed that the slip could occur within the grain, with no evidence of slip having started at grain boundaries [169].

Gibala et al. did many investigations of the effect of surface coating on the stress—strain curve for bcc metals single crystals [101—104]. They found that surface oxide reduced the critical resolved shear stress and increased the ductility of the crystals [104]. They believed that surface film could act as an efficient semi—continuous
source of mobile non-screw dislocations which can move at greatly reduced stresses compared to the Peierls stress of screw dislocations, although the dislocation generation and motion processes were different for different materials [101–103]. The effect of the surface oxide on the operating slip systems was not mentioned.

Pridans and Bilello [105] studied the effect of a surface coating of chromium on high purity copper single crystals. The surface layer was 1µm thick chromium fine grain polycrystal which was incompatible with the substrate. The flow stress was increased by the interaction between the surface layer and substrate.

Kelly indicated that electro-polishing of the specimen could lead to a flow stress drop in aluminum crystal [170]. Sumino and Yamamoto found that electro-polishing off the surface from aluminum specimens could cause a flow-stress drop at any stage of the deformation [171]. Kramer found a similar phenomenon in aluminum specimen [32,56,172]. But he also found that these flow-stress curves climbed up until it was equivalent to an non-electro-polished specimen. He believed that the reason was the removal of the hard surface layer by polishing. As strain increased, the surface hardening layer effect would not show.
Slip propagation and continuity at grain boundary

No matter where the slip starts, grain boundaries at "low temperature", interacting with the lattice dislocations will be unfavorable to the slip and would change the behavior of the slip at or near grain boundaries. Ogilvie studied the slip band continuity at grain boundaries in polycrystalline aluminum and α-brass specimens [173]. He found that continuity occurred across the straight grain boundaries provided the lines of intersection of the slip plane with the boundary were observed within 2° of one of the directions <110>, <112> or <123> for each grain, not necessarily the same direction in the adjacent grains. In α-brass, the slip band continuity was observed not to be confined to the surface but a volume effect. In Kratochvilova and Sestak's [174] study of slip bands in 3% silicon iron alloy, they found good continuity for all small angle grain boundaries, whereas for large angle boundaries, those slip bands associated with dislocations of predominantly edge character gave the best continuity. The screw dislocation slip bands were wavy and tended to spread near the grain boundaries, presumably by cross-slip. Other observations of slip band continuity across the boundaries were interpreted in terms of the stress activation of slip sources in grain boundaries [175,176].

The interactions between grain boundaries and dislocations have been studied with a variety of techniques [177–180]. For instance, Shen, Wagoner and Clark used static transmission electron microscopy and
dynamic in situ high voltage electron microscopy (HVEM) to study the behavior of the dislocations across the grain boundary [179]. Four criteria for predicting the activated slip system across from a pile-up at a grain boundary were employed to compare with the experiment. One of the criteria, for the dislocation transmitting from one grain to its neighbor grain, was the geometric factor which was originally proposed by Livingston and Chalmers [1]. Shen, Wagoner and Clark's [179—180] important criterion was to use the geometric factor to choose the emitting slip plane from the dislocation pile-up in the adjacent grain. They defined $\beta$ angle as follows: two slip planes intercept at grain boundary, $\beta$ is the angle between the two slip plane traces at the boundary. If a slip plane has a minimum $\beta$ angle, dislocations are predicted to transmit from one grain onto this slip plane. Then, the slip direction is so chosen that the glide force would be the maximum on the slip system. In their stainless steel polycrystal experimental TEM observation, the criterion worked well. Fig. 1.3. shows the definition of $\beta$ angle.

![Fig. 1.3 Intersection of slip planes and boundary [180]](image-url)
Schmid law in polycrystals

The Schmid law can be stated as follows: a single crystal yields on any particular slip system if the shear stress resolved on that slip plane and slip direction reaches a critical value, which is called the critical resolved shear stress (CRSS). In general, the Schmid law works well for single crystals. However, for polycrystals, because of the grain boundary effect, Schmid law may not be valid any more. For example, Hashimoto and Margolin studied some special information on which slip system operated and the factors affecting the onset of slip in polycrystalline α-brass [126]. They found that, of the 195 slip systems studied, 118 slip systems were strongly operated and the remainder were secondary or cross slip systems. Among these 118 slip systems only 30 had the highest Schmid factor based on the applied stress. It was inferred that, at least, Schmid factor was not the only factor affecting the operation of slip. Similar results were obtained in the shear incompatible bicrystals of Fe—3%Si [114—116] and of the shear incompatible β-brass bicrystals and tricrystals [124]. Unfortunately, from the early investigations made by Livingston [1], Chalmers [106, 107] and Suits [162] et al. the Schmid law applicability was not stated.

Stress—grain size relationship
A relationship between the grain diameter and flow stress was first proposed by Hall [3] and Petch [4], and modified into an empirical equation which assumes that the grain boundaries make the propagation of the dislocations difficult and cause them pile up behind the grain boundaries on their slip planes [164]. The Hall–Petch equation is generally expressed as follows:

\[ \sigma_y = \sigma_0 + K_y d^{-n} \quad (1.2.1) \]

where \( \sigma_y \) is the yield stress, \( \sigma_0 \) is a constant related to the contribution of the lattice friction, \( K_y \) is a constant and \( d \) is the grain diameter. \( n \) is a material parameter ranging from 1/3 to 1 for different materials.

In fact, a 1/d law was first proposed by Bragg [181] prior to the Hall–Petch equation. Kocks [136] found out that this 1/d law worked well for fine-grained material, because in fine-grained polycrystal, the internal stresses cannot be relieved by multiple slip at the grain boundaries, and higher grain boundary strength would result in flow stress increasing. Whereas for a relatively large-grained polycrystal, in which local stress concentrations can cause the generation of dislocations or cracks that spread at lower stresses, the flow stress was proportional to \( d^{-1/2} \). In fact, many experimental data favor the \( d^{-1/2} \) relation [113].

Later a more general model directly describing the relationship between the yield stress and dislocation density, rather than the yield stress and grain diameter was developed by Li [182]:

\[ \sigma_y = \sigma_0 + \alpha Gb\rho^n \quad (1.2.2) \]

where \( \sigma_y \), \( \sigma_0 \) and \( n \) have the same meanings in (1.2.1). \( \alpha \) is a
numerical constant generally between 0.3 and 0.6. $G$ is the shear modulus, $b$ is Burgers vector, and $\rho$ is the dislocation density, an inverse function of the grain size [183—184].

A grain boundary is assumed to be a barrier for dislocation motion. Dislocations would pile-up at grain boundary. At the head of such a pile-up, the stress concentration ($\tau^*$) produced by superposed stress fields of the dislocation is

$$\tau^* = n \tau$$

1.2.3

$n$ is the number of dislocations in the pile-up. From detailed calculations, $n$ is proportional to $L^{1/2}$, $L$ is proportional to $d$, and $\rho$ is proportional to $n$ [185—186]. Hence, equations 1.2.1 and 1.2.2 are equivalent.

However, experimental data did not always fall into these stress size relationships. Lucke reported that no size effect was found in aluminum crystals for grain sizes between 0.25—2.5mm [20]. Suzuki mentioned that size effect became apparent in aluminum in the case of radius less than 0.25mm [49].

Suits and Chalmers analyzed the grain size effect in silicon iron [162]. Their results showed that the average flow stress for finer grains was higher than that of coarse grain on the stress strain curves. The two stress strain curves were almost parallel to each other, i.e. the work hardening rates were similar. Upper and lower yield point phenomena
were observed in finer grain size specimen. They believed that pre—yield plastic microstrain was the main reason. For fine grains, as stress increased, more and more individual grains yielded and clusters were formed in each grain. At the upper yield point, about one percent of grains, the clusters were found to grow large enough to cross grain boundary. So the stress was released and a stress drop appeared on the stress strain curve. On the contrary, for coarse grain specimens, slip began considerably below the macroscopic yield stress, and most grains yielded at the macroscopic yield point. Therefore, no stress drop showed.

Thompson studied the grain size effect on yielding in nickel [187]. He found that a conventional Hall—Petch behavior showed for grain size larger than $1\mu m$. Below that size, yield strength was nearly independent of grain size. Thompson et al. found that any texture effect on work hardening in copper was overwhelmed by grain size effects [188,189]. The stress—strain curve of very weakly—textured copper samples whose grain size was $3.4 \mu m$ differed considerably from the Taylor—predicted [19] curve based on a $<111>$ single crystal. However, the samples with large grain size, either strongly fiber—textured with $150 \mu m$ grain size or weakly—textured cast material used in their experiment, agreed much more closely with the Taylor prediction.
Jago and Hansen analyzed grain size effects in the deformation of polycrystalline iron [190]. Similar to the silicon iron result obtained by Suits and Chalmers [162], they found that the operative mechanisms differed with grain size. The finer grain size material suffered more inhomogeneous deformation. The frequency of grain boundary crossing, caused by microscopic shear bands transferring from grain to grain, increased with strain and grain size, but finally reached a constant value for strains $\geq 0.15$. They believed that the increased activity of microscopic shear bands as deformation proceeds was associated with a reduced effectiveness of grain boundaries in blocking slip transmission.

An investigation on grain size—related microyielding was performed by Berntnall and Rostoker [191]. It was found that a grain size dependence occurred for Fe–3%Si and Ni samples, but not for pure iron.

In the study of the relation between macroscopic and microscopic strain hardening in fcc polycrystals, Kocks et al. [192] found that the rate of work—hardening depended on the number of slip systems being activated along each deformation method (i.e. tension, compression etc.); for a given number of slip systems, the hardening rate depended on the detailed crystallographic configuration of the active systems.
To explain why some materials have a strong stress and grain size relationship and some do not, two kinds of stresses were assumed [193]: one stress is the stress for dislocation generation; the other is for dislocation propagation. For materials which do not show much stress and size relationship, the stress for dislocation propagation is approximately zero. On the contrary, for materials which show strong stress and size dependency, the dislocation propagation requires large stress.

Summary of polycrystals

Dislocations were usually observed to nucleate at grain boundaries, free surfaces, point defects or other imperfections. But they could also occur within the grain without evidence of slip at grain boundaries.

In polycrystalline metals, the Schmid law worked poorly. The slip system with low Schmid factor may be activated because of the continuity requirements and sources of internal stress.

In f.c.c. metals, slip may occur not only on \{111\} plane, but also on \{100\} and \{110\} planes [197].
Grain boundaries usually act as obstacles for dislocation transmission. One of the criteria for the selection of slip system during dislocation transmission proposed by Shen, Wagoner and Clark appears to work well [178—180].

Many studies were done with focus on the relationship of stress and grain size. Although many experimental data fit $d^{-1/2}$ rule, $d^{-1}$ relationship was found to work well for small size polycrystals [136]. The hardening effect was caused by the interaction between primary slip and secondary slip which was induced at grain boundary. Some studies showed that no size effect on flow stress was observed for pure aluminum with grain sizes from 0.25mm to 2.5mm. But for aluminum with small sub-grain size, the flow stress showed an increase [195] and fell into the $d^{-1/2}$ relationships with the flow stress.

1.2.4 Aluminum

Some research on the orientation dependence of the stress strain curve for single crystals of pure aluminum has been performed [20—22]. Fig. 1.1 shows some typical stress strain curves for pure aluminum samples with different orientations from these studies.
Chalmers et al. [106,107] studied the deformation behavior of aluminum bicrystals. They found that incompatibility at grain boundary caused multiple slip. The bicrystal with one crystal surrounded by the other one showed more multiple slip at grain boundaries compared with two crystals parallel to each other, and the stress–strain curve for this surrounded bicrystal was close to that of polycrystal.

Davis et al. concentrated on the effect of orientation on the plastic deformation of aluminum single and bicrystals [22]. Their isoaxial bicrystal specimens were made by rotating one crystal 45° about the stress axis with respect to the other to make the one, two, four, six or eight slip systems initially equally favored. They found that for orientation 1, 2 and 3 where easy glide was observed in single crystals, the bicrystal curve was appreciably higher than those of single crystals. These results were similar to those observed by Kocks [136], in both <221> and <210> Al bicrystals with 45° boundaries. For orientation 5 and 7, with four and eight slip systems equally favored to operate, the bicrystal curve was close to single crystals. They believed that the influence of a 45° grain boundary could be greatly dependent on the orientation. For orientation 6 (two directions in each of three planes initially equally favored) of the 45° grain boundary bicrystal, the stress–strain curve did not show any rise relative to the corresponding single crystal. It was concluded that a 45° boundary bicrystal did not raise the stress–strain curve for the bicrystal over that of its component single crystals if two or more planes were initially equally favored.
In Elbaum's study, he found that substantial easy glide (about 0.5 percent strain) was observed only in the case of single crystals, whereas small remnants of easy glide could be detected in compatible bicrystals and no easy glide was observed in compatible tricrystals and quadrucrystals [99]. His result showed that the stress–strain curves, in the case of compatible multicrystals, was independent of the number of grain boundaries.

Stevens and Pope studied the secondary slip in impact–loaded aluminum single crystals [100]. Deformation bands without kinks were observed on the faces of the recovered samples. Secondary slip of <110> type was found to be activated to satisfy the compatibility requirement.

Aust and Chen investigated the effect of orientation difference on the plastic deformation of aluminum bicrystals (deformed in tension) [137]. The crystals were similarly oriented with respect to the specimen axis which was located near <110> in the stereographic projection. It was observed that an increase in orientation difference (the degrees of angular rotation about the specimen axis) had a marked effect on inhibiting the initial plastic deformation and on modifying the subsequent stages of the deformation. The yield stress and the rate of strain hardening increased. The "easy" glide region relating to the increased amount of double glide observed in the vicinity of the grain boundary
was found to be shortened as the orientation difference between the neighboring crystals increased. They proposed that the effect of the orientation difference might be interpreted in terms of the obstruction of dislocations at the grain boundary. The existences of two short-range forces, one related to the stress field of dislocations at the grain boundary or the system of stresses at the regions of poor fit in the boundary, and the other related to an interaction where the law of force between atoms in the slip plane changed at the grain boundary as a result of the lack of continuation of the slip system across a boundary, may prevent the leading mobile dislocations from penetrating the grain boundary barrier. No calculation was done to explain the observations. Later on, the elastic effect was treated mathematically by Wagoner [118], using anisotropic elasticity. He showed that the dislocation pile-ups can be affected by crystal anisotropy and bicrystal orientation relationship in several ways.

Davis et al. performed an investigation on the continuity of slip bands across the grain boundaries in pure aluminum bicrystals [138]. They found that small amounts of deformation caused by specimen mounting in the tensile machine could cause some "unexpected" slip near the grain boundary, interfering with subsequent slip band formation. Certain combinations of the slip systems in the two crystals were unfavorable for the continuity, and the slip continuity would be retarded if the slip developed on both sides of the boundary in such unfavorable combinations before the boundary sources were activated. An assumption
of pure shear was made in the analysis of the stress activation. The shear stress $\tau_2$ produced on the slip system 2 by a shear stress $\tau_1$ acting on the slip system 1 can be expressed as follows [138]:

$$
\tau_2 = \tau_1[(\hat{e}_1 \times \hat{e}_2)(\hat{g}_1 \times \hat{g}_2) + (\hat{e}_1 \times \hat{g}_2)(\hat{e}_2 \times \hat{g}_1)] = N\tau_1 \quad (1.2.4)
$$

where $e$ is the slip plane normal and $g$ is the slip direction. A high value for $N$ would be associated with good slip continuity.

Miura and Saeki investigated the effect of the grain boundary on the plastic properties of $<100>$ aluminum bicrystals with symmetric tilt boundaries of $4^\circ$, $14^\circ$ and $37^\circ$ [139]. They found that the fine multiple slip which appeared in the early stage of deformation was suppressed at the grain boundary. It was proposed that at grain boundaries, the clustered slip accompanying prominent cross slip could pass grain boundaries having a small misorientation. For the large misorientation grain boundaries ($37^\circ$), however, the clustered slip was not able to pass and additional multiple slip was introduced at the grain boundaries. Those induced slip systems could be inferred by the combination of the Schmid factor and geometry factor of individual slip in the two crystals.

Miura and his co—workers did some analyses on aluminum bicrystals having different tilt coincidence boundaries [140,141]. They found that the $<111>$ oriented bicrystal deformed similarly to the $<111>$ oriented aluminum single crystals. The yield stress of the bicrystal was
greatly affected by interactions between the primary slip and secondary slip in each grain. They believed that the effect of the coincidence grain boundary on the flow stress was largely due to the multiple slip introduced in the vicinity of the grain boundary to form obstacles such as sessile dislocations by slip interactions. The unusual slip plane (100) was observed once at 473°K [140].

Miura used three kinds of aluminum bicrystals to study the effect of grain boundary on the plastic deformation of bicrystals in tension [142]. He found that additional slip on {110} planes was activated in the [100] oriented crystal and the flow stress was higher in the bicrystal composed of a crystal for single glide and a crystal with [100] tensile orientation. For the bicrystal of single glide and with [111] tensile orientation, a few slip clusters were observed near the grain boundary in the [111] orientation crystal and the flow stress was smaller than that of the corresponding single crystals. The flow stress was found to be approximately the same in the bicrystal composed of a crystal with [100] tensile orientation and that with [111] tensile orientation as that of the corresponding single crystals. These results were similar to those observed by Davis et al. [22].

Rey and Zaoui studied the slip heterogeneities in deformed pure aluminum bicrystals with Σ=5, Σ=7, Σ=25 and Σ=37 tilt coincidence grain boundaries [122]. They found that the heterogeneity which occurred at the beginning of plastic deformation in each component of
the bicrystals either vanished or not as deformation increased according to the specimen orientation. They assumed that two different plastically deformed areas might exist simultaneously because of the appearance of a new interface inside each component that was caused by the primary slip impeded at the grain boundary and this was the reason of formation of the new intergranular incompatibilities which must be taken into account when calculating the internal stresses.

Fleischer and Backofen [194] studied mechanical behavior of Al single crystal, bicrystal and polycrystal deformed in tension at different temperatures. It was found that the easy glide stage I in single crystal was short or absent in the bicrystal, and disappeared in the polycrystal stress—strain curve. Polycrystals started to deform in a manner similar to that of single crystal in stage II. Approximately the same work—hardening rates were observed for single crystals, bicrystals and polycrystals (corresponding to that of stage II) at low temperature. For bicrystals, at 4.2\(^{\circ}\)K, no stage III was observed. Only at 77\(^{\circ}\)K and 295\(^{\circ}\)K, were transitions observed from stage II to stage III.

The effect of the grain boundary on deformation in aluminum polycrystals was studied by Elbaum [198]. It was found that the plastic deformation relied upon the existence of the grain boundaries which can contribute to the necessary multiple slip. He supposed that the stress—strain relationship of a crystal depended on the number and kind of operating slip systems, regardless of the way in which these slip
systems were made to operate (geometry, orientation, grain boundaries, and so forth).

Summary of aluminum

The influence of grain orientation dependence of stress strain curves for pure aluminum single crystals has been well investigated [20—22]. However, the grain boundary effect for aluminum has not been understood clearly yet. Some grain boundaries exhibited a hardening effect, but some grain boundaries even showed a decrease in flow stress [111]. The strengthening effect was believed to be caused mainly by the interaction between primary slip and secondary slip [139]. As the misorientation between the two grains increased, more multiple slip was observed.

The stress strain relationship for a bicrystal with one grain surrounded by the other grain was found to be similar to that of polycrystals [107]. More secondary slip was observed in this kind of bicrystal than that of the corresponding bicrystal with two of the grains parallel to each other.

The grain boundary hardening effects of bicrystals were studied by Davis, Livingston, Chalmers [22] and Miura [142]. Similar results were observed. For the bicrystals with initially one favored slip plane in each component, the flow stress is higher than that for the corresponding
single crystals, and the flow stress did not show any increase for the bicrystals with initially more than one favored slip plane in each component compared to the corresponding single crystals. The stress strain curve for polycrystals was found to be independent of the number of grain boundaries [99,198]. No size effect was observed on the stress strain curve for aluminum from size of 0.25mm to 2.5mm. It was also found that electro—polishing might lead to a drop in flow stress of the strain strain curve of an aluminum specimen.

In the study of mechanical behavior of Al single crystal, bicrystal and polycrystal deformed in tension [194], similar work—hardening rates were obtained for single crystal, bicrystal and polycrystals at low temperature (corresponding to that of stage II in single crystal).

One study showed that non—\{111\}<110> slip was observed in impact—load aluminum single crystal [100]. The slip was believed to be more compatible with deformation slip bands. Some "unexpected" slip was observed at a grain boundary that was favorable for slip continuity [136]. It was found that electro—polishing could affect the flow stress greatly in stage I and II by removing the surface hardened layer [32,56].
1.2.5 Stress Analysis

Mathematical models

Many models have been developed to predict both quantitative and qualitative properties of the elastic-plastic behavior of polycrystalline metals [143—157]. For example, one of the first theories was proposed by Sachs [143]. In his model, it was assumed that all grains had the same stress; an assumption which automatically violated the compatibility condition. The texture was due only to the orientation of each constituent grain linked to boundary conditions applied to it. The dislocation interaction and the stress caused incompatibility at grain boundary were neglected. The model was found to predict rolling texture of brass well.

Taylor [19], on the contrary, considered identical deformation of each grain; an assumption that violated the equilibrium condition and hence prevented strain heterogeneities. It was found that Taylor’s model could predict texture well at medium strain for material having small plastic anisotropy and strain heterogeneity. For large grain size materials, the prediction of the model showed a much closer result [188—189]. But, at large strain, a large deviation was found between the predicted and measured textures.
Taylor's model was tested by Honda and Hosckawa using aluminum and low carbon steel [196]. They found that although the stress in each grain depended on its orientation, the stress was unloaded uniformly in each grain. Therefore, a uniform local stress model could be applied to both loading and unloading processes in the case of elastic deformation. They indicated that the materials they used could be approximately predicted by Taylor's model in the initial stage of plastic deformation. However, the elastic and plastic transition was not taken into account in their model. So the correctness of their extrapolation from elastic to plastic deformation is very limited.

Taylor's model was extended by Bishop and Hill for the yielding of a polycrystal [144]. They assumed that the only mechanism of plastic distortion in a single crystal was by glide parallel to preferred planes and directions. They proved the hypothesis suggested by Taylor [19] by introducing two principles for a single crystal: Maximum Work Principle and Minimum Shear Principle.

Hill showed that an upper bound was given for elastic moduli of the polycrystal if compatibility conditions were fulfilled (although equilibrium conditions may be violated), whereas a lower bound was given if the equilibrium conditions were fulfilled (although compatibility conditions may be violated) [145]. It was found that for realistic degrees of anisotropy, the arithmetic average between these two bounds was often a sufficiently accurate solution to the problem of an elastic polycrystal.
Budiansky & Wu later developed a model, the so called "self-consistent" method, in which the grain interaction was taken into account [147]. The model was not limited to very small strains. Some calculations were performed by Budiansky, Wu, and Hutchinson [148-149]. It was found that the model could predict the yield surface well, and provided very similar results to Taylor's models [19].

A so called "relaxed constraints" theory was proposed by Kocks and Canova [150]. In this model, the elasticity was neglected, certain strain compatibility components and other equilibrium stress components were enforced. It was equivalent to the large strain limit which brought about the same result as rolling. But it lacked elastic-plastic transition and it had difficulty in predicting slip systems. The advantage of the model was to apply to large deformation fast and easily as long as the grains had more or less the same shape as the overall sample. However, it was not appropriate for uniaxial compression for f.c.c material because of some grain curling.

Iwakuma and Nasser recently applied Hill's self-consistent method to the finite elastic-plastic deformation of a polycrystalline solid [155]. They reported that the model could predict Bauschinger effect, hardening, formation of a vertex or corner on the yield surface for both microscopic non-hardening and hardening crystals. Nasser and Obata used the same
method to analyze rate—dependent finite elastic plastic deformation of polycrystals [156]. They found that Taylor's averaging scheme provided an accurate estimate of the incremental quantities at large strains, and the total overall quantities differed largely from the one obtained by the self—consistent method.

Finite element simulation

There is no doubt that the existence of the grain boundary is unfavorable to the motion of the dislocation at low temperature, and consequently the dislocation pile—ups cause a stress concentration at a grain boundary, which therefore results in the material to show an increment in the flow stress in the process of the deformation of a polycrystal. Not only does the obstacle impede dislocations, it causes multiple local slip and dislocation interaction such that the regions near grain boundaries are harder than the grain interiors. However, in order to correctly interpret the real mechanisms of grain boundary which affects the deformation and attempt to do predictive analysis, it is evident that the means of analysis must be raised from experimental level to a theoretical one. Since the grain boundary is the major concern in the analyses, Finite Element Method (FEM) is selected as a proper numerical means for interpreting the experimental observations.
FEM simulation for single crystal and bicrystal

Miyamoto et al. [200,201] created a model based on dislocation theory to analyze the elastic and plastic deformation of Al single crystal using FEM. For the first stage work—hardening of Al, their FEM model predicted close $\sigma-\varepsilon$ relationships with those of experimental ones [200]. But the FEM did not work well for the specimen with tensile axis at the corner of the standard triangle. No experiment was done for the tensile specimen with tensile axis sitting at the symmetry boundary of the standard triangle. By considering the interactions between the slip systems, their model improved for the specimens with the tensile axes at the corner of the standard triangle. Further Miyamoto et al. used a FEM model to estimate whether secondary slip was active or not for aluminum single crystal [201]. It was found that secondary slip occurred near the stress concentration region close to grip. Their calculation of stress–strain curve showed a close match with the experimental result. The latent hardening of aluminum was lower than that of copper. But they did not mention the relationship between the Schmid law and the secondary slip systems activated. Their FEM calculation only showed where the secondary slip began, but not which secondary slip system to operate.

A FEM analysis of ductile single crystal deformation behavior was done by Peirce, Asaro and Needlemen [202]. They presented the first full solution to a boundary value problem for a non–homogeneous
deformation elastic—plastic single crystal. Their single crystal constitutive law agreed with that of Hill and Rice [157]. The flow rule was based on the Schmid CRSS law and accounted for both self hardening and latent hardening of the slip system. They found that Taylor's isotropic hardening model was not appropriate because of the initial thickness was not uniform, heterogeneous slip occurred. Their FEM calculation showed the importance of continuum kinematics in the development of nonuniform and localized deformation in ductile single crystals. And it also showed a good qualitative and quantitative agreement between the experiments and calculations.

Kitagawa et al. used isoparametric elements to perform an elastic stress analysis on two types of isoaxial bicrystals, a normal strain incompatible bicrystal and a shear strain incompatible bicrystal containing a planer grain boundary parallel with tensile axis [203]. The material was body centered cubic type alloy ($\beta$ phase Cu—Zn—Al alloy) with high anisotropy factor. It was found that the stress concentration at the grain boundary was three times higher than the average stress for a single crystal in the normal strain incompatible bicrystal and four times higher than in shear strain incompatible bicrystal. It was also found that the affected zone of the grain boundary on the stress distribution did not spread widely (one tenth of the grain width) from the grain boundary. It was evident that the slip behavior at the early stage of yielding depended not only on the type of incompatibility at the grain boundary but also on the dimensional ratio of the cross section of the
bicrystals. The role of the elastic interaction stress was believed to be important in yielding of polycrystalline metals with high anisotropy factor. The role of grain aspect ratio is also the basis for the relaxed constraints model.

Baxter and Wang did an analysis on the FEM calculation of the deformation of a persistent slip band [204]. Their proposed model was composed of two phases: a much softer phase in the persistent slip band, surrounded by a matrix phase. The dislocations were assumed to be emitted from the tip of the persistent slip band into the matrix by precipitate dissolution followed by solute transport. And the two phases were assumed to deform independently. The two stress-strain relationships were given as follows:

\[
\sigma(m) = 276 + 44 \varepsilon^{0.11} \quad \text{MPa} \quad (1.2.5)
\]

which represented the strong 6061-T6 aluminum and

\[
\sigma(psb) = 55 + 97 \varepsilon^{0.22} \quad \text{MPa} \quad (1.2.6)
\]

which corresponded to that of 1100 aluminum. The FEM simulations were made for plane stress conditions with the finite element program ABAQUS. It was found that the strains in the persistent slip band were up to a hundred times larger than the strain in the matrix during the first load cycle. The calculated strain distribution demonstrated the existence of a plastic zone in the matrix material at the tip of a persistent slip band. It was believed that this plastic zone was an
essential condition for the elongation of a persistent slip band. They indicated that the hardening of a persistent slip band was very important in controlling its rate of elongation, and this persistent slip band was not uniform along its length.

Ohashi [205] and Wagoner [206] did 3-D FEM simulations for the compression experiment results of Fe–3%Si bicrystal obtained by Hook and Hirth [114–116] respectively. Ohashi analyzed three out of the nine bicrystals with isoaxial bicrystals. In his elastic anisotropic model, 175 elements were used with finer mesh at grain boundary. For the "pseudo–identical" bicrystal, the RSS's were calculated for primary slip and secondary slip systems whose Schmid factor were in first and second ranking, respectively. The RSS for the secondary slip was observed to be higher than that for the primary slip at the grain boundary in both components. He found that the gradient of stress was affected by the width, thickness and length ratio, and the non–diagonal terms in the elastic matrix showed a bigger effect on the elastic incompatibility stress distribution. He mentioned that, in the early stage of deformation (less than about 5% strain), the slip process strongly depended on the elastic stress field, and the initial distribution of dislocations played an important role in forming multi–slip systems along the grain boundary.
In the experiments made by Hook and Hirth [114—116], the slip systems observed at front and back surfaces were different. However, in Ohashi's simulation [205], only the slip systems with the secondary Schmid factor show higher RSS at the grain boundary and no distribution was made between crystal surfaces. Wagoner et al. [206] performed an anisotropic 3—D FEM analysis using 500 elements with a finer mesh at grain boundary and perfect friction at the ends of the specimen which could predict the activated slip systems at both front and back surfaces consistently with the experiment. They found that the resolved shear stress on each potential slip system of $\{111\} \{110\}$ type was determined by the stress distribution from the applied axial strain, the frictional end constraints and the elastic incompatibility stresses at the grain boundary. For the isoaxial bicrystals, it was found that the secondary slip had the highest resolved shear stress at some regions of the grain boundaries in the front or back surfaces; corresponding to different specimens, although those secondary slip systems had the second largest Schmid factor. This may be the explanation for why Hook and Hirth observed secondary slip occurring at the positions where no primary slip was observed. On surfaces of the specimen, the observed secondary slip system was different from the first one. Here the secondary slip had the third largest Schmid factor. In FEM calculation, on these surfaces, the RSS’s for the secondary slip systems had #2 ranking, while the primary slip was the same in bulk materials. For nonisoaxial bicrystals, their results of FEM simulation showed that the activated secondary slip systems, for the (001) tensile axis grain, were different
from the front surface to the back surface of the crystal. The slip systems with the maximum calculated RSS matched the results observed from the experiments on each surface. It was found that the friction at the compression interfaces enhanced secondary slip. A comparison between the results of FEM simulation and experiments on a pure aluminum tricrystal was made, and it showed good agreement with FEM predicted and experimental results. Similar to the result of Kitagawa's bicrystal simulation [203], the RSS at the grain boundary increased about 20% to 30% compared with that of single crystal. And the high stress region did not extend far into the grain (about one tenth of the grain width). Later, Wagoner et al. extended their study on deformation from elastic to elastic—plastic. First of all, they did a trial elastic calculation to get the stresses in each element in each component of the bicrystal. Secondly, they use a CRSS yield criterion in each finite element to check which elements yielded. Thirdly, they applied a typical constitutive law for Fe–3%Si to those yielded elements. Steps two and three were repeatedly performed for the large incremental boundary displacements and updated yield stresses. Their simulation showed that the yield could begin at grain boundary or the corner of the specimen in one crystal and then propagate to other crystal as the displacement increased. Rounded elastic—plastic transitions were observed for bicrystals simulated in this way.
In general, it was found that Wagoner's FEM model [206] could work very well on the predictions of primary slip, secondary slip, elastic incompatibility slip and secondary activated slip. Since the numerical calculations were done under the elastic assumptions, Wagoner indicated that the second order slip caused by plastic incompatibility could not be predicted well by FEM, and the micro plastic effects such as dislocation nucleation could not be represented by the current continuum FEM. However these effects appeared to apply to smaller volumes of material rather than the long range stress induced slip. A surprising success of the FEM prediction was in secondary slip related to plastic incompatibility.

FEM for polycrystals

Miyamoto et al. used FEM to estimate the mechanical behavior of coarse grained pure aluminum polycrystal [207]. Some assumptions were made for estimating the location and the direction of the slip lines which initially appeared on the surface of their polycrystal sample: the FEM calculation was an elastic one, and the grain boundary was the one at which only the orientations of two adjacent grains were different. The specimen was divided into triangular prism elements following the geometry of the grain boundaries. The FEM simulation showed the heterogeneous stress distribution which was consistent with the slip trace density results. But the operating slip systems were not mentioned in either experiment result or FEM calculation.
A model for predicting the grain size effect on the yield stress of polycrystalline metals was proposed by Meyers and Ashworth [208]. In their paper, they discussed the process of yielding in three stages. First of all, the elastic incompatibility stresses arising from the elastic anisotropy of adjacent grains established localized stress concentrations at the grain boundaries, and secondly, localized plastic flow defined as microyield resulted from those stress concentrations. At the third stage, when the applied stress made the grain boundary film reach its flow stress level, macroyielding occurred. Some assumptions were also made for his modeling analysis: the stress acting on all the grains was assumed to be the same, and the grains were classified into "rubber" type grains and "steel" type grains. In other words, all the individual grains did not undergo the same strain as the tensile specimen did. They concluded that the elastic anisotropy stresses exceeded the flow stress of the grain boundary region before the polycrystalline aggregate was able to deform plastically and a work-hardening layer of grain boundary was formed in a continuous network pattern relieving the elastic incompatibility stresses. Their FEM predictions were seemingly in good agreement with the experimental results.

Hashimoto and Margolin performed a numerical calculation of resolved shear stresses (RSS) using 3-D elastic FEM [209]. They observed that the RSS was not uniformly distributed within the grains, and the predicted slip system based on the highest total RSS was operative in the 9 of the 11 grains of a polycrystalline α-brass.
specimen. Because the RSS's for the two slip systems in the other two grains respectively, were too close, it was impossible to tell which one of them would operate first. Some low RSS slip systems operated were found not to form strong obstacles with the primary slip system. It was believed that the elastic interaction stresses played an important role in the yield process and in strains of at least several percent.

Nagashima et al. used two—dimensional FEM to study the effect of oriented grain arrangement on the elastic modulus of polycrystalline sheet metals [210]. The iron specimens were simulated by an aggregate of triangular finite elements, and the values of the stiffness of each cubic crystal about its local axes were assumed to be the same as those of the single crystals of pure iron: $C_{11}=237$ GPa, $C_{12}=141$ GPa and $C_{44}=116$ GPa. Four kinds of models were proposed to calculate Young's modulus. The models were composed of two sorts of fiber texture having the same volume ratio but different arrangements. It was found that the values of Young's moduli varied by the arrangement of the grains which had different orientations. The author suggested that taking the distribution of the oriented grains and the volume fraction of each texture component of the grains into account was important in the estimation of the elastic behavior of polycrystalline metals.

In order to analyze on the local elastic stress in the vicinity of grain boundary junctions, Ohashi established a multicrystal model containing some grain boundary planes and their junctions such as triple
lines and quadruple points [211,212]. The magnitude of local elastic stress was described in terms of the elastic anisotropy of the material and geometrical arrangement of the grain boundary planes. The geometrical structure of the model containing five grains had three-fold symmetry with regard to the y axis, and all the angles between the remaining six grain boundary planes were defined to have the same value \( \beta \). The finite elements facing a grain boundary plane were connected to the neighboring elements in the next grain in the same manner that the elements inside a grain connect. The assumptions for this model were that each crystal grain was elastically isotropic and homogeneous, and that Young's modulus and Poisson's ratio were the same in every finite element. Young's modulus in any arbitrary direction \( (\alpha \beta \gamma) \) with respect to three [100] axes of a cubic crystal could be given as follows:

\[
1/E(\alpha \beta \gamma) = S_{11} - 2(S_{11} - S_{12} - S_{44}/2)(\alpha^2 \beta^2 + \beta^2 \gamma^2 + \gamma^2 \alpha^2)
\]

(1.2.7)

And the magnitude of elastic anisotropy was measured by following equation:

\[
A = 2( S_{11} - S_{12} ) / S_{44}
\]

(1.2.8)

The results of the analytical simulation showed that the magnitude of the local stress was largely dependent on both the shape of the grains in the multicrystal and the combination of the elastic constants of each grain. It was found that at the quadruple points, changing the angle \( \beta \) between the two grain boundary planes significantly changed the stress value. No stress concentration was exhibited when
\( \beta = 90^\circ \), but it became more and more significant while \( \beta \) was getting larger. It was concluded that the stress concentration in the vicinity of triple lines or quadruple points of the grain boundaries would increase if there are larger differences in the elastic constants and the sharper the angle between the two grain boundary planes. The stress field, in the vicinity of the quadruple points, was found to exhibit a multiaxial character.

Abe, Nagaki and Furuno analyzed plastic deformation and Taylor slip in f.c.c. polycrystals using the rigid—plastic finite element method [213]. The principle of the maximum plastic work introduced by Bishop and Hill was employed to select five independent slip systems out of the twelve possible slip systems. Their result showed that heterogeneous plastic deformation occurred in particular region, and the operating slip system changed near grain boundary due to the multiaxial deformation. Grain shape had an influence on the deformation behavior. Unfortunately, no experimental results were available for comparing their simulation.

Several stress analyses were done for texture materials [214—218]. For example, Asaro and Needleman proposed a rate dependent constitutive model for polycrystals subjected to arbitrary strains [214,215]. The results showed that large strain hardening rates and strain rate sensitivity both increased the failure strains on the slip system level, and the texture had a strong influence on the localized necking. Dawson et al.
used FEM simulation to analyze the mechanical properties of aluminum polycrystals [214—217]. It was found that the increases in hardness and the temperature rises were both larger for greater reduction or lower initial temperature [216]. The friction condition was important to increase the hardness. By comparing relative changes in hardness and the yield strength for the rolled products, the model seemed to predict good results. For the formation of crystallographic texture for rolling, extrusion and drawing, Dawson et al. [217] developed a mathematical formulation, and applied it numerically using FEM to the forming process. As a result, the predicted deformation texture showed good correlation with reported experimental results. Crites et al. investigated the crystal slip of small strain yield behavior in textured copper tubing using a self-consistent method [218]. It was found that the Bishop—Hill [144] model poorly represented the texture effect, whereas Kroner, Budiansky and Wu's [147] model gave accurate predictions for biaxial yield surfaces at small strain levels (about $10^{-4}$).

Summary of FEM

The deformation behavior of single crystals was analyzed using FEM [200—202]. Miyamto et al. [200,201] experimentally and numerically analyzed aluminum single crystals deformed in tension. Work—hardening behavior in stage I was predicted well for the specimen with the tensile axis at the center of the standard triangle [200]. Secondary slip was observed and predicted by their FEM at the region close to grip [201].
However, the prediction of which secondary slip system to operate was not mentioned. Peirce et al. [202] found their single crystal constitutive law agreed with that of Hill and Rice [157]. The calculation showed the dependence of nonuniform and localized deformation in ductile single crystals. It also showed good qualitative and quantitative agreement between experiment and calculations.

The deformation behavior for bicrystals or two phase composites was studied using FEM by many investigators [203—206]. The bicrystals with the grain boundary parallel to the load axis were either deformed in tension or compression. Because of the incompatibilities at grain boundary, all these calculations showed stress concentration at grain boundary, and the stress can be up to four times higher than the average stress in the corresponding single crystals. From both Wagoner's [206] and Ohashi's [205] simulation of the Hook and Hirth [114—116] experiment bicrystal results, it was possible that the secondary slip (Schmid factor ranking in the second) had a highest RSS at grain boundary, and the stress concentration decreased rapidly away from the boundary. Wagoner's FEM simulation also showed the consistent RSS rankings for different secondary slip at front and back surfaces in each crystal component as the experiment did, and surprisingly, the FEM predicted well for some secondary slip related to plastic incompatibility. The critical resolved shear stress criterion seemed work well.
In the FEM simulation for deformation of persistent slip bands [204], Baxter and Wang found that the strains in the persistent slip band were up to 100 times larger than the strain in the matrix during first load cycle, and the persistent slip band was not uniform along its length.

For polycrystals, many simulations have shown heterogeneous stress distributions [207—218]. In Miyamoto's analysis, the heterogeneous stress distribution was consistent with the slip trace density distribution. A model for predicting the grain size effect on the yield stress of polycrystalline metals was proposed by Meyers and Ashworth [208]. They concluded that the elastic anisotropy stresses exceeded the flow stress of the grain boundary region before the polycrystalline aggregate was able to deform plastically and a work—hardening layer at the grain boundary was formed in a continuous network pattern, relieving the elastic incompatibility stresses.

Margolin et al. performed a numerical calculation of RSS for a multicrystal using elastic FEM [209]. The predicted slip system based on the highest RSS was operative in the 9 of the 11 grains. They indicated that the elastic interaction stress played an important role in the yield process for at least several percent strain. The geometry and grain orientation were found important to the local stress concentration [211—212]. It was concluded that the stress concentration in the vicinity of triple lines or quadruple points of the grain boundaries would be
higher if the difference in elastic constants is bigger and the angle between the two grain boundary planes is sharper.

For the analysis of texture formation during deformation, Asaro et al. [214—215] found that texture had a strong influence on the localized necking. Dawson et al. [216—217] found that in Al polycrystals (deformed in rolling), friction, reduction and temperature were all important in hardness, and the predicted texture showed good correlation with reported experimental results. Crites et al. [218] found that for small grain yield behavior, the model of Kroner, Budiansky and Wu gave an accurate prediction for biaxial yield surfaces at small strain levels ($10^{-6}$).

In general, it is believed that FEM simulations can provide very good solutions for the deformation behavior of single crystals, bicrystals and polycrystals. The degree of agreement depends on the assumptions made for each particular FEM model.
CHAPTER II

EXPERIMENTAL PROCEDURES

All the bicrystal, tricrystal and polycrystal tensile specimens were obtained from pure aluminum by the strain annealing method [219]. Most grain orientations were determined by X-ray Laue back reflection. The grain boundary structures were calculated.

Aluminum tensile specimens were deformed from 1% up to 7% strain in one to three steps at room temperature. Slip observations were made using an optical microscope after deformation at each step.

2.1 Preparation of bicrystals, tricrystals and polycrystals

The specimens used were made of pure aluminum (99.99%) sheets which were in hot rolling state. The aluminum sheets were cut parallel to the rolling direction into strips about 20cm to 25cm long by 3cm to 4cm wide to make sample plates. In order to remove the retained stress, these sample plates were annealed at 600°C for a week. Etching in a solution of 1 part HCl, 1 part H₂O, and 1 part HNO₃ plus several drops of HF acid [220], revealed several larger grains which came
from the secondary recrystallization. However, these grains were not large enough to make full size bicrystal or tricrystal tensile specimens. So, some tested samples were cold rolled from 2% up to 5% strain to obtain even larger grains, but these grains did not gain size. Some other samples were subjected to about two percent tensile strain, annealed at 600°C for seven to ten days, and then were etched in the same solution mentioned above. Large grains appeared and could be clearly seen by the naked eye. The sizes of some grains could reach from 30mm to 90mm long by 15mm to 30mm wide through the sample’s thickness. Certain sample plates even became a single crystal. Bicrystal, tricrystal and polycrystal tensile specimens were cut from these annealed aluminum plates in a rectangular shape at sizes of 30mm to 60mm long and 7mm to 15mm wide. The tensile specimens were again annealed at 250°C for 3 hours to relieve the stresses caused by cutting. Fig. 2.1 is a micrograph of the strain—annealed aluminum plate from which the B specimen was cut.

Fig. 2.1 Strain annealed Al plate B
Mechanical polishing to 1/4 μm diamond paste on the specimens was done before electro-polishing. Electro-polishing was first carried out in a solution of 20% perchloric acid plus 80% ethanol at 0°C [221]. Since perchloric acid is very explosive, the solution composed of only 20% perchloric acid plus 80% ethanol could be on fire even in the ice bath because of its high volatility. So methanol was used instead at 0°C. The electro-polishing current varied from 1 ampere to 2 amperes and the voltage was kept at about 15 volts. After electro-polishing, the thickness of the aluminum specimens was approximately 3.3mm.

2.2 Labelling of specimens

Five original large sample plates cut from aluminum sheets for strain annealing were labeled as plate B, C, D, E and F. For instance, specimens labeled with B were cut from plate B, specimens labeled with C were cut from plate C, and so on. All the bicrystal, tricrystal and polycrystal specimens were cut from these five sample plates and labeled with a number from 1 to 5. For instance, four tensile specimens cut from plate C were labelled as C1, C2, C3 and C4. In each tensile specimen, there were from 1 to 6 grains. These grains were arbitrarily named as grain a, grain b, or grain c, and so forth. If two tensile specimens contained grains which came from the same original grain, for example, if two specimens both had part of grain a, the grains in these two specimens were called C1a and C2a.
Sample plate B had only one specimen named B which contained three grains. Fig. 2.2 is a schematic plot of the strain annealed aluminum sample plate B.

Four specimens were cut from sample plate C, Fig. 2.3. Specimens C1 and C2 were oriented the same, except for the orientation of the grain boundary between grains a and b. There was a small grain c sitting in the C1a grain. Specimens C3 and C4 were the same, except for the orientation of small internal grains. The tensile axes of C1 and C2 were 18 degrees twisted around the plane normal X axis from C3 and C4 grains. Grains c, d and e had different orientations.

Sample plate D had two specimens D1 and D2, Fig. 2.4. D1c was a small grain at the corner of the grain D1a. D1d was a small half grain inside the large grain D1a.

There were five specimens cut from sample plate E which contained mainly two large grains, a and b, Fig. 2.5. Specimen E1 had two grains E1a and E1b. Specimen E2 had a small grain E2c at the boundary between grains E1b and E1a. Specimen E3 contained grains E3b and E3a. Specimens E4 and E5 were two single crystals with two different orientations in grain b. Specimens E2, E3 and E4 were cut parallel to each other, as were specimens E1 and E5. The tensile axes of the specimens E2—E4 were rotated by 47 degrees relative to the tensile axes of specimens E1 and E5.
Fig. 2.2 The schematic plot of strain annealed B plate
Fig. 2.3 The schematic plot of strain annealed C plate
Fig. 2.4 The schematic plot of strain annealed D plate
Fig. 2.5 The schematic plot of strain annealed E plate
Sample plate F had only one tensile specimen F containing six grains from Fa to Ff, Fig. 2.6. The orientations of these grains were independent of each other. Fb was a small grain sitting in the large grain Fa. Fc and Fd were two small grains residing at the boundary between the two large grains Fa and Fe.

2.3 Determination of orientations

The orientations of each grain were determined either by SEM electron-channeling or by X-ray Laue back reflection.

1). For electron-channeling, the surface condition of the sample is very important. It should be absolutely deformation-free. Even soft paper tissue can damage the specimen's surface. Before putting the sample into the SEM JEOL 35A, an intensive electro-polishing was performed. In order to obtain crystal orientations, the image mode was selected with the lowest magnification (20X, assume no rotation under 20X)) to take the ordinary surface morphology micrograph to determine the relationship between the channeling pattern and edge of the specimen, as the channeling pattern's were taken.

Fig. 2.7(a) is a picture of an electron channeling pattern for grain Bc. First of all, the pole was indexed as the (T12) pole. Then the surface normal of the specimen was achieved by using a computer
program, Polesearch [219]. Before starting the Polesearch program, each line on the pattern should be identified. By applying the rule of the f.c.c structure factor, the pole and the lines were distinguished. Fig. 2.7(b) is a schematic plot of (T12) pattern. Each pair of the lines were indexed as shown in Fig. 2.7(b).

The purpose of using Polesearch program is to find the center point of the picture which is normally assumed to be the electron beam direction. Knowing the relationship between the center point and the reference direction, along with the camera length for each line, the surface normals for the three grains in specimen B were obtained. The camera length $L$ is given as Eq. 2.1 [221]:

$$L = \left( \frac{a \times R}{(h^2 + k^2 + l^2)^{0.5} \times \lambda} \right)$$

where $a$ is lattice parameter, $R$ is the distance between the two parallel lines, $\lambda$ is the wavelength of the electron beam, and $h, k, l$, are the indices of each line.

Using an image, the angle between the specimen edge and the pair of channeling line can be found. Assuming that $Z$ is the specimen tensile axis, $n$ is the surface normal, $I$ is one of the channeling line directions, the $Z$ direction was identified by solving the following
Fig. 2.6 The schematic plot of strain annealed F plate
Fig. 2.7(a) Electron channeling pattern for grain Ba
Schematic plot of orientation determination
equations:

\[ \mathbf{Z} \cdot \mathbf{n} = 0 \quad 2.3 \]
\[ \mathbf{Z} \cdot \mathbf{I} = \cos \theta \quad 2.4 \]

where \( \theta \) is the angle between \( \mathbf{Z} \) and \( \mathbf{I} \). See Fig. 2.7(b). Since the beam direction is usually a few degrees off the pole direction and the indices of the poles are integers, the pole directions were used instead of the beam directions for this grain. According to the channeling patterns, the coordinate axes and the tensile axis of the three grains were determined with respect to the global coordinates. The global coordinates were the same for the three grains, but the local coordinates were different. Only the coordinates for each grain in specimen B were determined using this method.

2). For obtaining a X—rays Laue pattern, the requirement for the surface condition is not as critical as that for electron—channeling pattern. But a clean and undeformed surface is still required [224]. Another advantage of using X—rays is that the orientations of large specimens can be obtained without damaging the specimens. But it is impossible for SEM—35A to index the orientation of a small grain inside a large one without cutting the specimen. Since the sample holder is very small, about 1cm in diameter, the specimen must be cut small enough to fit in it. Normally, a X—ray Laue pattern can provide the same accuracy [219] as electro—channeling. (Error analysis is shown in
appendix A). All the grain orientations of specimens were analyzed by X-ray Laue back reflection method, except specimen B.

To determine the crystal orientation from X-ray Laue pattern, the following steps were taken: first of all, the Laue spots were transferred from the negative film into a stereodiagram by Graniger chart. Then a big Laue spot, which several parabolic Laue spot curves intercept it, was found (probably low index spot). The stereodiagram was rotated so that this big spot was on the center of the stereodiagram, and other spots were rotated correspondingly. This stereodiagram was compared with several standard pole projection diagrams. If all the spots coincided with one of these standard projection diagrams and the angles between each spot were the same as those in the standard projection diagram, then the center point must be indexed the same as the pole in the standard projection diagram. All the other spots were indexed also (with accuracy of ± 2°). Thus, the crystal orientation can be determined. For detail procedure of orientation determination, see 'Elements of X-ray diffraction', [224].

All of the grain orientations determined by these two methods are shown in the appendix A.
2.4 Grain boundary structures

Grain boundary structures (local crystallography, defect structures) can affect the transmission of slip in nearby regions. In particular, special boundaries which have collinear slip directions or high symmetry to slip may promote easy slip transmission [179—180]. Other special boundary configurations include CSL’s (coincident lattices) [177—180]. In order to understand whether the grain boundary possesses one or more of these properties, some matrix transformations were performed. Since the orientation for each grain is fixed and known, the grain boundary description can be found through the following procedures:

Define three base vectors (global) along long axis (Z), width (Y) and thickness (X) (right hand system). Let the components of these three vectors be (1,0,0), (0,1,0) and (0,0,1) in the global coordinate system and (a,b,c), (d,e,f) and (g,h,i) in local coordinate system defined such that $\bar{e}_1$ is parallel to $<1,0,0>$, $\bar{e}_2$ is parallel to $<0,1,0>$, and $\bar{e}_3$ is parallel to $<0,0,1>$. Define a coordinate rotation matrix that transfers the components for the fixed vectors specified in local coordinate (a,b,c), (d,e,f) and (g,h,i) to components in the global coordinates (1,0,0), (0,1,0) and (0,0,1) as follows: Fig. 2.8.

\[ \{I\} = \{R\} \{X\} \quad 2.4\]
\[
\begin{bmatrix}
1 & 0 & 0 \\
0 & 1 & 0 \\
0 & 0 & 1 \\
\end{bmatrix}
= \begin{bmatrix}
R_{11} & R_{12} & R_{13} \\
R_{21} & R_{22} & R_{23} \\
R_{31} & R_{32} & R_{33} \\
\end{bmatrix}
\begin{bmatrix}
a & d & g \\
b & e & h \\
c & f & i \\
\end{bmatrix}
\]

Therefore, \( R \) is an orthogonal 3X3 matrix which has each line corresponding to the local component of the global base vector. For the \( i^{th} \) grain or \( i^{th} \) local coordinate system, the corresponding rotation matrix is \( R_i \), and \( X_i \) is the matrix containing three base vectors in the local coordinate system. Then we have:

\[
I = R_i \ast X_i 
\]

and

\[
I = R_j \ast X_j 
\]

Fig. 2.8 Relationship between local and global coordinate systems
Since the global coordinate system for each grain is the same, the following can be deduced:

\[ R_i \ast X_i = R_j \ast X_j \quad 2.8 \]

\[ X_i = R_i^{-1} \ast R_j \ast X_j = R' \ast X_j \quad 2.9 \]

\[ R' = R_j^{-1} \ast R_j \quad 2.10 \]

where \( R' \) is the rotation matrix from grain \( i \) to grain \( j \). Because a cubic crystal can be represented 24 equivalent ways, the rotation matrix with the minimum rotation angle was used to describe the grain boundary. Comparing with the M13 table [226], the grain boundary structure was shown as \( \Sigma \) number in terms of rotation matrix \( R \) by choosing the rotation angle and the axis closest to the rotation angle and the axis of the experimental matrix \( R' \). Usually the actual boundaries are not exactly coincidence boundaries. The matrix \( R^{02} \) was defined as the difference between rotation matrices \( R \) and \( R' \). The matrix \( R^{02} \) was calculated as follows:

\[ R = R^{02} \ast R' \quad 2.11 \]

\[ R^{02} = R \ast R'^{-1} \quad 2.12 \]

\[ \theta = \arccos \left( \frac{\text{tr}(R^{02}) - 1}{2} \right) \quad 2.13 \]

\[ h : k : l = (A_{32} - A_{23}) : (A_{13} - A_{31}) : (A_{21} - A_{12}) \quad 2.14 \]
where \( \text{tr}(R^{02}) \) is the sum of the diagonal elements of the \( R^{02} \) matrix. \( A_{ij} \) (\( i=1,3, \ j=1,3 \)) are the elements of the \( R^{02} \) matrix. The angle \( \theta \) obtained from \( R^{02} \) is the angle difference between the two rotation axes of matrices \( R \) and \( R' \).

The minimum angle between the grain boundary rotation axis and grain surface normal were also calculated. All the grain boundary geometric descriptions are shown in the appendix B. No tilt boundary was found since no tilt angle (around the surface normal) was less than 5 degrees.

2.5 Deformation and slip analysis procedure

After electro—polishing, specimens were deformed from 1% to 7% strain in tension, at a cross head speed of about \( 2 \times 10^{-2} \text{mm/s} \) on an Instron testing machine at room temperature. In the first step, at a strain of about 1%, the dislocation etch—pits and grain boundary could be seen after being etched in 50% HCL, 47% HNO\(_3\), and 3% HF for 2 or 3 seconds. The slip bands appeared as strain increased. They could be seen even by the naked eye. The etch—pit method was used only for specimen sets B and C after 1% strain. The rest of specimens were analyzed directly (i.e. without etching) under the optical microscope after deformation, because it was very difficult to identify the operating slip system from etch—pits.
Specimens in set B and set C were deformed in two steps. At the first step, the specimen was deformed to about one percent strain followed by etch—pit analysis. Then these specimens were subjected to an additional 6% strain followed by direct slip trace analysis.

To remove the residual stress caused by cutting, specimens cut from sample plates D, E, and F were first annealed at 150°C for four hours. No new grains were observed after this low—temperature annealing. These specimens were then directly deformed to about 4% strain. In order to observe how dislocation nucleation and propagation occurred, some of the specimens were annealed again at 200°C for four days to remove the previous dislocations. The orientation for each grain was assumed to be unchanged. Specimen D1, E2 and E3 were deformed sequentially to one percent of strain at step one and to an additional one percent of strain at step two after taking micrographs. The experimental procedures for each specimen are showed in Table 1.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Step I</th>
<th>Step II</th>
<th>Step III</th>
</tr>
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<tbody>
<tr>
<td>B</td>
<td>1% etch-pits</td>
<td>7% slip</td>
<td></td>
</tr>
<tr>
<td></td>
<td>polishing</td>
<td>trace</td>
<td></td>
</tr>
<tr>
<td>C1-C4</td>
<td>1% etch-pits</td>
<td>6% slip</td>
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<tr>
<td></td>
<td>polishing</td>
<td>trace</td>
<td></td>
</tr>
<tr>
<td>D1,E2,E3</td>
<td>4% annealing</td>
<td>1%</td>
<td>2% all slip trace</td>
</tr>
<tr>
<td></td>
<td>&amp; polishing</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D2,E1,E5,F</td>
<td>4% all slip</td>
<td></td>
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<td></td>
<td>trace</td>
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<tr>
<td>E4</td>
<td>8% slip</td>
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</tbody>
</table>
For the operated slip system identification, several methods were used to assure the correctness of the results. First, the angles between the slip traces at the specimen's surface and load axis Z were measured. The angles between the intercept of each {111} plane with the surface and Z direction were calculated. The slip planes were identified by comparing the calculated results with the measured ones. Secondly, the angles between the slip traces were compared with the calculated angles. The angle between the traces should match the calculated results if the labels of the slip plane were right. Thirdly, the pole of the {111} type of slip plane was figured by using a stereogram. The sample surface pole was rotated to the center of the stereogram. The (111) pole, for example, was put on the equator line. On the great circle at the south pole or north pole, (defined as A), pole A was the direction perpendicular to both (111) plane and surface. A and the surface normal pole (center point Px) were connected. The line A—Px was the slip trace of (111) plane on the specimen surface. Similarly, all other {111} type slip traces were drawn in this way. Knowing the load direction on the stereogram, which allows superposition of the diagram on the specimen, slip planes were easily identified. If the results provided by these three methods were the same, the slip planes had correctly been identified.
2.6 Difficulties of the study:

a). It is still not possible to identify the slip directions just by analyzing the slip traces or slip bands on the surface of specimen. Only the slip planes can be determined uniquely.

b). It is impossible to observe the nucleation of dislocation in pure aluminum and to identify the slip planes directly from isolated etch pits. The slip traces or bands appears at about 1% strain.

c). For large size specimens it is difficult to obtain and to maintain a good electro-polishing surface. The reaction between the specimen and electro-polishing solution may create some pitting lines on the specimen surface that may be confused with slip traces. Once the polishing is done, the surface of the specimen would be contaminated quickly for Al. And room temperature recovery may occur after deformation.

d). For small grains, because of the limitation of the x-ray beam size, it is very difficult to obtain a X-ray Laue pattern for grain size less than 4mm.
2.7 Approach for overcoming the difficulties:

For each known \{111\} type slip plane, the slip direction was assumed to be the one with the largest RSS from our FEM calculations.

Defining where the dislocation nucleation started was difficult because controlling the strain to observe only the initial few dislocation etch—pits was very subtle. For instance, at one percent of stain, the dislocation etch—pits could be found everywhere. So a slip trace analysis was involved. The slip trace observations were recorded after 1%, 2% and 4% strains for D and E set of specimens. By tracing back along the slip plane, the dislocations were assumed to originate at the place where the first few slip traces were found.

To obtain good quality polishing surfaces of large size specimens, low polishing current was used. Once the polishing was done, the specimens were deformed and the slip traces were recorded immediately.
CHAPTER III

FINITE ELEMENT MODELING

The finite element method (FEM) is a general technique for constructing approximate solutions to boundary value problems. The method involves dividing the domain of the solution into a finite number of simple subdomains called finite elements, and using variational formulations to construct an approximation of a solution. All of our FEM calculations were performed using a FEM software package, ABAQUS provided by HIBBIT. KARLSSON and SORENSEN Company [227].

The effects of grain boundaries are complicated because the grain boundary structures are various in kind, and their configurations are dynamic during deformation. In our case, the key point is how to treat the behavior of grain boundaries. Since the mechanical behavior of grain boundaries during deformation has not been understood clearly yet, some assumptions were made in our FEM analysis:
1) Anisotropic elastic continuum FEM was used. In other words, strictly speaking, the results are not precise after yielding, or after first slip line appears.

2) At the grain boundary, only the orientations of two adjacent grains were considered to be different. The mechanical behavior of the grains in bicrystals, tricrystals and polycrystals were assumed to be like those of a single crystal with the same orientation (i.e. the grain boundary was treated as a mathematical surface across which equilibrium and compatibility, in the continuum sense, were enforced). Local grain boundary structure was not considered.

3) Displacements were continuous at grain boundary.

4) In each grain, the material was assumed to be homogeneous. Voids and impurities were not considered.

5) Slip system ranking (i.e. prediction of slip system activation) was based on the critical resolved shear stress criterion.

A three dimensional model with eight-node isoparametric elements was employed in the current FEM simulation. Different meshes were generated for different specimens. After the boundary position had been found, different material properties (i.e., different elastic constants) were applied to different grains. By using an anisotropic model, the
elastic moduli were adopted as $C_{11}=108.2$ GPA, $C_{12}=61.3$ GPA, $C_{44}=28.5$ GPA for Al [228]. Since the transformation matrix is a 3 by 3 matrix, matrix transformations were first done to obtain a 6 by 6 elastic constant matrix which is required for ABAQUS data input. Equation 3.1 shows that the local coordinate system which coincides with the axes of a cubic unit cell of an arbitrary grain in the specimen is related with the global coordinates system of the specimen,

$$\{I\} = \{R_i\} \{X_i\} \quad 3.1$$

where $R_i$ a is rotation matrix which rotates a vector in the local coordinate system ($i^{th}$ grain) to global coordinate system.

The relation between the stress $\{\sigma\}$ and strain $\{\epsilon\}$ in the standard coordinate system defined by the conventional cube symmetric axes is as follows in Eq. 3.2, [213]:

$$\sigma = \begin{bmatrix} \sigma_x \\ \sigma_y \\ \sigma_z \\ \tau_{yz} \\ \tau_{zx} \\ \tau_{xy} \end{bmatrix} = \begin{bmatrix} c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & c_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & c_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & c_{44} \end{bmatrix} \begin{bmatrix} \epsilon_x \\ \epsilon_y \\ \epsilon_z \\ 2\epsilon_{yx} \\ 2\epsilon_{zx} \\ 2\epsilon_{xy} \end{bmatrix}$$

$$\sigma = \{\epsilon\} \{\epsilon\} \quad 3.2$$
where \( c_{ij} \) \((i=1,6, j=1,6)\) are the elastic constants. Equation 3.2 is valid only for cubic systems. In the new coordinates, the stress \( \{\sigma'\} \) can be written as a function of \( \{\sigma\} \) in Eq. 3.3:

\[
\{\sigma'\} = \{R\}^T \{\sigma\} \{R\} \\
\text{or} \\
\sigma'_{ij} = R_{ik} R_{j1} \sigma_{k1}
\]

The elastic constants in the new coordinates can be calculated by Eq. 3.5:

\[
C'_{ijkl} = R_{im} R_{jn} R_{ko} R_{lp} C_{mnop}
\]

Where \( C_{mnop} \) refer to the elastic constants in the standard 3 by 3 by 3 by 3 scheme rather than the 6X6 scheme shown in eq. 3.2.

The boundary conditions were chosen according to the experiments. Since the upper holder of the Instron test machine is fixed, the displacements in the X, Y and Z directions of the bottom surface of the specimen were chosen as zero, Fig. 3.1. The displacement of the nodes at the top surface of the specimen in Z direction was one percent of the length of each specimen, whereas, the displacements in X and Y directions were both zero. Because it was an elastic simulation, the calculated stresses are relative to the prescribed displacement.
The stress calculation was done in three dimensions. Eight node elements with isoparametric interpolation functions were selected [230]. Different element and node numbers were chosen for different size specimens. To exhibit a better grain boundary effect, finer meshes were applied at grain boundaries for the flat boundaries. Although using elements with large width and length ratios usually causes inaccuracy in the long dimension side, this is not a serious inaccuracy in our current FEM simulation. Along the grain boundary, stress did not vary rapidly, whereas, perpendicular to the grain boundary plane, stress changed rapidly [230]. Since the specimens were very thin relative to their other dimensions, one layer of elements was used in the X direction. The grain boundaries were assumed to be straight through the thickness direction. For numerical integration, eight Gauss points were used [230]. The stress and strain at each Gauss point were directly determined by running ABAQUS on a VAX 8550 or CRAY super computer.

A program was written to resolve the stress tensor onto the twelve possible slip systems for face centered cubic crystals in each element, at each Gauss point. Because the transformation matrix \( \{R^{-1}\} \) is the rotation matrix from the global coordinates to each local crystal coordinates, and the transformation matrix \( \{T\}_s \) is the rotation matrix from local crystal coordinates to the coordinates composed of 12 slip systems \( (s=1,12) \), so the transformation matrix \( \{A\} \) can be derived as following:
PLEASE NOTE:

Page(s) not included with original material and unavailable from author or university. Filmed as received.
\{A\} = \{T\}_s \ast \{R\}^{-1} = \{T\}_s \ast \{R\}^T \quad 3.6

\begin{align*}
\text{Crystal Coordinates System} & \quad \xrightarrow{T_s} \quad \text{Slip System Coordinates} \\
R & \quad \uparrow R^{-1} & \quad \uparrow A = T_s \ast R^{-1} = T_s \ast R^T
\end{align*}

Global Coordinates System

Fig. 3.2 Schematic plot of matrix transformation.

The stress in the slip system coordinates \{S'\} can therefore be written as a function of the stress \{S\} in the global coordinates:

\{S'\} = \{A\}\{S\}\{A\}^T \quad 3.7

\begin{align*}
&= \{T\}_s \{R\}^T \{S\} \{R\}^T \{T\}_s^T \\
&= \{T\}_s \{R\}^T \{S\} \{R\} \{T\}_s^T \quad 3.8
\end{align*}
The stress \{S\} was directly calculated from ABAQUS. There are 12 possible slip systems in f.c.c metals, therefore, these 12 slip systems can form 12 \{\mathbf{T}\}_s matrices. In our calculations, for example, for slip system (111)[101], the slip plane normal [111] was chosen as \(X_1\) axis, the slip direction [101] as \(Y_1\) axis. The \(Z_1\) axis was the cross product of \(X_1\) and \(Y_1\), which was [121]. The remainder \{\mathbf{T}\}_s matrices were calculated similarly. With this choice of slip system coordinate system, the resolved shear stress on each slip system was \(\sigma_{12}\). The slip systems were ranked from largest to smallest based on the resolved shear stress on each slip system. Hence, it was easy to distinguish which slip system was predicted to operate. The maximum resolved shear stress \(\sigma_{12\text{max}}\) for each element in each crystal was plotted. (Note: the slip system corresponding to \(\sigma_{12\text{max}}\) is usually different in each crystal because the orientation for each grain is different, and it can also be different within each grain because of the grain boundary effect or grip effect etc.).

It is worth mentioning that some non-{111} slip systems were observed in some specimens. The RSS for these slip systems were also calculated for the use of slip system ranking.
CHAPTER IV

EXPERIMENTAL AND FEM CALCULATION RESULTS

In this chapter, experimental results and FEM calculations will be presented and the comparisons between them will be discussed. The chapter is divided into five sections corresponding to five sample plates. For example, the results and comparison for sample plate B are in section 4.1, the results and comparison for sample plate C in section 4.2, and so on.

There are several kinds of plot in this chapter. The first plot is the schematic plot of the slip result after each deformation. Second is a contour plot of the FEM—calculated the maximum resolved shear stress (RSS) throughout. The number on the contour is in arbitrary units, with large number representing high RSS value. Since the contours are based on the highest RSS acting on any slip system at each point, the map represents different slip systems at different locations. The third plot is a plot showing the RSS ranking for each slip system which observed in experiment. In fcc metals, there are
twelve possible slip systems. In the FEM simulation, the RSS on all the 12 slip systems were calculated. Because in the experiment, only the slip plane can be identified, the slip system was chosen as the slip direction which maximized the RSS on the slip plane. Hence, the slip system ranking reduced from twelve to four based only on slip planes, and the slip system was chosen such that for this slip system had the highest RSS value. For example, B₄(231) means slip trace B ranking 4th in RSS, and with RSS value at that position is 231 (corresponding to 1% strain from elastic FEM calculation). Again, the stress value is in arbitrary unit. If there are no subscript number, it means that the slip systems are in sequential order from RSS large to small. If there is an unusual slip (defined as slip plane other than {111} type), the slip system can be more than four. The last kind of plot is the FEM mesh for simulation.

There are three kinds of plots for the relations of true stress $\sigma$, and true strain $\varepsilon$. First is a plot of true stress—true strain curves. In this kind of plot, the solid lines are the experiment results. The dotted lines are the smoothed $\sigma$—$\varepsilon$ curves for the use of calculation of $\ln\sigma$ and $\ln\varepsilon$. The second kind of plots are the curves of $\ln\sigma$—$\ln\varepsilon$. The last one is the plot of work—hardening rate $n$ ($n=d\ln\sigma/d\ln\varepsilon$) versus true strain $\varepsilon$ (ignore the elastic strain).

The schematic plot for each sample plate, Fig. 2.2—2.6 will be repeated in each sample set.
4.1 Sample Set B

Sample plate B had only one specimen, B, with two grain boundary planes roughly perpendicular to the load axis, Fig. 2.2.

Fig. 2.2 Schematic plot of strain-annealed plate B
Fig. 4.1(a) is a schematic plot of the slip results after 7% strain and the trace analysis. Fig. 4.1(b) is a contour plot of the FEM calculated maximum resolved shear stress (RSS) throughout the specimen. The slip system with the highest RSS at each point is shown in Fig. 4.1(c). The details of the observed slip systems and their FEM results are shown in Table 2. Fig. 4.2(a) is a typical micrograph at the grain boundary between grain a- and grain b (for short: GBab) after 1% strain and etching. Fig. 4.2(b) is the micrograph of the same region as Fig. 4.2(a) after additional 6% strain without etching. The etch—pits were the previous ones which were not polished out.

Results and Comparison

After 1% strain, the densities of dislocation etch pits were high at the high RSS regions, Fig. 4.1(b) and Fig. 4.2(a). The average the dislocation density was high in grain b as well as the average RSS. Along the two grain boundaries, dislocation density changed consistently with FEM prediction.

After 7% strain, slip trace densities were high corresponding to the high RSS region. Also, secondary slip was observed at these high stress regions, Fig. 4.2(b). In grain a (Ga) away from the GBab, the maximum RSS contour reached the maximum and very fine slip traces were observed. The secondary slip system B was observed there, too. At GBab near +Y edge, slip system B with the secondary Schmid factor
Fig. 4.1(a) Schematic plot of slip trace on B after 7% strain
4.1(b) FEM-calculated maximum RSS plot for specimen B
4.1(c) FEM-calculated RSS ranking for the observed slip in B
Fig. 4.2(a) Typical micrograph at GBab after 1% strain, 400X
4.2(b) Typical micrograph at same place at 7% strain, 100X
Table 2. The Schmid factor and RSS ranking in specimen B for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ba</td>
<td>A(111)[110]</td>
<td>1, 0.483</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at Uc+y, +yg</td>
</tr>
<tr>
<td></td>
<td>B(111)[110]</td>
<td>#2, 0.453</td>
<td>G, hdr</td>
<td>#2 throughout, except #1 at Uc+y, +yg</td>
</tr>
<tr>
<td>Bb</td>
<td>A(111)[101]</td>
<td>#1, 0.428</td>
<td>Bulk</td>
<td>#1 throughout</td>
</tr>
<tr>
<td></td>
<td>B(111)[011]</td>
<td>#2, 0.324</td>
<td>Bulk</td>
<td>#2 throughout</td>
</tr>
<tr>
<td>Bc</td>
<td>A(111)[101]</td>
<td>#2, 0.409</td>
<td>Bulk</td>
<td>#1 at -yg large area #2 else</td>
</tr>
<tr>
<td></td>
<td>B(111)[101]</td>
<td>#1, 0.414</td>
<td>hdr</td>
<td>#1 at +yg, far from G</td>
</tr>
</tbody>
</table>

Key: U = Upper end  
L = Lower end  
G = Grain boundary  
E = Edge  
sb = Slip band  
hdr = Highly deformed region  
e.g.: +yg = grain boundary at +Y edge
had the highest RSS ranking from the FEM simulation, Fig. 4.1(c). In grain b (for short: Gb), since the difference of Schmid factor between slip systems A and B was small, there was no RSS ranking change, Fig. 4.1(c). In grain c (Gc), the observed slip traces were mostly of type A, although the Schmid factor for A ranked second. Few slip traces for B were observed in the highly deformed region, Fig. 4.1(a). The FEM calculation showed RSS in a large area for slip system A ranking in the first, and in the rest places, slip system B was of the highest RSS, Fig. 4.1(c). In general, after 7% strain, the densities of slip traces changed consistently with the FEM calculation at grain boundaries, and the densities for secondary slip sites were high at grain boundaries. But the average slip trace density was high in grains Ga and Gc where localized slip was observed.

4.2 Sample Set C

There were four specimens cut from sample plate C, Fig. 2.3. The deformation procedure was similar to that of specimen B.

4.2.1 Specimen C1

In specimen C1 there were three grains, Fig. 2.3. The grain boundary between the two big grains a and b was inclined 60 degrees to the load axis. There was a small grain C1c grain inside grain C1a.
Fig. 4.3(a) is a schematic plot of the slip results after 6% strain and the trace analysis. Fig. 4.3(b) is a plot of the FEM—calculated maximum RSS of specimen C1. Fig. 4.3(c) is a plot of FEM—calculated observed slip system ranking in each grain. The details of the observed slip systems and their FEM results are shown in Table 3.

Fig. 4.4(a) is the typical micrograph at grain boundary between the big grain C1a and the small grain C1c after 1% strain. Fig. 4.4(b) is the micrograph after 6% strain at the same region as Fig. 4.4(a).

**Fig. 2.3 Schematic plot of strain-annealed plate C**
Specimen Cl

Fig. 4.3(a) Schematic plot of slip trace on Cl after 6% strain
4.3(b) FEM-calculated maximum RSS plot for specimen Cl
4.3(c) FEM-calculated RSS ranking for the observed slip in Cl
Fig. 4.4(a) Typical micrograph at GBac after 1% strain, 400X
4.4(b) Typical micrograph at same place at 6% strain, 100X
Table 3. The Schmid factor and RSS ranking in specimen C1 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[011]</td>
<td>#2, 0.481</td>
<td>Bulk</td>
<td>#2 all</td>
</tr>
<tr>
<td>C1c</td>
<td>#1, 0.495</td>
<td>-yc</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>#3, 0.347</td>
<td>-ylc</td>
<td>#3 all</td>
</tr>
<tr>
<td>A(111)[011]</td>
<td>#1, 0.451</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at L</td>
</tr>
<tr>
<td>C1a</td>
<td>#4, 0.04</td>
<td>-yc</td>
<td>#4 throughout, except #3 at L</td>
</tr>
<tr>
<td>A(111)[110]</td>
<td>#1, 0.497</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td>C1b</td>
<td>#2, 0.453</td>
<td>G</td>
<td>#2 all</td>
</tr>
<tr>
<td></td>
<td>#4, 0.207</td>
<td>hdr</td>
<td>#4 all</td>
</tr>
</tbody>
</table>

Key: U = Upper end  
L = Lower end  
Y = +Y edge  
- Y = -Y edge  
G = Grain boundary  
E = Edge  
sb = Slip band  
hdr = Highly deformed region  
e.g.: -ylc = lower end -Y corner

Fig. 4.5 FEM mesh for specimen C1
Fig. 4.6(a)  Refined FEM mesh around the small grain C1c
(b)  FEM-calculated maximum RSS contour for fine mesh C1
Results and Comparison

After 1% strain, it seemed that dislocations nucleated quite randomly in the whole specimen. In some regions, dislocations were residing at the grain boundaries, Fig. 4.4(a). Not many etch pits were exhibited in both grains C1c and C1a and these etch pits were not connected.

After 6% strain, slip bands were seen clearly by naked eye. Two or three slip systems were observed in each grain. Most of them were of high Schmid factors and RSS's. In the small grain C1c, slip trace A occupied the whole grain, Fig. 4.3(a). Although slip system B had the largest Schmid factor, it was not observed in the whole grain. Only at the grain boundary between grains C1a and C1c (GBac) where slip system C (111) was observed in grain C1a, slip trace C was observed near the corner of the grain C1c, Fig. 4.3(a). The FEM simulation for the small grain basically showed the same result as Schmid factor did, Table 3.

For grain C1a, slip system A was the primary slip as the FEM predicted, Table 3. Slip trace C was also observed. But the Schmid factor and RSS for slip trace C were much less than that of slip system A, although the FEM calculation showed a relative increase in RSS around the small grain C1c. Slip trace B in grain C1c was connected
with slip trace C in grain C1a. The continuity occurred at the small
grain corner with grain boundary normal 39 degrees to +Z direction,
appendix C.

In grain C1b, three slip systems A, B and C operated. Slip
system A, with the largest Schmid factor and RSS, was observed
throughout the grain. The density of slip trace A reached the maximum
at the region halfway between the grain boundary and the Z=1 end of
the specimen where the FEM simulation showed the maximum RSS, Fig.
4.3(b). Slip system B with the secondary highest Schmid factor was
observed only at GBab on +Y side, Fig. 4.3(b). Slip system C, with
the Schmid factor and RSS ranking 4th, originated at the high density
slip band A, Fig. 4.3(a), where the FEM result showed the highest RSS
region.

After the first FEM calculation, the FEM mesh was refined at
grain C1c, Fig. 4.6(a). Much thinner elements were used along the
grain boundary. Boundary conditions were chosen from the previous
FEM calculation with the same displacements at the same boundary
nodes. The FEM—calculated RSS showed increasing at elements along
the grain boundary, Fig. 4.6(b). There was no slip system ranking order
change. The comparison of the RSS for the observed slip in two kind
of meshes is shown in Table 4.
In general, the slip band density in grain C1a was relatively lower than that in grain C1b which was consistent with the FEM prediction.

Table 4. The comparison of FEM-calculated RSS for each slip in different meshes in specimen C1

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>RSS in coarse mesh at GBac</th>
<th>RSS in fine mesh at GBac</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1c</td>
<td>A(111)[011]</td>
<td>352</td>
<td>362</td>
</tr>
<tr>
<td></td>
<td>B(111)[011]</td>
<td>362</td>
<td>375</td>
</tr>
<tr>
<td></td>
<td>C(111)[110]</td>
<td>252</td>
<td>244</td>
</tr>
<tr>
<td>C1a</td>
<td>A(111)[110]</td>
<td>331</td>
<td>342</td>
</tr>
<tr>
<td></td>
<td>C(111)[101]</td>
<td>30</td>
<td>32</td>
</tr>
</tbody>
</table>

Here, the numbers are the RSS values in arbitrary unit.
4.2.2 Specimen C2

In specimen C2, there were two grains a and b, Fig. 2.3. The grain boundary between the two grains was inclined 45 degrees to the load axis.

Fig. 4.7(a) is the schematic plot of the slip results after 6% strain and the trace analysis. Fig. 4.7(b) is a contour plot of the FEM-calculated maximum RSS throughout the specimen C2. The slip system with the highest RSS at each point is shown in Fig. 4.7(c). The details of the observed slip systems and the FEM results are shown in Table 5.

Fig. 4.8(a) is the micrograph at slip bands where the unusual slip D (101) originated (will be discussed in Chapter V). Fig. 4.8(b) is the micrograph of the highest slip trace density region. Fig. 4.8(c) is the typical micrograph at GBab on +Y side after 6% strain.

Results and Comparison

Since grain C2a and C2b are orientated the same as grains C1a and C1b, the slip systems operated were presumed the same. Indeed, in grain C2b, the same slip systems A, B and C were observed, Fig. 4.7(a). The only difference was their locations. There was another slip trace D with slip plane (101) originating at the high primary slip bands
Specimen C2

Fig. 4.7(a) Schematic plot of slip trace on C2 after 6% strain
4.7(b) FEM-calculated maximum RSS plot for specimen C2
4.7(c) FEM-calculated RSS ranking for the observed slip in C2
Fig. 4.8(a) Micrograph where slip D observed, 100X
Fig. 4.8(b) Micrograph at high slip band density, 100X
Fig. 4.8(c) Micrograph at grain boundary, 100X
Table 5: The Schmid factor and RSS ranking in specimen C2 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>C2a</td>
<td>A(111)[011]</td>
<td>#1, 0.451</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>B(110)[110]</td>
<td>#4, 0.221</td>
<td>+yg</td>
<td>#4' throughout, except #3' at L-y</td>
</tr>
<tr>
<td>C2b</td>
<td>A(111)[110]</td>
<td>#1, 0.497</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[110]</td>
<td>#2, 0.453</td>
<td>+yg, hdr</td>
<td>#2 all</td>
</tr>
<tr>
<td></td>
<td>C(111)[101]</td>
<td>#5, 0.207</td>
<td>U</td>
<td>#5 all</td>
</tr>
<tr>
<td></td>
<td>D(101)[101]</td>
<td>#4', 0.343</td>
<td>sb</td>
<td>#4' throughout, except #3' at U</td>
</tr>
</tbody>
</table>

Here 4' means slip systems ranking include {110} slip plane.

Key:
- U = Upper end
- L = Lower end
- G = Grain boundary
- E = Edge
- +Y = +Y edge
- -Y = -Y edge
- sb = Slip band
- hdr = Highly deformed region
- e.g.: +yg = grain boundary at +Y edge

Fig. 4.9 FEM mesh for specimen C2
density place, Fig. 4.8(a). Although slip trace D (101) was not \{111\} type, the RSS for D was high and ranked 4th.

From the FEM calculation, it was found that the highest RSS region was away from the grain boundary, Fig. 4.7(b), which was consistent with the experiment results, Fig. 4.7(a). Fig. 4.8(b) is the micrograph corresponding to the FEM highest RSS region. The spacing between slip traces reached the minimum. Cross slip was observed quite commonly in the specimen. At the grain boundary, Fig. 4.8(c), the slip trace density was obviously lower than that in Fig. 4.8(b).

In grain C2a, primary slip A took place as expected because slip system A had the highest Schmid factor and RSS, Table 5. No slip system C was observed since no small grain was inside the grain C2a. However, slip system B occurred surprisingly, because slip plane B was \{110\} type, and it was observed only at +Y edge of the specimen.

In general in specimen C2, slip band densities were high at the high RSS regions. But the maximum RSS region was away from the grain boundary. Our experimental result showed the same profile.
4.2.3 Specimen C3

In specimen C3, there were two grains with one small grain C3d inside the big grain C3b, Fig. 2.3.

Fig. 4.10(a) is a schematic plot of the slip results after 6% strain and the trace analysis. Fig. 4.10(b) is a contour plot of the FEM-calculated maximum RSS throughout the specimen. Fig. 4.10(c) is the FEM-calculated slip system ranking for each observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 6.

Fig. 4.11(a) is the micrograph at the grain boundary region where slip continuity occurred after 6% strain. Fig. 4.11(b) is the micrograph along the grain boundary at the region next to one shown in Fig. 4.11(a).

Results and Comparison

In the small grain C3d, only slip system A operated. Since slip trace A had the highest Schmid factor and RSS, the slip system A was expected.
Specimen C3

(a)

(b)

(c)

Fig. 4.10(a) Schematic plot of slip trace on C3 after 6% strain
4.10(b) FEM-calculated maximum RSS plot for specimen C3
4.10(c) FEM-calculated RSS ranking for the observed slip in C3
Table 6. The Schmid factor and RSS ranking in specimen C3 for observed slip systems

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>C3d</td>
<td>A(111)[110]</td>
<td>#1, 0.486</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>A(111)[110]</td>
<td>#1, 0.481</td>
<td>Around C3d grain</td>
<td>#1 throughout, except #2 or #3 at L-y, U+y</td>
</tr>
<tr>
<td>C3b</td>
<td>B(111)[110]</td>
<td>#2, 0.449</td>
<td>Bulk</td>
<td>#2 throughout, except #1 at U+y, L-y</td>
</tr>
<tr>
<td></td>
<td>D(111)[101]</td>
<td>#3, 0.414</td>
<td>Connected A in C3d</td>
<td>#3 throughout, except #1 at U-y, L+y</td>
</tr>
</tbody>
</table>

Key: U = Upper end, L = Lower end, G = Grain boundary, E = edge, +Y = +Y edge, -Y = -Y edge, sb = Slip band, hdr = Highly deformed region

Fig. 4.12 FEM mesh for specimen C3
In grain C3b, slip traces A, B and D were observed on the specimen surface. Although FEM simulation predicted that trace A had the highest Schmid factor and RSS, Table 6, slip system A did not look like primary slip and it was observed only around the small grain. It seemed that slip trace A was restricted by the small grain. On the other hand, slip system B with the second highest Schmid factor and RSS (at most places) was observed to occupy a large area of the grain. Slip system D, with the third highest Schmid factor and RSS (at most places), was observed around the small grain too. It was found that slip trace D in grain C3b was connected with the slip trace A in the small grain, at grain boundary normal 48° to Z direction, Fig. 4.11(a), appendix C. The continuity decreased and finally disappeared (at grain boundary normal less than about 20° to the Z direction). Fig. 4.11(b) is the micrograph along the grain boundary adjacent to Fig. 4.11(a). But the slip traces A and D were not connected. In fact, slip trace D vanished in grain C1b.

In general, the FEM calculation showed the higher RSS around the small grain. In the experiment, the slip trace densities were high around the small grain too.
4.2.4 Specimen C4

Specimen C4 had two grains with one small grain C3e inside the big grain C3b, Fig. 2.3.

Fig. 4.13(a) is a schematic plot of the slip results after 6% strain and the trace analysis. Fig. 4.13(b) is a contour plot of the FEM—calculated the maximum RSS throughout the specimen. Fig. 4.13(c) is a plot of FEM—calculated slip system ranking for the observed slip in each grain. The details of the observed slip systems and their FEM results are shown in Table 7.

Fig. 4.14(a) is the micrograph of etch pits at grain boundary (GBbe) after 1% strain. Fig. 4.14(b) is the micrograph in the same region as Fig. 4.14(a) after 6% strain where slip trace continuity was observed. Fig. 4.14(c) is the micrograph at Z=0 end of the specimen.

Results and Comparison

After 1% strain, the dislocation etch pits were quite uniform, except at the certain places along the grain boundary, Fig.4.14(a), where more etch pits showed.
Specimen C4

Schematic plot of slip trace on C4 after 6% strain

FEM-calculated maximum RSS plot for specimen C4

FEM-calculated RSS ranking for the observed slip in C4
Table 7. The Schmid factor and RSS ranking in specimen C4 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[011]</td>
<td>#1, 0.453 Bulk</td>
<td>#1 all</td>
<td></td>
</tr>
<tr>
<td>C4e B(111)[011]</td>
<td>#4, 0.054 Connected C in C4b</td>
<td>#4 all</td>
<td></td>
</tr>
<tr>
<td>C(111)[011]</td>
<td>#3, 0.163 Connected A in C4b</td>
<td>#3 all</td>
<td></td>
</tr>
<tr>
<td>A(111)[110]</td>
<td>#1, 0.481 Around C3e</td>
<td>#1 throughout, except #2 or #3 at L, U</td>
<td></td>
</tr>
<tr>
<td>C4b B(111)[110]</td>
<td>#2, 0.449 Bulk</td>
<td>#2 throughout, except #1 at U+y, L-y</td>
<td></td>
</tr>
<tr>
<td>C(010)[101]</td>
<td>#5', 0.127 Connected B in C4e</td>
<td>#5' all</td>
<td></td>
</tr>
<tr>
<td>D(111)[101]</td>
<td>#3, 0.414 Connected A in C4e</td>
<td>#3 throughout, except #1 at U-y, L+y</td>
<td></td>
</tr>
<tr>
<td>E(111)[101]</td>
<td>#4, 0.267 L</td>
<td>#4 throughout, except #3 at L</td>
<td></td>
</tr>
</tbody>
</table>

Here 5' means slip system ranking include {010} slip plane.

Key: U = Upper end +Y = +Y edge
L = Lower end -Y = -Y edge
G = Grain boundary sb = Slip band
E = Edge hdr = Highly deformed region
e.g.: +yg = grain boundary at +Y edge

Fig. 4.15 FEM mesh for specimen C4
After about 6% strain, three slip systems were observed in the small grain, Table 7. Slip traces A with the highest Schmid factor and RSS was observed all over the grain. The slip systems C and B, having the 3rd and 4th Schmid factor respectively, were activated too. They were found to have connections with two other slip traces in the big grain C4b. Although the FEM calculation showed the same slip system ranking as that of Schmid law, the FEM simulation did show a relative increase in RSS for the two slip systems at the grain boundary where high continuity occurred, Fig. 4.13(c).

In C4b grain, there were five slip traces observed. Because the maximum number of \{111\} type slip traces which can be observed on specimen's surface is four, there must be at least one slip system which is not \{111\} type of slip planes. It was found that slip system C slipped on plane (010). Since C4b had the same orientation as grain C3b, slip A, B and D were the same as those in grain C3b. The difference was that two more slip traces C and E occurred at certain regions, Fig. 4.13(a). Slip traces A, C and D were observed continuously to cross the grain boundary to the small grain. Again, the angle $\beta$ between the two slip plane intercepts at grain boundary was calculated for all these three pairs of slip. One of the slip pair had the minimum $\beta$ angle at the position where slip continuity occurred, (appendix C). If the angle $\beta$ is very small at certain grain boundary location, it implies that dislocations could easily cross over the grain.
boundary from one grain to another. Only could small range of grain boundary satisfy the criterion [177–180]. Fig. 4.14(b) is a micrograph at the grain boundary where most slip traces were observed. Slip system E was observed at Z=0 end of the specimen. The Schmid factor and RSS were ranked 4th at most places, except at the place where slip trace E was observed, the RSS ranking for slip system E shifted from the 4th to the 3rd, and RSS for E increased relatively, Fig. 4.13(c). The RSS ranking for slip system B shifted to the first at the same region as slip E shifted to 3rd, Table 7. There was no primary slip trace observed at that region, even though the RSS for primary slip A was still ranked second.

The maximum RSS plot is shown in Fig. 4.13(a). It showed that the stress was higher around the small grain. Because the small grain in specimen C4 was closer to the end of specimen, the higher stress region was closer to the end of the specimen compared with that in specimen C3.

4.2.5 The stress strain curves for specimens in set C

Fig. 4.16(a) shows the true stress strain curves for specimens C1—C4 at 1% strain. The flow stress for C1 specimen is the highest. C4 specimen has the lowest flow stress.
Fig. 4.16(a) Stress-strain curve for C1-C4 after 1% strain
Fig. 4.16(b) Stress-strain curve for C1-C4 after 6% strain
Fig. 4.17 A plot of $\ln\sigma$-$\ln\epsilon$ curves for specimens C1-C4
Fig. 4.18 A plot of $\frac{d\ln \sigma}{d\ln \varepsilon}$ curves for specimen C1-C4
These specimens were pulled to an additional strain of 5%. Fig. 4.16(b) are the reloading true stress—strain curves. The solid lines are the experimental result. The dotted lines are the smoothed stress—strain curves for the use of calculation of lnσ—lnε curves and dlnσ/dlnε—ε curves. All the four solid stress—strain curves showed three stage deformation behaviors, easy glide stage I, secondary slip stage II, and last, multiple slip stage III. Again, the average flow stress for specimen C2 is still the highest. Stress strain curves for specimens C2—C4 showed flow stress drops in stage I or stage II, but no stress drop for specimens C1. Comparing Fig. 4.16(a) and (b), it can be seen that the yield stresses were similar. Room temperature recovery occurred to release the stress caused by straining.

Fig. 4.17 is a plot of lnσ—lnε curves for specimen C1—C4. Fig. 4.18 is a plot of work—hardening rate (n=dlnσ/dlnε) versus true strain ε. It is obvious that the work—hardening rate for specimen C1 is the highest at stage II. This is expected since C1 is a tricrystal, grain boundary induced more secondary slip at stage II. The work—hardening rate for specimen C2 is also quite high in stage II. Whereas, for specimen C3 and C4, the n values are relatively low. The peak n value is the lowest for C3. Comparing C4 with C3, the peak n value for C4 is as twice as that for C3. It is obvious that there was much more secondary slip in C4, Fig. 4.13(a), than in Fig. 4.10(a). Probably, because in C4, the small grain is much more close to the end of the
specimen. Gripping caused the complicated stress field which induced more secondary slip at grain boundary. After 4% strain, the n value for all the four specimens reached a approximately constant value (about 0.15).

4.3 Sample Set D

In sample set D, there were two specimens, D1 and D2, which were cut parallel from the strain annealed aluminum plate, Fig. 2.4. Grains D1a and D2a have the same orientation, whereas all the other grains had different orientations.

![Plate D Diagram](image)

Fig. 2.4 Schematic plot of strain-annealed plate D
4.3.1 Specimen D1

Specimen D1 had a total of three grains, with two small grains inside the big grain D1a, Fig. 2.4. One grain was a half grain at the edge of the specimen. The other was a quarter of a grain sitting at the corner of the specimen. For specimen D1, the deformation was made in three steps, from 1% to 2%, then to 4% strain (discussed in Chapter II).

Fig. 4.19(a) is a schematic plot of the slip results after 1% strain and the trace analysis. Fig. 4.19(b) is a schematic plot of the slip results after 2% strain and the trace analysis. Fig. 4.19(c) is a schematic plot of the slip results after 4% strain and the trace analysis. Fig. 4.20(a) is a contour plot of the FEM—calculated maximum RSS throughout specimen D1. Fig. 4.20(b) is the FEM—calculated slip system ranking for each observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 8.

Fig. 4.21(a) is the micrograph of slip behaviors at GBad close to Z=0 end after 4% strain. Fig. 4.21(b) is the micrograph at GBda close to +Z side. Fig. 4.21(c) is the micrograph at GBda where the grain boundary was parallel to the load direction Z. Fig. 4.21(d) is the micrograph of the place which is in between the two small grains. Fig. 4.21(e—g) are the micrograph at GBac after 1%, 2% and 4% strain respectively.
Fig. 4.19(a) Schematic plot of slip trace on D1 after 1% strain
4.19(b) Schematic plot of slip trace on D1 after 2% strain
4.19(c) Schematic plot of slip trace on D1 after 4% strain
Fig. 4.20(a) FEM-calculated maximum RSS plot for specimen D1
Fig. 4.20(b) FEM-calculated RSS ranking for the observed slip in D1
Fig. 4.21(a-b) Micrographs at GBad after 4% strain, 50X
Fig. 4.21(c) Micrograph at GBad, (d) at highly deformed region, 50X
Fig. 4.21(e-g) Micrographs at GBac of 1%, 2%, 4% strain, 50X
Table 8. The Schmid factor and RSS ranking in specimen D1 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[101]</td>
<td>#1, 0.495</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at Lc</td>
</tr>
<tr>
<td>D1a</td>
<td>#2, 0.476</td>
<td>-yc</td>
<td>#2 throughout, except #1 at Lc</td>
</tr>
<tr>
<td>C(111)[101]</td>
<td>#4, 0.206</td>
<td>L, G, hdr</td>
<td>#4 throughout, except #3 at Lc</td>
</tr>
<tr>
<td>A(111)[110]</td>
<td>#2, 0.418</td>
<td>Bulk</td>
<td>#2 all</td>
</tr>
<tr>
<td>D1d</td>
<td>#1, 0.490</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td>A(111)[011]</td>
<td>#1, 0.382</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at U+yc</td>
</tr>
<tr>
<td>D1c</td>
<td>#3, 0.189</td>
<td>G, E</td>
<td>#3 60%, #1 at Uc, #2 U</td>
</tr>
</tbody>
</table>

Key: U = Upper end +Y = +Y edge
L = Lower end -Y = -Y edge
G = Grain boundary sb = Slip band
E = Edge hdr = Highly deformed region
e.g.: +yg = grain boundary at +Y edge

Fig. 4.22 FEM mesh for specimen D1
Results and Comparison

A) Grain D1d

After 1% strain, slip system A, with Schmid factor and RSS ranked second, originated from the edge of the specimen, Fig. 4.19(a). Here, the slip system with the highest Schmid factor and RSS was not observed.

After 2% strain, slip trace B with the highest Schmid factor and RSS also started from the edge of the specimen, Fig. 4.19(b). The density for slip trace A rapidly increased. Although slip system B was the primary slip (as defined by the Schmid factor), the slip trace density for slip system A was higher than that for slip system B. It indicated that slip system A dominated during the deformation at the early stage of deformation.

After about 4% strain at the third loading, the two slip traces were in grain D1d, Fig. 4.19(c). The average slip trace density of A was higher than that for B. It was observed that at the region close to Z=0, where the grain boundary trace was approximately parallel to slip trace A, the density of slip trace A reached the maximum, Fig. 4.21(a). Away from the boundary, the spacing between the slip traces of A decreased. Comparing with Fig. 4.20(a), the RSS was higher at
the region where the density of slip system A reached the maximum
close to Z=0 end. Whereas, at GBda facing +Z direction, the average
slip trace densities for A and B were less than that at Z=0 end, Fig.
4.21(b).

B) Grain D1a

After 1% strain, there was only one slip system A with the
largest Schmid factor and RSS originating from the edge of the specimen,
Fig. 4.19(a), where the RSS for A reached the maximum. Slip system
A also nucleated from +Y edge of the specimen close to the corner
grain D1c and from the grain boundary GBac. Fig. 4.20 showed that
this is the expected site for the A slip. The average slip trace density
for A on +Z side was much higher than that at Z=0 side of the
specimen.

After 2% strain, the slip trace A was defined more clearly and
the density for slip trace A was mainly increased in the region between
the two small grains, Fig. 4.19(b). At GBad close to Z=0 end, where
slip trace A was nearly parallel to the grain boundary trace, the spacing
between the slip traces of A reached the minimum.

Close to Z=0, at GBad after the second time loading, slip trace
B with the second largest Schmid factor and RSS (at most the places)
started, Fig. 4.19(b). The FEM calculation showed that at Z=0 end
and +Y corner of the specimen, slip system B had the highest RSS,
Fig. 4.20(b).

Slip trace C with the 4th Schmid factor and RSS (at most places) was observed at the same place at the Z=0 end where slip trace B was observed. FEM simulation showed that the RSS ranking for C increased from 4th to 3rd at that position.

After 4% strain, all the three slip trace densities for A, B and C increased, Fig. 4.19(c). The slip trace density for A in grain D1a reached the maximum at the region between the two small grains, Fig. 4.20(b). Fig. 4.21(d) is the micrograph of this region. A large region between the two small grains had the same RSS value, Fig. 4.20(a). At the same stress region, the slip trace density seemed higher at the area away from the grain boundary than at the grain boundary. Along Y=0 edge of the specimen, the RSS increased to the maximum as Z coordinate increased, Fig. 4.20(a). The high stress region extended along the edge for about one third of the total length of the specimen. And the high RSS region was very narrow at the edge of the specimen. It was inferred that at one percent strain, slip system A nucleated at the edge of the specimen and did not extend far into the grain.

After deforming to 4% strain, many more slip traces were observed at both ends of the specimen in grain D1a, Fig. 4.19(c). Around the corner grain, slip trace B nucleated from the edge of the specimen and at GBac. Since the RSS ranking for B was high there,
the observation of the operation of slip system B was expected.

More slip trace C was observed at both ends after the third loading. At high slip density of A region, a few slip trace C were observed, Fig. 4.21(d).

C) Grain D1c

Slip traces A and B were hardly observed after 1% strain, Fig. 4.21(e). Slip trace A was the primary slip with the highest RSS and Schmid factor. Slip trace B with, the 3rd Schmid factor ranking was observed at the grain boundary GBac close to the upper end of the specimen. The FEM calculation showed the RSS for slip B increased from average value 138 to 219 in two elements at the corner, and the RSS ranking jumped up from the 3rd to the 1st. Away from +Y corner, the RSS ranking gradually decreased to the third.

After 2% strain, both slip traces A and B were clearly defined, Fig. 4.21(f).

After 4% strain, there were still two slip traces A and B in grain D1c. The slip trace densities increased, and both slip A and B were distributed over the whole grain, Fig. 4.21(g). The slip trace B in grain D1c seemed connected with slip trace C in grain D1a at the grain boundary and lined up a straight line. However from the calculation, it was found out that at grain boundary normal 10° inclined to Z
direction, angle $\beta$ was not the minimum, appendix C.

**Summary of the results for specimen D1**

In general, the dislocation nucleation started at the region with high RSS. At same stress region, the dislocation nucleation with the assistance of a free surface or grain boundary was preferred. The activated slip systems generally obeyed Schmid law, except at the ends of the specimen. The slip systems having lower ranked Schmid factor had higher RSS rankings which is consistent with the experiment observations.

Among the three grains, the average slip trace density in grain D1a was the highest, so was the RSS. The RSS and slip trace density in grain D1d were less and more uniform than those in grain D1a. As strain increased, the difference between the slip trace densities in each grain decreased. At the same stress region, the slip trace density was higher in the area away from the grain boundary than at the boundary.
4.3.2 Specimen D2

In specimen D2, there were two grains with the grain boundary roughly parallel to the load direction $Z$, Fig. 2.4. specimen D2 was cut parallel to specimen D1 with grain $a$ in common, so the orientation of grains D1a and D2a were the same. For specimen D2, the deformation was carried out in one step to 4% strain.

Fig. 4.23(a) is the schematic plot of the slip results after 4% strain and the trace analysis. Fig. 4.23(b) is the FEM plot of the maximum RSS of specimen D2. Fig. 4.23(c) is the FEM calculated observed slip system ranking in each grain. The details of the observed slip systems and the FEM results are shown in Table 9.

Fig. 4.24(a) is the micrograph of slip behaviors at the grain boundary between grains D2a and D2b close to $Z=0$ end after 4% strain. Fig. 4.24(b) is the micrograph along the boundary next to Fig. 4.24(a). Fig. 4.24(c) is the micrograph where all the slip systems were observed. Fig. 4.24(d) is the micrograph where slip trace densities reached the minimum. Fig. 4.24(e) is the micrograph at the place close to the top end of the specimen. Fig. 4.24(f) is the micrograph at the top end of the specimen.
Specimen D2

Fig. 4.23(a) Schematic plot of slip trace on D2 after 4% strain
4.23(b) FEM-calculated maximum RSS plot for specimen D2
4.23(c) FEM-calculated RSS ranking for the observed slip in D2
Fig. 4.24(a-b) Micrographs along the grain boundary, 50X
Fig. 4.24(c-d) Micrographs along the grain boundary, 50X
Fig. 4.24(e-f) Micrographs along the grain boundary, 50X
Table 9. The Schmid factor and RSS ranking in specimen D2 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[101]</td>
<td>#1, 0.495</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at Lc</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D2a B(111)[101]</td>
<td>#2, 0.476</td>
<td>Bulk</td>
<td>#2 throughout, except #1 at Lc</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C(111)[101]</td>
<td>#4, 0.206</td>
<td>L, G</td>
<td>#4 throughout, except #3 at Lc</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A(111)[011]</td>
<td>#2, 0.478</td>
<td>Bulk</td>
<td>#2 throughout, except #1 at L</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D2b B(111)[011]</td>
<td>#1, 0.499</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at L</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C(111)[110]</td>
<td>#3, 0.301</td>
<td>E, G</td>
<td>#3 all, σ increase at G</td>
</tr>
</tbody>
</table>

Key: U = Upper end  +Y = +Y edge
L = Lower end       -Y = -Y edge
G = Grain boundary  sb = Slip band
E = Edge            hdr = Highly deformed region
E.g.: +yg = grain boundary at +Y edge

Fig. 4.25 FEM mesh for specimen D2
Results and Comparison

A) Grain D2a

In grain D2a, the operated slip systems were exactly like those in grain D1a after 4% strain. The differences were the slip trace density and the location of the slip traces. Slip system A with the maximum Schmid factor and RSS was observed at most the places through the entire grain, except at the Z=0 end, where no slip A was observed in grain D1a, Fig. 4.23(a). Slip trace B with secondary Schmid factor was observed there, too: Fig. 4.24(a). The FEM calculation showed that slip system B had the highest RSS ranking at the Z=0 end of the specimen, Fig. 4.23(c). Along the grain boundary, the slip trace density for B was gradually decreased in +Z direction, Fig. 4.24(b). The slip density for A, on the contrary, increased and finally dominated in a large region in grain D2a, Fig. 4.23(a). Slip system C with a Schmid factor ranking of 4th was also observed, Fig. 4.24(c). The FEM calculation showed a relative increase in RSS at this region.

At the middle of the specimen, with the change of the grain boundary curvature, the slip trace densities decreased to the minimum, Fig. 4.24(c,d). As the Z coordinate increased to the Z=1 end of the specimen, the slip trace densities for both A and B increased again. The density for slip trace B increased relatively more than that of A.
This phenomenon was similar to that at the \( Z=0 \) end. But the difference was that many slip traces \( A \) were observed here, Fig. 4.24(e,f). Few slip traces of \( C \) type showed in Fig. 4.24(e), where the FEM calculation indicated the RSS ranking for slip system \( C \) increased from 4th to 5th, Fig. 4.23(c). From the FEM result, Fig. 4.23(c), it can be seen that there was no slip system ranking change for \( A \) and \( B \). This is consistent with the experiment result that many slip traces \( A \) were observed at the \( Z=1 \) end of the specimen.

B) Grain D2b

In grain D2b, three slip systems were also observed after 4% strain. Slip system \( A \), with second Schmid factor and FEM ranking (at most the places) was observed throughout the whole grain. However, slip system \( B \) with the largest Schmid factor and RSS (at most the regions) occurred at some places along the grain boundary, Fig. 4.24(a–c), but not all through the boundary. Some of the slip trace \( B \) started at the edge of the specimen, similar to the slip system \( B \) in grain D1d. The FEM calculation showed that in two layers of the elements at \( Z=0 \) end, slip system \( A \) had the highest RSS.

Slip system \( C \) with the third largest Schmid factor and RSS was observed at some regions, including the positions at the ends of the specimen, but it was not uniformly distributed. It started at the grain boundary, and propagated into the grain. Along the grain boundary, the density of slip trace \( C \) was high at some places, but at some other
places, there was no slip trace C at all. Slip C in grain D1a was observed at the most places as slip C occurred in grain D2b. The average slip trace density for C in grain D2b was much higher than that for slip C in grain D2a.

The FEM calculation showed that at dense slip sites, the RSS values for all the three slip systems A, B and C in D2b grain were high. The RSS for slip system C reached the maximum at the grain boundary, but decreased rapidly in the area away from the boundary. This was consistent with the observation of slip trace C starting at grain boundary and propagating into the grain with decreasing slip line density.

Summary of the results for specimen D2

At Z=0 end, secondary slip was activated in both grains without the existence of primary slip. This is consistent with the FEM simulation, because the secondary slip systems (A in grain D2b and B in grain D2a) had the highest RSS.

The occurrence and the distribution of slip system C in both grains D2a and D2b were strongly dependent upon the curvature of the grain boundary between the two grains. First of all, at high RSS regions, many slip systems of type C operated in both grains. Secondly, at low RSS region, slip trace C was observed where the grain boundary trace was parallel to the slip trace C in grain D2b.
4.3.3 Stress Strain Curve For Specimens D1 and D2

Fig. 4.26 shows the stress strain curves for specimens D1 and D2. It is obvious that the average flow stress for specimen D1 was much higher than that for specimen D2, although both specimens D1 and D2 had a part of one common big grain a with same orientation. In grain D2a, the average grain boundary plane was parallel to the load direction. No matter how the grain boundary normal and curvature changed in the specimen, the stress for specimen D1 was higher than that for specimen D2. In specimen D2, there seemed no stage II in the stress—strain curve, or the stage II and III are indistinguishable.

Fig. 4.27 is the plot of lnσ—lnε for specimens D1 and D2. Fig. 4.28 is the plot of work—hardening rate \( n = \frac{d\ln\sigma}{d\ln\varepsilon} \) versus true strain \( \varepsilon \). The work—hardening rate in early stage II for D1 is much higher than that of D2, which is similar to that of C1. Since specimens C1 and D1 both are tricrystals, more secondary slip might be induced by grain boundary. After rapidly hardening in stage II, the work—hardening rate decreased fast to a constant value of 0.14. For specimen D2, although the peak n value is not as high as that for D1, the work—hardening rate is much higher than that of D1 after 1.2% strain. Probably, this is because in D2, stage II and stage III combined together. Hence, the higher rate of work—hardening continued.
Fig. 4.26 Stress-strain curves for D1 and D2
Fig. 4.27 A plot of lnσ-lnε curves for D1 and D2
Fig. 4.28 A plot of $\frac{d\ln\sigma}{d\ln\epsilon}$ curves for specimen D1-D2
4.4 Sample Set E

There were five specimens cut from a big strain annealed aluminum plate E which had two big grains, a and b, Fig. 2.5. Specimens E1 and E5 were cut parallel to each other. Specimen E1 contained grains a and b. Specimen E5 was a single crystal of grain b. specimens E2, E3 and E4 were also cut parallel to each other, but 47 degrees off from specimens E1 and E5.

Fig. 2.5 Schematic plot of strain-annealed plate E
4.4.1 Specimen E1

In specimen E1, there were two grains, a and b, with the grain boundary roughly parallel to the load direction Z, Fig. 2.5. For specimen E1, the deformation was done in one step to 4% strain.

Fig. 4.29(a) is the schematic plot of the slip results after 4% strain and the trace analysis. Fig. 4.29(b) is the plot of the FEM-calculated maximum RSS of specimen E1. Fig. 4.29(c) is the FEM calculated slip system ranking for the observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 10.

Fig. 4.30(a—d) are the micrographs of slip behavior along the grain boundary between grains E1a and E1b after 4% strain.

Results and Comparison

A) Grain E1a

In grain E1a, there were three slip systems observed, Fig. 4.29(a). Slip system A, with the highest Schmid factor and RSS ranking (in most places), was observed all over the grain. In Fig. 4.29(b), it can be seen that the high stress region were mainly two: one was close to the Z=0 end of the specimen and the other was at Z=1 end at −Y corner. Three slip systems were observed at this high RSS region,
Specimen E1

Fig. 4.29(a) Schematic plot of slip trace on E1 after 4% strain
4.29(b) FEM-calculated maximum RSS plot for specimen E1
4.29(c) FEM-calculated RSS ranking for the observed slip in E1
Fig. 4.30(a-b) Micrographs along the grain boundary, 50X
Fig. 4.30(c-d) Micrographs along the grain boundary, 50X
Table 10. The Schmid factor and RSS ranking in specimen E1 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[110]</td>
<td>#1, 0.435</td>
<td>Bulk</td>
<td>#1 most, except #2 at L, U, GB</td>
<td></td>
</tr>
<tr>
<td>E1a</td>
<td>D(111)[101]</td>
<td>#2, 0.057</td>
<td>G</td>
<td>#3 all</td>
</tr>
<tr>
<td>C(111)[011]</td>
<td>#1, 0.435</td>
<td>G, L</td>
<td>#2 most, except #1 at L, U, GB</td>
<td></td>
</tr>
<tr>
<td>E1b</td>
<td>D(111)[101]</td>
<td>#1, 0.438</td>
<td>E</td>
<td>#1 throughout, except #2 at L+y, U+y</td>
</tr>
<tr>
<td>B(111)[011]</td>
<td>#2, 0.305</td>
<td>E, G</td>
<td>#2 throughout, except #1 at L-y, U-y #3 at L+y, U+y</td>
<td></td>
</tr>
</tbody>
</table>

Key: U = Upper end +Y = +Y edge
L = Lower end -Y = -Y edge
G = Grain boundary sb = Slip band
E = Edge hdr = Highly deformed region
e.g.: +yg = grain boundary at +Y edge

Fig. 4.31 FEM mesh for specimen E1
although slip system C had the same highest Schmid factor and RSS ranking of first at the most places and D was ranked 2nd.

Slip D in grain E1a was observed in several high RSS regions along the grain boundary, Fig. 4.29(a). It was also found that the density for slip trace B reached the maximum when the grain boundary trace was parallel to the trace B, Fig. 4.30(a).

Slip trace C with the same Schmid factor ranking of one was observed everywhere in grain E1a, although the slip density for C was not high. FEM simulation showed that in most the places, slip system C had RSS ranking in two. Fig. 4.30(b) is the micrograph of the high RSS region where the grain boundary was quite flat. The slip trace was distributed quite uniformly. Fig. 4.30(c) is the micrograph of the region close to Z=0 end. The slip trace density for C increased dramatically. The FEM calculation showed that the RSS for C increased relatively at grain boundary, Fig. 4.29(c). Although the FEM calculation showed that the RSS for slip system C decreased gradually in the area away from the grain boundary, slip trace C still penetrated into the grain for a long distance, some reached to the edge of the specimen.

At the Z=1 end of the specimen, where the grain boundary trace was more parallel to slip trace A, the density for slip trace A reached the maximum. Fig. 4.30(d) is the micrograph of the region close to the
Z=1 end of the specimen. Since the FEM simulation did not show any change in RSS ranking for D, the slip trace density for D was expected to be low.

B) Grain E1b

In grain E1b, three slip traces were observed. Slip system A with Schmid factor and RSS ranking of one (at most places) was observed in the entire grain. At the Z=0 end at the grain boundary, the RSS ranking for slip trace B shifted to second. A decrement in slip density for B occurred obviously in the experiment, Fig. 4.30(c—d). But at those regions with high RSS, Fig. 4.29(b), the slip trace density for A reached the maximum, Fig. 4.30(a,b).

Even though its Schmid factor and RSS ranked third at most the places, slip trace A in grain E1b was observed almost in the whole grain. The slip trace density for A reached the maximum at the Z=1 end, and the RSS for A increased from third to second at Z=0 end and at the +Y corner, Fig. 4.30(c). The density of A decreased in the region away from the end. At the middle of the specimen, the spacing between slip bands was very large in the experiment, Fig. 4.29(a). This was in agreement with the FEM calculation since the RSS was slightly lower at the same region. At the Z=1 end of the specimen, the RSS for slip trace A also increased its order from third to second, which was consistent with FEM calculation. In the experiment, the slip trace density for A was relatively higher at both the ends of the specimen.
Slip trace B in grain E1b was mostly observed at the ends of
the specimen, although its Schmid factor and RSS ranking were second.
At the Z=0 end —Y corner, the RSS ranking rose to the first. But at
the same end +Y corner, the RSS ranking was reduced to 3rd for D.
No micrograph was taken at +Y corner at Z=0 end in the experiment.
Along the grain boundary at Z=0 end, the spacing between the slip B
bands increased in +Y direction, Fig. 4.30(c). The RSS for B, on the
contrary, increased to first at the Z=0 end —Y corner, but decreased to
the 3rd at +Y corner. In the experiment, the slip density for B
decreased in +Y direction. Slip trace B was mainly observed as the
normal of the grain boundary perpendicular to the load direction Z. In
general, the slip trace density for B in grain E1b was not high.

4.4.2 Specimen E2

Specimen E2 contained two big grains a and b, and a small
grain c which sits at the grain boundary between grains a and b. For
specimen E2, the deformation was carried out first to 4% strain followed
by annealing. Then, E2 was deformed 1%, and an additional 1% strain.
Fig. 4.32(a—c) are the schematic plots of the slip results after 1%, 2% and 4% strain respectively and the trace analysis. Fig. 4.33(a) is the plot of the FEM—calculated maximum RSS throughout specimen E2. Fig. 4.33(b) is the FEM calculated slip system ranking for each observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 11.

Fig. 4.34(a—e) are the micrographs of slip behavior at each grain after 4% strain.

Results and Comparison

A) Grain E2a

After 1% strain, slip systems B and C, with Schmid factors and RSS rankings of first and the 4th respectively, were observed at the regions away from the grain boundary, Fig. 4.32(a).

After 2% strain, much more slip trace B were observed, Fig. 4.32(b). Both slip C and slip B were connected with the grain boundary.

After 4% strain, slip system A with Schmid factor and RSS ranking of second was observed only at the region close to the grain boundary triple node at +Y side, Fig. 4.34(a). It was found that at this region, the RSS for slip system C increased, Fig. 4.33(b). Away
Fig. 4.32(a) Schematic plot of slip trace on E2 after 1% strain
4.32(b) Schematic plot of slip trace on E2 after 2% strain
4.32(c) Schematic plot of slip trace on E2 after 4% strain
Fig. 4.33(a) FEM-calculated maximum RSS plot for specimen E2
Fig. 4.33(b) FEM-calculated RSS ranking for the observed slip system
Fig. 4.34(a) Micrograph at GB triple node, (b) at GBac, 50X
Fig. 4.34(c) Micrograph at GB triple node at -Y edge, 50X
(d) Micrograph at GBbc, 50X
Fig. 4.34(e) Micrograph at GBbc, 50X
Table 11. The Schmid factor and RSS ranking in specimen E2 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>C(111)[110]</td>
<td>#4, 0.130</td>
<td>Bulk</td>
<td>#4 all</td>
</tr>
<tr>
<td>E2a</td>
<td>B(111)[011]</td>
<td>#1, 0.425</td>
<td>#1 throughout, except #2 at U</td>
</tr>
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<td></td>
<td>A(111)[011]</td>
<td>#2, 0.256</td>
<td>#2 throughout, except #1 at U</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>A(111)[101]</td>
<td>#1, 0.456</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[011]</td>
<td>#3, 0.392</td>
<td>#2</td>
</tr>
<tr>
<td>E2c</td>
<td>C(111)[011]</td>
<td>#3, 0.392</td>
<td>#3 all</td>
</tr>
<tr>
<td></td>
<td>D(110)[110]</td>
<td>#5', 0.009</td>
<td>#5' all</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>B(111)[110]</td>
<td>#2, 0.421</td>
<td>#2 most, except #1 at Lc-y</td>
</tr>
<tr>
<td>E2b</td>
<td>A(111)[110]</td>
<td>#1, 0.465</td>
<td>#1 throughout, except #2 at Lc-y</td>
</tr>
<tr>
<td></td>
<td>D(111)[101]</td>
<td>#4, 0.141</td>
<td>#4 all</td>
</tr>
</tbody>
</table>

Here 5' represents unusual slip system ranking in 5

Key: U = Upper end  +Y = +Y edge
L = Lower end  -Y = -Y edge
G = Grain boundary sb = Slip band
E = Edge  hdr = Highly deformed region
e.g.: +yg = grain boundary at +Y edge

Fig. 4.35 FEM mesh for specimen E2
from the grain boundary triple node, no slip trace A was observed. The slip densities for C and B increased again. Slip trace B had the highest slip trace density among the three slip traces. In general, the RSS for slip B was fairly uniform in a large area, Fig. 4.33(a). The maximum RSS showed little change along the grain boundary from \( Y=0 \) to \( Y=w \) side. Fig. 4.34(b) is a typical micrograph at GBac. Fig. 4.34(c) is the micrograph of grain boundary triple node at \( Y=0 \) side where no slip trace A was observed.

B) Grain E2c

After 1% strain, slip traces A and B were observed, Fig. 4.32(a). Slip system A, with the maximum Schmid factor and RSS, nucleated at the region close to the triple grain boundary node on \(+Y\) side, whereas, slip trace B started at GBcb.

After 2% strain, both slip traces A and B were observed with higher slip trace densities throughout the whole grain.

After the third loading, the slip trace densities for A and B increased. Two other slip traces C and D were observed, Fig. 4.32(c), Fig. 4.34(b,c). Slip trace C with the same secondary Schmid factor as slip trace B was observed at GBcb and \(-Y\) side, Fig. 4.34(c). However, its penetration distance was much shorter than that for B. The FEM simulation generally showed a higher RSS ranking for C than for B in a large area, although both C and B had the same Schmid factor, Fig.
4.33(b). Slip system D was the unusual slip system \((1\overline{1}0)[110]\), and was observed only at the center of the small grain after the third time loading, Fig. 4.34(b). D is the continuation of slip trace A in grain E2a on +Z side of the grain. The Schmid factor for D was the smallest, comparing with that for all the \(\{111\}\) type slip systems. On the other side of the grain, there seemed a small angle between the slip trace D in grain E2b and the slip trace D in grain E2c, Fig. 4.34(d).

C) Grain E2b

After 1% strain, three slip systems with few slip traces were observed at the region away from the boundary. Slip trace A was the primary slip with the maximum Schmid factor and RSS at most places. Slip system B, with Schmid factor ranking of 2th, nucleated at the \(-Y\) and \(+Y\) edges of the specimen. Slip system D with the Schmid factor and RSS ranking of fourth was observed at the regions close to \(-Y\) and \(+Y\) edges.

After 2% strain, there were still three slip traces in grain E2b. Slip trace A spread over the whole grain. The density for A increased greatly. Slip trace B propagated toward the grain boundary.

After 4% strain, no new slip system was observed. The maximum RSS contour plot basically showed the two high stress regions at the two sides of the small grain, Fig. 4.33(a). And at the \(-Y\) side,
the high stress region extended into the grain for the length of two thirds of the grain. In the experiment, higher slip trace densities were observed at the higher stress region along the two edges of the specimen near the small grain. Especially for slip trace B, it mainly nucleated at the two edges of the specimen near the small grain. Along GBbc, the density of the primary slip trace A changed largely. The slip trace density for A reached the maximum when slip trace A was parallel to the grain boundary trace, Fig. 4.34(e).

Generally, the slip trace distribution observed from the experiment matched the FEM simulation very well. The activation of the low Schmid factor slip system is probably because of the surface effect.

4.4.3 Specimen E3

Specimen E3 was cut parallel to specimen E2. The two grains a and b had the same orientation as grains E2a and E2b. The difference was that specimen E3 was a bicrystal with the grain boundary about 40 degrees inclined to the load direction. The specimen E3 was deformed in the same procedure as E2, Table 1.

Fig. 4.36(a—c) are the schematic plots of the slip results after 1%, 2% and 4% strain respectively and the trace analysis. Fig. 4.37(a) is the plot of the FEM—calculated maximum RSS for specimen E3. Fig.
4.37(b) is the FEM calculated slip system ranking for the observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 12.

Fig. 4.38(a) is the micrograph of slip behaviors at the grain boundary between grains E3a and E3b at the region close to +Y edge at 4% strain. Fig. 4.38(b) is the micrograph along the grain boundary next to Fig. 4.38(a). Fig. 4.38(c) is the micrograph where the slip densities reached the maximum in grain E3a. Fig. 4.38(d) is the micrograph where the slip trace densities reached the maximum in grain E3b.

Results and Comparison

A) Grain E3a

Since grain E3a was orientated the same as grain E2a, the operated slip systems should be the same. In fact, after 1% strain, slip trace C, with Schmid factor and RSS ranking of 4th, was observed at the region away from the grain boundary in grain E3a. Unlike the case of grain E2a, there was no slip system B observed after 1% strain.
Specimen E3

Fig. 4.36(a) Schematic plot of slip trace on E3 after 1% strain
Fig. 4.36(b) Schematic plot of slip trace on E3 after 2% strain
Fig. 4.36(c) Schematic plot of slip trace on E3 after 4% strain
Specimen E3

Fig. 4.37(a) FEM-calculated maximum RSS plot for specimen E3
Fig. 4.37(b) FEM-calculated RSS ranking for the observed slip in E3
Fig. 4.38(a-b) Micrographs along the grain boundary, 50X
Fig. 4.38(c) Micrograph in grain a, (d) in grain b, 50X
Table 12. The Schmid factor and RSS ranking in specimen E3 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C(111)[110]</td>
<td>#4, 0.130</td>
<td>Bulk</td>
<td>#4 all</td>
</tr>
<tr>
<td>E3a</td>
<td>B(111)[011]</td>
<td>#1, 0.425</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at Uc</td>
</tr>
<tr>
<td></td>
<td>B(111)[110]</td>
<td>#2, 0.421</td>
<td>edge</td>
<td>#2 most, except #1 at L</td>
</tr>
<tr>
<td>E3b</td>
<td>A(111)[110]</td>
<td>#1, 0.465</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at L</td>
</tr>
<tr>
<td></td>
<td>D(111)[101]</td>
<td>#4, 0.141</td>
<td>Bulk</td>
<td>#4 all</td>
</tr>
</tbody>
</table>

Key: U = Upper end +Y = +Y edge
L = Lower end -Y = -Y edge
G = Grain boundary sb = Slip band
E = Edge hdr = Highly deformed region
e.g.: +yg = grain boundary at +Y edge

Fig. 4.39 FEM mesh for specimen E3
After 2% strain, similar to the case of grain E2a, two slip systems (C and B) were observed in grain E3a, not only at the grain boundary, but also at the region away from the boundary. No much more slip trace C appeared, comparing with that after 1% strain.

After the third loading, the slip trace densities for both C and B increased. No new slip trace was observed. Along the grain boundary, Fig. 4.38(a,b), the slip trace densities did not vary much in grain E3a. Away from the grain boundary at same RSS value region, the slip trace densities reached the maximum, Fig. 4.38(c).

B) Grain E3b

After 1% strain, there were three slip traces observed. Slip trace A, with the maximum Schmid factor and RSS (at most the places), started at the +Y edge of the specimen, Fig. 4.36(a). Slip system B nucleated along the two edges of the specimen. Slip system D, with the Schmid factor and RSS ranking of fourth (at most places), originated from the grain boundary.

After an additional 1% strain, all the three slip traces A, B and D increased. Slip traces A and B mainly increased at the region away from the grain boundary. Whereas, more slip trace D was observed at GBab.
After 4% strain, slip trace densities for all the traces in grain E3b increased greatly. Fig. 4.38(d) is the micrograph at the place where the RSS and slip trace densities reached the maximum in grain E3b.

Summary the result for grain E3

In general, comparing with the FEM RSS plot, Fig. 4.37(a), the average slip trace density and RSS increased along GBab in +Y direction consistently in grain E3a. In grain E3b, the slip trace density and RSS were quite uniformly distributed. The average stress was higher in grain E3b than in grain E3a. The slip trace densities reached the maximum at the regions away from GBab in both grains. In grain E3b, the RSS had the highest value at the high slip sites away from GBab.

4.4.4 Specimen E4

Specimen E4 was a single crystal and was cut parallel to specimens E2 and E3. For specimen E4, the deformation was carried out in one step to 8% strain directly.

Fig. 4.40(a) is the schematic plot of the slip results after 8% strain. Fig. 4.40(b) is the plot of the FEM—calculated maximum RSS throughout the specimen. Fig. 4.30(c) is the FEM calculated slip system ranking for the observed slip. The details of the observed slip systems
and the FEM results are shown in Table 13.

Fig. 4.41 is the micrograph of slip behavior at the middle of the grain.

Results and Comparison

In Fig. 4.41, there were some dark Luders bands which were almost perpendicular to the Z direction. The distance between each Luders band was almost the same, about 2.4mm. Since specimen E4 was cut parallel to specimens E2 and E3, there were three same slip traces A, B and D as in grains E2b and E3b. Slip trace A with the maximum and RSS (at most the places), was the primary slip and it resided concentratively in the dark Luders band region. Slip traces B and D with Schmid factor and RSS (at most the places), ranking second and 4th respectively, were observed with much lower densities.

A torque effect is exhibited in the maximum RSS plot. Since the Luders band is mainly a plastic effect, it cannot be predicted by the current continuum elastic FEM.
Fig. 4.40(a) Schematic plot of slip trace on E4 after 8% strain
4.40(b) FEM-calculated maximum RSS plot for specimen E4
4.40(c) FEM-calculated RSS ranking for the observed slip in E4
Fig. 4.41 Typical micrograph in specimen E4, 50X
Table 13. The Schmid factor and RSS ranking in specimen E4 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain Slip System</th>
<th>Schmid Factor</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>B(111)[110]</td>
<td>#2, 0.421</td>
<td>edge</td>
<td>#2 most, except #1 at L, U</td>
</tr>
<tr>
<td>E4b</td>
<td>A(111)[110]</td>
<td>#1, 0.465</td>
<td>Bulk</td>
</tr>
<tr>
<td>D(111)[101]</td>
<td>#4, 0.141</td>
<td>Bulk</td>
<td>#4 all</td>
</tr>
</tbody>
</table>

Key: U = Upper end  
L = Lower end  
G = Grain boundary  
E = Edge  
sb = Slip band  
hdr = Highly deformed region  
e.g.: +yg = grain boundary at +Y edge

Fig. 4.42 FEM mesh for specimen E4
4.4.5 Specimen E5

Specimen E5 was a single crystal cut parallel to specimen E1. Specimen E5 was deformed to 4% strain in one step.

Fig. 4.43(a) is a schematic plot of the slip results after 4% strain. Fig. 4.43(b) is the plot of the FEM—calculated maximum RSS throughout the specimen. Fig. 4.43(c) is the FEM calculated slip system ranking for the observed slip in the specimen E5. The details of the observed slip systems and the FEM results are shown in Table 14.

Fig. 4.44(a) is the micrograph of slip behavior at the middle of the grain. Fig. 4.44(b) is the micrograph of slip behaviors at some impurities.

Results and Comparison

Since grain E5b had the same orientation as grain E1b, so the operating slip systems should be the same. Slip system D with the largest Schmid factor and RSS (in most the elements), was observed all over the grain. Slip traces B and A with the second and third largest Schmid factor and RSS were not clearly defined. The slip densities were generally uniform, except at some places having some impurity particles, Fig. 4.44(a). At those places, more secondary slip traces A appeared around those particles, Fig. 4.44(b).
Fig. 4.43(a) Schematic plot of slip trace on E5 after 4% strain
4.43(b) FEM-calculated maximum RSS plot for specimen E5
4.43(c) FEM-calculated RSS ranking for the observed slip in E5
Fig. 4.44(a) Typical micrograph in specimen E5, (b) at impurity, 50X
Fig. 4.45(a) Refined mesh around a particle for FEM simulation
(b) FEM-calculated maximum RSS around the particle
Table 14. The Schmid factor and RSS ranking in specimen E5 for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>A(111)[101]</td>
<td>#3, 0.286 Bulk</td>
<td>#3 throughout, except #2 at L, U</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E5b</td>
<td>D(111)[101]</td>
<td>#1, 0.438 Bulk</td>
<td>#1 throughout, except #2 at L, U</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B(111)[010]</td>
<td>#2, 0.305 Bulk</td>
<td>#2 throughout, except #1 at L, U</td>
<td></td>
</tr>
</tbody>
</table>

Key: U = Upper end  
L = Lower end  
G = Grain boundary  
E = Edge  
e.g.: -ylc = lower end -Y corner

Table 15. The comparison of FEM-calculated RSS for each slip in different meshes in specimen E5

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Uniform Mesh</th>
<th>Uniform Mesh with Impurity</th>
<th>Fine Smooth Mesh Around Impurity</th>
</tr>
</thead>
<tbody>
<tr>
<td>E5b</td>
<td>B(111)[011]</td>
<td>234</td>
<td>235</td>
<td>243</td>
</tr>
<tr>
<td>A(111)[101]</td>
<td>210</td>
<td>210</td>
<td>214</td>
<td></td>
</tr>
</tbody>
</table>

Here, the numbers are the RSS values in arbitrary unit.
FEM simulation was then carried out assuming there was a rigid particle in the matrix. The particle was first calculated as a rectangular shape. The RSS for the secondary slip A showed a slight increase, Table 15. FEM calculation was then made for a spherical shape rigid particle with very fine mesh around it. Only small part of the specimen around the particle was simulated. The boundary condition was chosen as there was a rigid rectangular shape particle. Fig. 4.45(a) is the refined mesh around the particle. Fig. 4.45(b) is a plot of the FEM—calculated maximum RSS. The simulation result showed a large RSS increasing for the secondary slip A, Table 15. Whereas, RSS for slip system A did not increase much, which explained that there was not many slip trace A around the particle, Fig. 4.44(b).

4.4.6 The stress—strain relationships

Fig. 4.46(a) is the plot of true stress—true strain curves for specimens E1, E4 and E5. Again, the solid lines are the experimental results. The dotted lines are the smoothed lines for the calculation of \( \ln\sigma - \ln\epsilon \) and \( \frac{d\ln\sigma}{d\ln\epsilon} - \epsilon \). In Fig. 4.46(a), the flow stress for E1 (bicrystal with grain boundary normal roughly perpendicular to the load direction) is lower than that for E4 or E5 (E4 and E5 are single crystals with different orientations). Stage I for E5 is very short, whereas, stage I for E1 and E4 was long. The transition from stage II
Fig. 4.46(a) Stress-strain curves for E1, E4 and E5
to stage III for E1 and E5 was smooth. Specimen E4 had a rapid work-hardening stage II, and a clear transition from stage II to III.

Fig. 4.46(b) is the plot of true stress—true strain curves for specimens E2, E3 and E4. The dotted line for E2 is the smoothed line for the use of dlnσ/dlnε calculation. E2 (tricrystal with a small grain sitting at the grain boundary between the two big grains) had a higher yield stress than that of E3 and E4. All of these three specimens had a long stage I. Specimen E3 had a highest flow stress at the transition point from stage II to stage III, whereas, specimen E2 had a highest working-hardening rate rate in stage III.

Fig. 4.47 is the lnσ—lnε plot for E set specimens. Fig. 4.48(a) is the plot of work-hardening rate (n=dlnσ/dlnε) versus true strain for specimen E1, E4 and E5. There are two n peaks for specimen E1 because of the two sharp transition in σ—ε curve. Although the second peak n value is less than that of specimen E4, the work-hardening rate decreased gradually in E1 than that in E4. Since specimen E1 is a bicrystal, grain boundary produced more secondary slip. Therefore, work-hardening rate should be higher than that of E4. The n—ε curve for E5 is very smooth. Since the stage II and stage III for E5 is combined together, at the transition region from stage II to III, the work-hardening rate is higher than that of E4 and E1 at the same regions.
Fig. 4.46(b) Stress-strain curves for E2, E3 and E4
Fig. 4.47 A plot of lnσ-lnε curves for E set specimen
Fig. 4.48(a) A plot of $\frac{d\ln\sigma}{d\ln\varepsilon}$--$\varepsilon$ curves for E1, E4 and E5
Fig. 4.48(b) A plot of $d\ln\sigma/d\ln\epsilon - \epsilon$ curves for E2, E3 and E4
Fig. 4.48(b) is the plot of work—hardening rate \( \left( n = \frac{\ln \sigma}{\ln \epsilon} \right) \) versus true strain for specimen E2, E3 and E4. All the three curves have a sharp transition from stage I to stage II. The peak \( n \) value is the largest for E3 among the three specimens. Comparing the schematic plot for E2 and E3 after 1% strain, it was found out that slip traces in grain E3b were mainly generated at grain boundary, Fig. 4.36(a). Whereas, in E2, slip traces were started away from the boundary. It was also found that the yield stress for E3 was the highest among E2, E3 and E4, Fig. 4.46(b). Hence, it can be concluded that the transverse grain boundary increases the yield stress and the flow stress in the Stage I, II and early stage III more than other sort of grain boundaries. After 1.5% strain, the work—hardening rate for E2 is higher than that of E3, Fig. 4.48(b). The result can be shown in the schematic plot of the slip analysis for E2 and E3 that E2 had a higher slip trace density, Fig. 4.30(c), than that of E3, Fig. 4.36(c).
Sample Set F

There was just one specimen from the plate F, with six grains, Fa, Fb, Fc, Fd, Fe and Ff, Fig. 2.6. Grains Fa and Fe were two big grains. The other four grains were embedded in these two big grains. Grains Fc and Fd sit at the grain boundary between the two big grains. Specimen F was deformed in one step to about 4% strain.

Fig. 2.6 Schematic plot of strain-annealed plate F
Fig. 4.49(a) is a schematic plot of the slip results after 4% strain. Fig. 4.49(b) is the plot of the FEM—calculated maximum RSS throughout the specimen. Fig. 4.49(c) is the FEM—calculated slip system ranking for the observed slip in each grain. The details of the observed slip systems and the FEM results are shown in Table 16. Fig. 4.50(a—i) are the micrographs at grain boundaries.

Results and Comparison

1) Grain Fa

There were three slip traces observed after deformation in grain Fa. Slip system A, with the maximum Schmid factor and RSS, was observed almost everywhere. Along the grain boundary GBba, slip A was not uniformly distributed. Fig. 4.50(a) is the micrograph of the grain boundary at edge of the specimen close to +Z side, where few slip traces of A were observed. Fig. 4.50(b) is the micrograph at the middle of the grain, where more slip traces A appeared. Fig. 4.50(c) is the micrograph at the same boundary at the specimen edge, but close to −Z side. It can be seen that many slip traces A appeared. The density of slip trace A in grain Fa increased tremendously as the grain boundary was parallel to trace A, and the grain boundary seemed not to be distinguishable. Because of the darkness of the grain boundary and the discontinuity of the secondary slip at the region, the grain boundary could still be identified. Away from the small grain Fb, slip trace density for A was low, Fig. 4.50(a).
Fig. 4.49(a) Schematic plot of slip trace on F after 4% strain
4.49(b) FEM-calculated maximum RSS plot for specimen F
4.49(c) FEM-calculated RSS ranking for the observed slip in F
Fig. 4.49(d) Enlarged Fig. 4.48(a) at grains Fb, Fc, Fd and Ff
Fig. 4.50(a-c) Micrographs at GBab, 50X
Fig. 4.50(d-f) Micrographs around grain c and d, 50X
Fig. 4.50(g-i) Micrographs around grain f, 50X
Fig. 4.51 FEM mesh for specimen F
Table 16. The Schmid factor and RSS ranking in specimen F for observed slip systems.

<table>
<thead>
<tr>
<th>Grain</th>
<th>Slip System</th>
<th>Schmid Factor Ranking</th>
<th>Where Observed</th>
<th>FEM Result Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fa</td>
<td>A(111)[101]</td>
<td>#1, 0.457</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at Lcenter</td>
</tr>
<tr>
<td></td>
<td>B(111)[101]</td>
<td>#2, 0.430</td>
<td>Bulk</td>
<td>#2 throughout, except #1 at Lcenter</td>
</tr>
<tr>
<td></td>
<td>C(111)[011]</td>
<td>#3, 0.114</td>
<td>G</td>
<td>#3 all</td>
</tr>
<tr>
<td>Fb</td>
<td>A(111)[011]</td>
<td>#2, 0.461</td>
<td>Bulk</td>
<td>#2 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[011]</td>
<td>#1, 0.477</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>C(111)[110]</td>
<td>#4, 0.174</td>
<td>G</td>
<td>#4 all</td>
</tr>
<tr>
<td></td>
<td>D(111)[110]</td>
<td>#3, 0.302</td>
<td>G-y</td>
<td>#3 all</td>
</tr>
<tr>
<td>Fc</td>
<td>A(111)[110]</td>
<td>#1, 0.412</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[110]</td>
<td>#3, 0.230</td>
<td>Bulk</td>
<td>#3 all</td>
</tr>
<tr>
<td></td>
<td>C(111)[101]</td>
<td>#4, 0.154</td>
<td>G-z</td>
<td>#4 all</td>
</tr>
<tr>
<td>Fd</td>
<td>A(111)[101]</td>
<td>#1, 0.44</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[101]</td>
<td>#2, 0.436</td>
<td>Bulk</td>
<td>#2 all</td>
</tr>
<tr>
<td>Fe</td>
<td>A(111)[101]</td>
<td>#1, 0.494</td>
<td>Bulk</td>
<td>#1 throughout, except #2 at U</td>
</tr>
<tr>
<td></td>
<td>B(111)[101]</td>
<td>#2, 0.491</td>
<td>Bulk</td>
<td>#2 throughout, except #1 at U</td>
</tr>
<tr>
<td></td>
<td>C(111)[110]</td>
<td>#3, 0.288</td>
<td>G</td>
<td>#3 all</td>
</tr>
<tr>
<td>Ff</td>
<td>A(111)[110]</td>
<td>#2, 0.389</td>
<td>Bulk</td>
<td>#2 all</td>
</tr>
<tr>
<td></td>
<td>B(111)[011]</td>
<td>#1, 0.447</td>
<td>Bulk</td>
<td>#1 all</td>
</tr>
<tr>
<td></td>
<td>C(111)[011]</td>
<td>#3, 0.332</td>
<td>G+y</td>
<td>#3 all</td>
</tr>
<tr>
<td></td>
<td>D(111)[011]</td>
<td>#4, 0.044</td>
<td>G-z</td>
<td>#4 all</td>
</tr>
</tbody>
</table>

Key: U = Upper end
L = Lower end
G = Grain boundary
E = Edge

+Y = +Y edge
-Y = -Y edge
sb = Slip band
hdr = Highly deformed region

e.g.: +yg = grain boundary at +Y edge
Slip trace B, having the secondary largest Schmid factor and RSS, was observed mostly at the GBad and GBac, and its density was far less than that of A, although their Schmid factors were close. Very few slip traces B appeared at GBab, Fig. 4.49(a). Fig. 4.50(d,e) showed more details of the slip around the small grain Fc. Fig. 4.50(e) showed the grain boundary triple junction point. In grain Fa, there were lots of slip traces B. The slip trace density for B varied along GBac. When grain boundary plane was parallel or close to parallel to the slip trace direction, the slip trace density of B was high.

Slip trace C, with the third largest Schmid factor and RSS ranking was observed in certain regions, Fig. 4.49(a). First, slip trace C appeared close to the Z=0 end of the specimen. The FEM simulation showed that at the end, near +Y corner, the RSS changed its value from average 58 to 128, Fig. 4.49(c). Secondly, slip trace C was observed at the small grain Fb, Fig. 4.50(b). When the slip trace C was parallel to the grain boundary plane, its density reached the maximum. The spacing of slip trace C increased rapidly away from the grain boundary. Thirdly, slip trace C also appeared at the GBac and GBad where the grain boundary normals were nearly perpendicular to the slip trace C. In some regions of grain Fa near grain Fc, slip trace C might be caused by the slip trace C in grain Fc, or visa versa. Furthermore, slip trace C was also observed near Y=0 edge at the
2). Grain Fb

Grain Fb was the half grain embedded in grain Fa and it was on the edge of the specimen. Four slip traces A, B, C and D were observed in there, Fig. 4.49(a).

Slip trace A, with the second largest Schmid factor and RSS, was activated at the middle of the grain where the grain boundary plane was close to parallel to the load direction Z, Fig. 4.50(b,c). Few slip traces were extended to the whole grain. No slip system A was observed away from the middle position of Fb, Fig. 4.50(a).

Slip trace B with the largest Schmid factor and RSS was observed all over the grain. As the grain boundary plane was close to or nearly parallel to the slip trace B, the slip band density of B was greatly increased, Fig. 4.50(c). At these positions, slip trace B could cross over the GBab to grain Fa, and coincided with the slip trace A (111) in grain Fa.

Slip trace C (111), with the fourth Schmid factor and RSS, was observed too, Fig. 4.50(a), but only at the localized position with only three slip traces of C. No primary slip trace A was observed there.
Slip trace D (11\overline{1}), with the third Schmid factor and RSS, was observed also in the same micrograph, Fig. 4.50(a). The difference was that slip trace D appeared closer to the edge of the specimen.

3. Grain Fc

Grain Fc was a small grain sitting at the grain boundary between the two big grains Fa and Fe. Three slip traces A, B and C were observed, Fig. 4.49(a).

Slip trace A (\overline{1}11), with the maximum Schmid factor and the RSS, was observed all over the grain. At the grain boundary triple node and Fc, Fa and Fe junctions, the slip trace densities were very low for all the slip systems, Fig. 4.50(e). In Fig. 4.49(b), the FEM calculated maximum RSS was lower in grain Fa than that in grain Fe. Away from the triple point, the slip band density increased significantly. About three quarters of the grain, slip band A was distributed quite evenly.

The slip B (11\overline{1}), with Schmid factor and RSS ranking of third, was observed after the deformation, Fig. 4.49(a). The main profile was similar to that of A, but with a lower density than that of A.

Slip trace C with Schmid factor and RSS ranking of fourth, was observed near the triple grain boundary node at the grain boundary between grain Fa and grain Fc, Fig. 4.50(e).
Generally, the slip trace densities in grain Fc were slightly higher on the +Z side of the grain boundary than that at the low Z side. It was consistent with the result of the FEM simulation.

4). Grain Fd

Similar to grain Fc, grain Fd was also a small grain sitting in the grain boundary between the big grains Fa and Fb. There were two activated slip systems A and B, Fig. 4.49(a).

Slip trace A (111), with the maximum Schmid factor and RSS, was observed everywhere in the grain. Generally, the slip band was uniformly distributed. Only at the region close to grain Fc, the slip density was low. This was expected because the FEM simulation showed a lower RSS at the region adjacent to grain Fc. Similar to the case of grain Fc, at the two triple grain boundary node points, there was not much heterogeneous slip and stress concentration in grain Fd.

Slip trace B, with the Schmid factor and RSS ranking of second, was observed throughout the entire grain. Although the Schmid factor for B was very close to that of A, the slip density for B was quite less than that of A, Table 16.
5). Grain Fe

Grain Fe was the other larger grain which located at the upper end in specimen F. Three slip systems were observed to operate, Fig. 4.49(a).

Slip trace A (111), with the highest Schmid factor and RSS among all the grains in specimen F, was observed in the whole grain. The slip trace density for A at GBec and GBed, was much higher than those at GBac and GBad. In Fig. 4.49(b), the RSS has a much higher value at the +Z side of grain boundary than that of the lower Z side of grain boundary of the two small grains. Fig. 4.50(f) is the micrograph of the GBed at +Y side. It was found that slip trace A in grain Fe could cross over the grain boundary into the small grain Fd and be connected with slip trace A in grain Fd. The two slip traces were not exactly parallel to each other and slip trace A rotated at grain boundary. At the places with good continuity in grain Fd, highly condensed slip trace A was observed, Fig. 4.50(e). At this position, the β angle is minimum for these two slip planes, appendix C. As the grain boundary normal changed, the continuity became weak. The slip trace A in grain Fd even disappeared, although the slip trace A had the maximum Schmid factor.

Around the small grain Ff, the density of slip trace A changed also. The spacings between the slip traces on the grain boundary close to +Z end was smaller than that on the other side since the RSS
showed a larger value close to +Z end. Fig. 4.50(g,h) are the micrographs from the other side of the grain. Generally the slip trace density was higher in Fig. 4.50(g—h). In Fig. 4.50(h), slip trace A was also connected with slip trace C in the small grain Ff. Similar to the case at the GBed, slip trace C in grain Ff also rotated at the grain boundary. The difference was that at high continuity places, no condensed slip trace C was observed, although slip trace C was the primary slip in grain Ff. When the trace of GBeF changed to parallel slip trace A, the slip density for A reached the maximum.

Slip trace B (111), with the second largest Schmid factor and RSS (at most the places), operated at only a few places. It first appeared around the small grain Ff at +Y side, Fig. 4.50(h). The density for slip trace C reached to the maximum as the trace of GBeF was parallel to the slip trace C. Slip trace C in grain Fe was observed at GBed close to Y=0 edge and close to the grain boundary triple point. Similarly, C slip trace was also observed at GBeC at +Y side close to the triple grain boundary node.

6). Grain Ff

Grain Ff was a small grain residing inside of the big grain Fe. There were four operated slip systems, A, B, C and D, Fig. 4.49(a).
Slip trace B, with the maximum Schmid factor and RSS was observed all over the grain, and it was distributed uniformly throughout the grain.

Slip trace C, with the third Schmid factor and RSS, nucleated at the grain boundary facing to $+Y$ direction, Fig. 4.50(i). The density of slip trace C along the grain boundary was not as high as those for A and B.

Slip trace D ($1\overline{1}1$), with Schmid factor and RSS ranked fourth, started at the grain boundary facing to $+Y$ side only, Fig. 4.50(i). The density for D at the grain boundary was high. Away from the grain boundary, the spacing between the slip bands increased. Slip trace D disappeared not far from the grain boundary.

7). The stress strain relationships

Fig. 4.52 is a plot of true stress–true strain curve for specimen F. Obviously, because of the existence of the grain boundaries, the stage I was much shortened. Rapidly work–hardening stage II started earlier than those of single crystals and bicrystals. Fig. 4.53 is a plot of $\ln\sigma$–$\ln\epsilon$ curve. Fig. 4.54 is a plot of work–hardening rate versus true strain. It was found that even in stage III, the work–hardening rate is as high as 0.3 at 3% strain, and it tended to increase continuously. Comparing with those bicrystals, the $n$ value in stage III
Fig. 4.52 Stress-strain curve for specimen F
Fig. 4.53 A plot of lnσ-lnε curve for specimen F
Fig. 4.54 A plot of $\frac{d \ln \sigma}{d \ln \varepsilon}$ curve for specimen F
is about 0.15. Hence, it can be concluded that in polycrystalline material, stage I is shortened or even disappeared. Rapid work—hardening is caused by multiple—slip which is induced by the grain boundary.
5.1 Q: Does Schmid law work?

A1: The Schmid law works for many slip systems

In general, the observed slip systems had high ranking Schmid factors. There was a total of 32 grains in 13 specimens. In each grain, the number of the observed slip systems varied from two to five. Totally, there were 91 slip systems observed in the 32 grains. Among the 91 slip systems, 66 slip systems had the highest Schmid factors among the 12 possible slip systems in each grain. Therefore, statistically the Schmid law worked on 72.5% of the slip systems in the aluminum experiments.
A2: The Schmid law is not always valid

The Schmid law obviously did not work on all the slip systems. There were 25 slip systems observed, out of 91 slip systems, with low Schmid factors. Actually, besides the 25 slip systems, some other slip systems with the second or third largest Schmid factors were observed prior to the primary slip systems. For example, in grain Bc, the slip system A (111) having the secondary largest Schmid factor was observed throughout the grain, Fig. 4.1(a), whereas, slip system B, with the highest Schmid factor, was observed only at a highly deformed region. Similarly, in grain C1c, slip system A (\{11\}) with the second highest Schmid factor dominated in the whole grain, but the primary slip system B was observed only at the corner of the grain connected with the slip system C (111) in grain C1a. Similar cases were also observed in other grains, such as grain C3b, grain C4b and grain D1d.

5.2 Q: Does FEM simulation work?

A1: Yes, FEM better than Schmid law

1) For high Schmid factor slip systems

Generally, the FEM calculations showed the same RSS rankings for most of the slip systems as the Schmid law did in a large region in each grain. However, the FEM simulation was able to show the RSS
ranking change at different places within the grain. In grain Ba, for instance, at the grain boundary between grains Ba and Bb at +Y edge, slip system B \((1\bar{1}1)\), with the secondary largest Schmid factor, had the highest RSS, i.e. the FEM calculation predicted that slip system B would be observed. In the experiment, slip B was observed at the same region, although it was unclear which one of the two slip systems A and B operated first. Similarly, in grain Bc, FEM calculations showed that slip system A with the secondary largest Schmid factor had the largest resolved shear stress in a large area, Fig. 4.1(c). It was consistent with the experimental result, because the slip trace density for A was much higher than that for B. This kind of observation is supported by the FEM simulation of Wagoner [206] on the experiments of Hook and Hirth [114—116], where the secondary slip systems had the maximum resolved shear stresses at grain boundaries in several compression bicrystals. This proves that the secondary slip could be activated in the absence of primary slip. This kind of secondary slip was caused by elastic incompatibility at the grain boundary.

2) For slip systems with low Schmid factor

For the slip systems with low Schmid factor, our current FEM simulation showed a better result, especially at the ends of the specimen. For example, in grain C4b, slip system E \((\bar{1}1\bar{1})\) with the Schmid factor ranking of 4th was observed at the \(Z=0\) end in the absence of the primary slip system A, Fig. 4.13(c). Our FEM calculation showed that the RSS ranking increased from 4th to 3rd. This explained why slip
system E was observed only at that position. Similar cases were observed in grains Ba, Bc, C1a, C1b, C2b, C3b, D1c, D2a, E1b, E3b, E4b, E5b, Fa and Fe.

3) For elastic incompatible slip

At grain boundaries, because of elastic incompatibility, the stresses usually increase, especially for secondary slip systems. In the bicrystal experiment of Hook and Hirth [114—118], a lot of secondary slip was caused by the elastic incompatibility at the grain boundary. The FEM simulation showed very good agreement with the experiments [206].

In our aluminum analysis, similar cases can be found. A typical example is specimen B. Because of the elastic incompatibility at the grain boundary between grains Ba and Bb at the +Y edge, the secondary slip system B in grain Ba had a higher RSS at GBab than the primary slip.

Another example is in grain D2b, [Fig. 4.23(a)], slip system C, with the third Schmid factor originated at the grain boundary and propagated into the grain. At the grain boundary, the slip trace density was high. However, in the region away from the boundary, slip density was very low, and slip trace C even disappeared. Along the grain boundary, the density of slip trace C changed greatly. The FEM calculation basically showed the same variation in RSS for slip C. The RSS for C was high at the grain boundary, and rapidly decreased away
from the boundary. RSS reached the maximum at the grain boundary region where the slip system C was observed. Therefore, our current elastic FEM simulations showed not only the stress increase by elastic incompatibility, but also the stress change by changing the grain boundary curvature.

4) For dislocation nucleation and distribution

It is well known that dislocations usually nucleate at grain boundaries, free surfaces, scratches or other imperfections. However, dislocation nucleation can also begin from the interior of a grain [169]. Our FEM simulation showed that the highest RSS region could be at the middle of a grain (but perhaps at the specimen surface). The examples are grains Ba, C1c, and Ff. In our experiments, dislocations usually nucleated at the high RSS regions and with high dislocation densities or slip trace densities. For instance, in grain Ba, the dislocation etch pits density at the grain boundary between grains Ba and Bb at the +Y edge was very high, Fig. 4.2(a), which corresponded to a high RSS region, Fig. 4.1(b). The average maximum RSS and the average dislocation etch pits density in grain Bb were higher than that in the other two grains in specimen B.

In specimen E3, the maximum RSS was generally higher in grain E3b than in grain E3a. After 1% strain, the average slip trace density in grain E3b was higher than that in grain E3a. As strain increased, the difference in the slip trace density decreased. Specimens B, C1, C2
and E2 are some of the similar cases.

Along the grain boundary and in each grain, not only dislocation nucleation, but also the slip trace distribution in the grains can be predicted by FEM analysis. For example, the spacing between the slip traces in grains Ba and Bb along the grain boundary increased as the RSS decreased while traversing from the +Y to −Y directions. Similar phenomena were also observed at the grain boundaries of grains Bb and Bc, grains C2a and C2b, grains E3a and E3b and some others.

As mentioned above, the RSS is not always high at the grain boundary. In specimen G2, for instance, the maximum RSS regions were away from the grain boundary at the edges of the specimen. The dislocation densities were very low at the grain boundary, Fig. 4.8(c), but very high away from the grain boundary, Fig. 4.8(a), corresponding to the high RSS region. The high RSS region and the high slip sites were also found at the region away from the grain boundary in specimen E3.

A2: No, FEM cannot predict all the slip systems

1) For observed low Schmid factor slip systems

In some grains, such as in grains E2a and E3a, the common slip system C with Schmid factor and RSS ranking of 4th (fifth ranking among 12 possible slip systems) was observed even after 1% strain. The
dislocations nucleated away from the grain boundaries. The slip plane for C was (111), which was almost perpendicular to the specimen surface (will be discussed later).

2) For observed high Schmid factor slip systems

In several grains, the slip systems with the maximum Schmid factors and RSS rankings were observed to be suppressed by the secondary slip systems. For example, in grain C3b, although slip system A had the highest Schmid factor and RSS, it appeared only around the small grain. Whereas, slip system B with the secondary Schmid factor and RSS was distributed throughout the entire grain. In grain C4b, the same type primary slip A was connected with slip C (111) in grain C4e. Even though the orientations for grain C3b and grain C4b were the same, the density of the primary slip A was higher in C3b than that in C4b grain. The slip A in grain C4b was therefore constrained even more. This was probably caused by the continuous crossing of the slip A in grain C4b over the grain boundary to connect with slip trace C in grain C4e. Similarly, in grain C1c, slip trace B with the maximum Schmid factor and RSS was observed at a small region at GBac, where slip trace B was found to be connected with slip trace C in grain C1a.

3) Slip density often reached the maximum where the slip trace was parallel to the grain boundary trace
When the slip trace was parallel to the grain boundary trace, the slip density reached the maximum. For example, in specimen E2, at the grain boundary between grain E2c and grain E2b, slip trace density changed as the grain boundary normal changed. When the slip trace A was parallel to the grain boundary trace, its density increased dramatically at grain boundary, Fig. 4.34(e). Away from the grain boundary, the density decreased. But the FEM calculation did not show any change in stress at that region. These phenomena were observed in some other specimens, such as specimens D1, D2, E1 and F. Especially in specimen F, along the grain boundary between grain Fa and grain Fb, different slip trace densities reached the maximum at different portions of the grain boundary as the grain boundary normal changed, Fig. 4.49(a).

It was also observed that dislocation glide preferred to occur at the region away from the grain boundary if the RSS at the grain boundary was the same as that at the region away from the boundary. For example, in grain D1a, the RSS in a large region are of the same. But the slip trace density reached the maximum at the region away from the grain boundary in between the two grains.

5.3 Unusual \{110\} and \{100\} type of slip

5.3.1 Slip caused by free surface effect
The observations of unusual surface slip for both f.c.c. and b.c.c. crystals [185,100] revealed predominant slip on the most favored type of glide planes, \{111\} and \{110\} respectively, but not on the plane with the maximum Schmid factor. It is a so-called image effect [186] which could favor the unusual slip either by enhancing deformation on unusual slip plane or impeding it on the plane with the maximum Schmid factor. The dislocations laying normal to the surface would promote kinks to nucleate and hence favor gliding.

Since aluminum is a face centered cubic material, the slip plane usually is of \{111\} type which is the close packed plane. The unusual slip system defined as slip system occurred on the plane other than \{111\}. There was a report on observing unusual slip in aluminum [100]. In our experiment, a few unusual slip systems were observed. After 6\% strain, slip system B (110)[\overline{1}10] in grain C2a was observed only at the edge of the specimen, Fig. 4.7(a). It was supposed that the unusual slip was caused by the retained stress by cutting the sample, and they might not appear if a higher annealing temperature was chosen. To verify this hypothesis, another single crystal with the same orientation was examined after being annealed at 350\(^\circ\)C for two hours. Recrystallization was observed along one edge of the specimen, because the annealing temperature was 100\(^\circ\)C higher for this specimen than for the previous ones. This implied that one edge of the specimen had a higher stored energy than the other side. The same \{110\} type of slip trace was observed to nucleate in another single crystal which was heat—treated in
the same way as grain C2a followed by deformation in tension. It is obvious that the occurrence of this kind slip was not from the grain boundary effect. This \{110\} type slip probably was due to the free surface effect. If the slip plane was perpendicular to the free surface, the unusual surface slip could be enhanced. In grain C2a, slip plane (101) was almost perpendicular to the surface (\(-0.052, -0.9925, 0.1045\)). That might be the reason why the unusual slip occurred.

As mentioned before, the \{111\} type of slip systems with low Schmid factor might also be caused by free surface effect, since the slip planes were perpendicular to the edge surfaces. For example, in E2a and E3a, slip system C(11\overline{1}) with 4th Schmid factor and RSS was observed after 1% strain, even without existence of primary slip system. The surface normal for E2a and E3a is \((0.636, 0.1993, 0.7431)\) which is almost perpendicular to the plane (11\overline{1}).

5.3.2 Slip originated at primary slip band

Slip trace D with slip plane (101) originated at the high primary slip bands density region in grain C2b, Fig. 4.7(a), which was similar to the case that some slip traces B started at slip band A in grain C1b. The difference was the slip plane for D was not \{111\} type, but \{110\} type. Although slip trace D was not \{111\} type, it had a very high RSS which was just lower than that of slip system B in grain C2b.
5.3.3 Slip caused by continuity for \{111\}, \{110\} and \{100\} types

Several slip systems with low Schmid factor and RSS were observed at grain boundary which were connected with the other slip system having high RSS at the adjacent grain. In specimen C4, for example, there were three pairs of slip lines connected along the grain boundary between grains C4b and C4e. They are slip systems A(111), B(111), and C(111) in grain C4e connected with D(111), C(010) and A(111) in grain C4b, respectively. The Schmid factor and RSS ranking for slip systems C, B in grain C4e, and C, D in grain C4b were low. Although the FEM calculation showed a relative increase in stresses at the grain boundary, the RSS for these slip systems were still ranked low. According to the criterion of Wagoner and Clark for slip transmission at grain boundary [177–180], if the two slip planes in two grains met at the grain boundary and the angle $\beta$ between the two slip traces at grain boundary reached the minimum, dislocations could easily continuously cross over the grain boundary to the next grain. In C4 specimen, the values of the angle $\beta$ for one major continuous slip was found to be the minimum at the grain boundary where the slip continuity was observed, appendix C. These slip continuity might be found only at higher level strain because there was no dislocation connection at the grain boundary at 1% strain, Fig. 4.14(a). Similarly, in specimen F, four pairs of slip continuities were observed. Most of them are of the minimum $\beta$ angles, appendix C.
In specimen E2, there was a slip continuity occurred at the grain boundary between grain E2a and grain E2c. The slip system A (\(\overline{1}11\)) in grain E2a lined up with slip trace D (110) in grain E2c, (The angle \(\beta\) was not minimum at the grain boundary at the place where slip continuity occurred, appendix C). Slip trace D with (110) slip plane was observed only after 4% strain. This indicated that the unusual slip or the slip transmission at grain boundary with low Schmid factor slip systems usually occurred at the high level strain.

5.4 Grain boundary effect

5.4.1 Grain boundary effect on stress—strain curves

In our experiments, all the stress—strain curves showed three stage behavior. Stage I was long and clear in specimens C1, C3, C4, D1, and E1 to E4. It is expected that stage I in specimen F would be very short, because F is a multicrystal with six grains in it. It is surprising that single crystal E5 also had a short stage I. For specimen C2, D2 and E5, the stage II and stage III were indistinguishable.

Fig. 4.16(a) is a plot of the true stress—strain curves for specimens C1—C4 at 1% strain. Comparing the four stress strain curves, the average flow stress for specimen C2 was the highest among all the four curves. Among the four stress—strain curves in C set, the average flow stress for C2 is the highest, Fig. 4.16(b). There were some
differences from Fig. 4.16(a). In the stress strain curves for specimens C2, C3 and C4, the stress dropped at stage I. This phenomenon is believed to be the bicrystal effect, and it is similar to the yield point phenomenon. The first drop was the beginning of large amount of primary slip which released the stress in one grain. As flow stress increased, large amount of primary slip in the secondary grain began. Hence, the flow stress dropped again. Kramer [32,56] believed that this kind of stress drop was caused by removing of the surface hardening layer by electro—polishing of the pre—strained specimen. However, specimens E1 and E2 were directly deformed to 4% strain, and they still had flow stress drops, Fig. 4.46(a—b). Hence, it might not purely the electro—polishing effect. Chuang and Margolin also observed serrations in their bicrystal stress—strain curves [123]. They believed that the jerky stress flow started at onset of plastic flow or sometimes after onset of plastic flow, and the serration depended on the crystal orientation and the specimen size.

In specimen C4, because the small grain was very close to the end of the specimen, there was very little stress drop at stage I. But the interesting thing is that in specimen C1, there was no stress drop. Since there were three crystals, the stress strain curve was more close to that of a polycrystal. In our aluminum specimens, the stress drop usually occurred at less than two percent of strain in stage I or II. After that, the deformation proceeded smoothly.
It was found that the average flow stress for specimen C2 was higher than that for specimen C1. In specimen C2, the grain boundary was inclined 45° to the load direction. In specimen C1, the angle was 60°, and there was another small grain inside the big grain, the flow stress was still lower than that in specimen C2. This indicated that the inclining angle of the grain boundary between the two big grain is very important to the stress—strain curve. Although the grain boundary area for the small grain in specimen C1 was larger than that between the two big grains, the boundary of the small grain did not affect the stress as strongly as the 45° inclining boundary. It was inferred that because there was no transverse grain boundary specimens C3 and C4 as in specimens C1 and C2, the average flow stresses in specimens C3 and C4 were lower than those in specimens C1 and C2.

From both Figs. 4.16(a) and Fig. 4.16(b), it can be seen that the initial yield point did not rise after the 1% pre—strain. Room temperature recovery might be the reason. Grain boundary affected the plastic deformation by producing secondary slip and multiple slip at grain boundary to satisfy the continuity requirement.

Fig. 4.26 shows the stress strain curves for specimens D1 and D2. It is clear that the average flow stress for specimen D1 was much higher than that for specimen D2, although one of the common big grain a had the same orientation in these two specimens. In specimen D2, the average grain boundary plane was roughly parallel to the load
direction. No matter how the grain boundary normal changed, the hardening effect in specimen D2 was less than that in specimen D1 with two half grain sitting inside the big grain a. This was different from specimens C1 and C2 in which the flow stress for the tricrystal was less than that for bicrystal. Hence, it was concluded that if the grain boundary was parallel to the load axis, the grain boundary hardening effect was the least. And if grain boundary plane inclined 45 degrees to the load axis, the bicrystal stress would be the largest among all the bicrystals with different inclining angles. This observation is different from the one observed by Davis [22] in which a 45 degree boundary even reduced the flow stress, comparing with the case of the single crystals. Generally, it is difficult to say that the average stress for bicrystal is higher than that for tricrystals or vice versa. It really depends on the grains orientations and the grain boundary geometry.

The true stress—strain curves for specimens in E set are very close. The yield stresses for E2—E5 are similar, except E1 had a very low yield and flow stresses. Since the grain boundary normal is roughly normal to the load direction in E1, the work—hardening effect might be the least among other orientations, or even less than single crystals, E4 and E5, Fig. 46(a—b).

5.4.2 Relationship between work—hardening rate and strain
From the plot of work-hardening rate \( (n) \) versus strain curves, Fig. 4.18, Fig. 4.28, and Fig. 4.48(a-b), it was found out that at stage II, the work-hardening rate reached maximum for every specimen. Among each set of the specimens, \( n \) values are the largest for tricrystals, for example, C1 in Fig. 4.18, D1 in Fig. 4.28 and E2 in Fig. 4.48(b). Hence, it can be concluded that in stage II, the work-hardening rate is greatly depended on the existence of the grain boundary, and the number of the grain boundaries, although these tricrystals may not necessary have the highest average flow stress which is dependent upon more of the angle between the grain boundary normal and the load axis. After 4% strain, the \( n \) value reached roughly a constant about 0.15, except, for specimen E2 and F, the \( n \) values still have the tendency to increase. Because E2 is a tricystal with a small grain sitting at the grain boundary between the two large grain, and F is a multicrystal, the grain boundaries kept inducing more and more secondary slip to satisfy the continuity requirement.

5.4.3 Triple grain boundary node

At grain boundary triple node, because of the elastic or plastic incompatibility, the stress concentration would occur. For example, in specimen E2, slip system C (111) with the secondary largest Schmid factor in grain E2a was observed only at the grain boundary triple node after 4% strain.
However, the grain boundary triple node might not necessarily cause the stress concentration so that more slip systems would be operated. Many examples showed that at grain boundary triple nodes, the slip densities were low and slip traces were few. In specimen F, there were four grain boundary triple nodes, but none of them caused any stress concentration. At these nodes, the slip densities were low.

5.5 The mesh effect for FEM calculation

The current elastic continuum FEM calculation showed that the grip end boundary conditions were quite symmetric, although the conditions for the two ends were not exactly the same. One end had fixed x, y and z values, whereas the other one had fixed x and y, and prescribed z value. The result will not change much if the specimen is reversed upside down.

Since the specimens were very thin, the grain boundaries were assumed to be straight through the thickness direction and the stress does not vary much through the thickness. Because it was difficult to experimentally observe the difference between the two surfaces, so one layer of elements was used through the thickness (X direction) in the FEM simulation.
For the small grain inside the big one, the rectangular grain shape and exact grain shape with both coarse and fine meshes were examined for grain C1c. The stress contours around the small grain changed. The finer the mesh, the higher the RSS values at the elements along the grain boundary. The operating slip systems were predicted essentially the same. Since eight node brick elements were used in our FEM simulation, it was very difficult to model the exact grain shape without using triangular shape element. To model the small grain, either the six node element or the ten node element should be used. Because the six node element is not allowed in ABAQUS, the question was how to adapt the eight nodes to a triangular element. What we did was to use same node number twice at a given node to meet the eight node requirement, and at same time to simulate a triangular shape element. The program still generated eight Gauss points according to the element size. The coordinates of the Gauss points either were the same or different for the duplicated nodes. Thus, for all the FEM simulations, real grain shapes were used.

5.6 Summary of Conclusions

1) The experiments and the FEM calculations both proved that dislocations could nucleate at the interior of the grain.
2) The slip systems having the maximum Schmid factor calculated from single crystals with the same loading axis may not operate first in bicrystals or tricrystals because of grain boundary effect or free surface effect. They could even be restricted by other operating slip systems.

3) As mentioned before, both the grain boundary curvature and the grain boundary normal direction are very important to the flow stress—strain curve. It is impossible to tell that the flow stress for bicrystals are higher than that for tricrystals or visa versa. It really depends on the grain boundary geometry in a specimen. The angle between the grain boundary and load axis is also a very important factor to the flow stress. For example, if the inclining angle is 45 degree, the flow stress can reach the maximum comparing to that for all other angles, and for a bicrystal with a grain boundary plane parallel to the load direction, the flow stress is probably the lowest.

4) The grain boundary plays an important role in hardening the specimen by inducing multiple slip at grain boundary. Especially, at the beginning of stage II, the work—hardening rate is largely dependent on the number of grain boundaries. Among single crystals, bicrystals and tricrystals, tricrystals are of the highest n value in stage II.

5) In the specimens which contained more than two grains, it was found that multiple slip with low RSS would occur at the grain boundaries to satisfy the compatibility requirement, and the slip systems
would be favored to activate if the grain boundary trace was parallel to the slip trace on the specimen surface.

6) The finite element method (FEM) can model the deformation process quite well. In most cases, it can show a consistence with the experimental results. The FEM simulation is able to explain the grain boundary effect and special grip effect, but not the free surface effect. It can predict not only where the yield occurs first and how the yield propagates, but also the special slip behavior of bicrystals and tricrystals, and the heterogeneous plastic slip along the grain boundaries.

7) Since current FEM simulation is based on the continuum elasticity, it can predict the dislocation distribution on the specimen surface well under a few percent (about 4%) deformation. The grain with a higher RSS would yield first. The dislocation density would be higher in the grain with a larger RSS than that in the other grains. The distribution of slip traces within the grain would be controlled by the RSS in the grain. Within few percent of strain, the current FEM can show a very good prediction. The difference in the slip trace density between two grains or among more grains is reduced as strain increased.

8) In the same RSS region, the slip trace density is often higher at the region away from the grain boundary than that at the grain boundary, because generally the grain boundary is an obstacle to the movement of dislocations.
9) Wedge and triangle shape element can be used to simulate the odd shape boundary using duplicated node number. The slip system predicted by those methods did not show much difference, comparing with that by triangle shape element of similar element size.

10) Evidently not all the slip behavior was caused by the elastic incompatibility. Some slip systems were caused by plastic incompatibility. Some slip systems were caused by the direct slip transmission from one grain to another. Our FEM model cannot predict the slip system activating by slip transmission, although the FEM calculation did show some increasing in RSS along the grain boundaries because of the elastic and plastic incompatibility.

11) Almost all the specimens showed three stages on their stress–strain curves. In some of the bicrystals, including both deformation–free and pre–strained bicrystals, the flow stress dropping was common. This is similar to a yield point phenomenon, once a large amount of dislocations were generated, the stress could be released, and hence, the flow stress dropped. Grain boundary sliding might also be the reason of the stress dropping.

12) \{110\} and \{100\} type of slip was observed in several specimens. The slip is believed to be caused either by free surface and edge effect, or the stress at the highly deformed region and slip transmission.
13) {111} type primary slip systems can be restricted by other low Schmid factor slip, or by slip continuity at the grain boundary.
Appendix (A)

1) Error Analysis

All the orientations of the specimens listed in the appendix B, except specimen B by electron-channeling, were obtained by X-ray Laue method. According to the papers of Pumphrey and Bowkett [234,235], the error involved in the orientation determination by electron diffraction can come from several ways. First of all, the beam direction is a strong function of the angle $\theta$ between the pairs of g-vectors. If the angle $\theta$ is small, the error in $x$ and $y$ could be as high as $\pm 5^\circ$. Secondly, the error can be caused by the voltage varying since the electron wavelength $\lambda$ is proportional to $v^{-1/2}$. But if the voltage is higher than 2KV, $\Delta \lambda/\lambda \leq 1\%$, the error in the beam direction will not be larger than few minutes of arc. Thirdly, the sharpness of the Kikuchi lines can limit the $x$ and $y$ determinations by 0.2 to 0.5mm. There are some other facts, such as the effect of tilting the foil and the electron optical aberrations, were also mentioned in their papers. However, these kind errors are limited. The major error is caused by the actual means of the measurement.
In our electron–channeling orientation determination, the major error in the x and y was believed to be caused by the sharpness of the edge of the specimen. Because of electro–polishing, the edge of the sample may not keep in the same shape after polishing, the tensile axis could change within $\pm 5^\circ$.

Our X–rays orientation determination was done before the electro–polishing. The major error was believed to caused by the way of measurement. First of all, the specimen might not line up exactly parallel to the film. The error can be as large as $\pm 5^\circ$. Secondly, the error caused by transferring the Laue spots from the film to stereodiagram and indexing the spots can be $\pm 2^\circ$. To reduce to error induced by specimen line up with the film, the tensile axis was chosen as the one which fit all the slip traces well after deformation. Hence, the error can almost be eliminated. In our experiment, the average error in x, y and z was about $\pm 2.5^\circ$. 
APPENDIX (A)

2) Grain orientations

The orientation of specimen B.

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The orientations of specimens in set C.

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Appendix (B) ROTATION MATRIX

The rotation matrix present here is the rotation matrix which rotate one local coordinate system to the other local coordinate system. For example, C1ac means the rotation matrix in C1 rotate grain a coordinate system to grain c coordinate. Theta is the rotation angle around axis (H,K,L). The error in Theta is ± 3°.

C1ac

\[
\begin{pmatrix}
-0.2197590 & 0.7838187 & 0.5868480 \\
-0.7186448 & 0.2654832 & -0.6383380 \\
-0.6473260 & -0.5615472 & 0.5139740
\end{pmatrix}
\]

\[
\text{THETA} \quad H \quad K \quad L \\
102. \quad 0.076790 \quad 1.2341740 \quad -1.5024636
\]

C1ab

\[
\begin{pmatrix}
-0.3609926 & 0.2209756 & -0.9000134 \\
0.7608164 & -0.4920463 & -0.4231466 \\
-0.5392969 & -0.8420590 & 0.0095093
\end{pmatrix}
\]

\[
\text{THETA} \quad H \quad K \quad L \\
157. \quad -0.4180124 \quad -0.3667165 \quad 0.5398408
\]

C3bd

\[
\begin{pmatrix}
0.9795797 & -0.1999448 & 0.0210325 \\
0.0480563 & 0.1312126 & -0.9901872 \\
0.1952346 & 0.9709904 & 0.1381292
\end{pmatrix}
\]

\[
\text{THETA} \quad H \quad K \quad L \\
82. \quad 1.9611776 \quad -0.1742021 \quad 0.2480010
\]
C4be

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THETA  H   K   L
87.  -0.9314009  1.2074898  1.2904588

Dlac

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THETA  H   K   L
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123.  -1.2249408  0.4176308  -1.0472429

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THETA  H   K   L
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Appendix (C)

The minimum $\beta$ angle for each continuous slip at grain boundary in each specimen

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<th>Slip in Grain 2</th>
<th>Minimum $\beta$ angle</th>
<th>GB Normal with $\beta_{\text{min}}$</th>
<th>GB normal In Experiment</th>
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<td>A in C3b</td>
<td>D in C3d</td>
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<td>A in C4e</td>
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<td>B in C4e</td>
<td>C in C4b</td>
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<tr>
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<td>A in C4b</td>
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<tr>
<td>F</td>
<td>A in Fc</td>
<td>C in Fc</td>
<td>0.5</td>
<td>88</td>
<td>50</td>
</tr>
</tbody>
</table>

Here, GB normal in experiment means that in experiment, the slip continuity occurred at grain boundary normal inlined an angle to load axis Z. The range of these angle can vary from +5 to +10 degrees.
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