Load Dependent Fatigue Crack Initiation in High Purity Al

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Load Dependent Fatigue Crack Initiation in High Purity Al

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TABLE OF CONTENTS

LIST OF ABBREVIATIONS

GB	grain boundary
PSB	persistent slip band
TEM	transmission electron microscope
SEM	scanning electron microscope
LME	liquid metal embrittlement
EBSD	electron backscatter diffraction

SUMMARY

Fatigue crack initiation sites and mechanisms in metals and alloys have long been investigated, since metal components are often subjected to cyclic loading, and fatigue cracking is one of the major causes of failure. Therefore, understanding the dominant cracking mechanism under different conditions is essential for tailoring the composition and microstructure of metal components for better fatigue resistance under various loading conditions. Load dependent fatigue response in high purity aluminum (Al) is investigated. In low cycle fatigue, extrusions and intrusions are found to form on grain boundaries (GBs), especially prevalently at triples junctions. However, contrary to theories on extrusion formation from persistent slip bands (PSBs), no slip bands are observed in these specimens. Dislocation cells, on the other hand, are observed to form in higher densities and smaller sizes as stress amplitude increases. As extrusion formation occurs only after a threshold number of cycles, it might be a result of the progression of dislocation cell formation. In high cycle fatigue, no extrusions are observed at GBs, while microcracks form within grains. Therefore, high cycle fatigue life may be controlled by mechanisms other than dislocation cell formation, and involves transgranular, rather than intergranular, fracture.

CHAPTER I
INTRODUCTION

Fatigue in metals and alloys is a long-established field, dating back to roughly the beginning of the 20th century. In many industrial applications, metals are subjected to cyclic loading, rendering fatigue cracking one of the most dominant culprits of failure, which thus brings the mechanism of fatigue cracking into metallurgists and materials scientists' attention. As a fatigue life involves the initiation and propagation phases, most research has been devoted to the nucleation and early propagation stage, since an understanding of the crack initiation mechanism helps predict cracking sites and prevent propagation and final failure [1]. Basic fatigue nucleation mechanisms have been proposed and validated; however, studies on the influence of loading factors on mechanistic variations, Al and Al alloys, and crack nucleation sites on inner grains are largely deficient, and thus these topics will be addressed in this project.

Early researches in this area were almost exclusively focused on monocrystalline copper (Cu) due to the simplicity of pure monocrystals. These researches established the most fundamental understanding of fatigue cracking as a result of slip irreversibility. Dislocations are produced and annihilated in the process of gliding. When the amount of multiplication and annihilation are equal, their effects cancel and the slipping process is considered reversible, which is mostly the case in the bulk [2]. However, cross slipping and slipping at or to the surface are partly irreversible, which causes dislocations to accumulate and form structures such as dislocation walls, dislocation cells, and persistent slip bands (PSB) [1]. Such dislocation movement results in plastic strain, and thus cracks nucleate at sites of highest plastic deformation, which are the free surfaces of homogenous single crystals [2]. Polycrystalline Cu, with low stacking fault energy and thus little cross slip, was then widely studied for crack initiation at grain boundaries (GBs). The significant role of interaction between PSBs and GBs was quickly identified. Experiments pointed to the formation of extrusions and intrusions at GB-PSB intersections, and theories on their growth and role in cracking were immediately developed. With the progression of fatigue cycling, plastic strain within PSB reaches a saturation level where dislocations are arranged in a low energy equilibrium configuration [3]. Further plastic deformation can not be accommodated within the PSB, causing dislocations to pile up against the GBs, forming extrusions (and corresponding intrusions). These extrusions are thus stress raisers, or points of concentrated strain, which act as crack nucleation sites [4]. Such is the mechanism for intergranular fracture at GBs.

The above extrusion formation mechanism is relatively well established, with both theoretical foundation and experimental validation. In contrast to Cu, Al has much higher stacking fault energy (SFE) and thus a higher tendency for screw dislocations to cross slip. Such materials are characterized as wavy slip materials, as opposed to planar slip materials such as Cu. Rather than PSBs, dislocation cell structures are more commonly found in wavy slip material, as dislocations are free to cross slip away from a plane of maximum stress concentration [5]. However, as the accumulated total strain increases, dislocation density continuously increase, and finally dislocations become entangled, forming 3D cell structures instead of 2D PSBs. The structural transformation of dislocation cells is closely related to hardening and softening of the material, and is found to be influenced by the plastic strain amplitude and progression of fatigue life [6]. A distinctive difference between the cyclic deformation response of wavy and planar slip materials is the hardening—softening—secondary hardening behavior in wavy slip materials, as opposed to a single saturation of hardening (plateau region in stress strain curve) in planar slip ones [7]. During this process, dislocation cells form from dipoles through entanglement, and reach an equilibrium configuration with high dislocation density on cell walls and few dislocations within the cells [6]. The saturation stress during this equilibrium stage is related to the cell size, with higher stresses leading to smaller cells, a fact almost universally agreed upon by researchers [6, 8-10]. After this stage of lowest energy state, different researches point to different subsequent transformations of the cells: come proposed that cell size and misorientation remains unchanged (as in saturation) [8], some suggest that the cell boundary width decreases and the cells become more equiaxed [11], and some claim that the cells either rearrange or revert to dipole structure to enable softening [6]. Many studies have tried to correlate each stage of fatigue life with dislocation cell structure, while no consensus have been reached. Although some studies pointed to a correspondence between cell structures and PSBs [6, 7], researchers have failed to propose a mechanism for such relationship, nor has direct correlation been found on dislocation cell structure and fatigue crack initiation, although dislocation activities in cells are certain to affect crack nucleation.

In general, Cu and Cu alloys have been the major subject of investigation in the history of fatigue research, despite the extensive usage of Al alloys in many industries, the fatigue behavior of which is especially critical in aerospace applications. Even in the existing studies, observation of cracks has largely been confined to the specimen surface due to the difficulty in accessing inner grains without inducing further deformation. This study solves this problem by employing Gallium (Ga) induced liquid metal embrittlement (LME) to fracture Al grains. Due to similarity in bonding characteristics, Ga atoms easily diffuse into GBs within the Al bulk, weakening the bonds among Al atoms across GBs. The Al grains are thus separated in a brittle fashion without introducing damage to GB topography. In this way, crack initiation sites on GBs of inner grains can be observed.

In this research, high purity Al is used to preclude the influence of different phases, intermetallics and other inclusion particles. Combining observations of extrusions and dislocation cells, this study identified a dependence of preferential cracking mechanism on stress amplitude. At high stress amplitudes, extrusions and intrusions develop on GBs, preferentially at triple junctions, acting as stress raisers and crack initiation sites. At low stress amplitudes, cracks nucleate on interior grains through mechanism independent of surface roughening from dislocation pile-ups. The surfaces show severe deformation from striations possibly from intensified PSBs, since free surfaces are of higher energy. However, no PSBs have been observed with transmission electron microscopy (TEM) in interior grains. Dislocation cells are found in samples fatigued at all stress amplitudes tested, at various stages in fatigue life, with smaller cells and high cell density associated with higher stress amplitude and higher number of cycles. Therefore, dislocation entanglement in cell walls proves a viable mechanism for extrusion formation in low cycle fatigue, analogous to dislocation pile-up at PSB-GB junctions. Mechanism for high cycle fatigue still remains unclear, as no extrusions or other possible stress raisers are observed. As void like structures are observed to form into microcracks on interior grains during high cycle fatigue, point defects such as vacancies and impurities may play a role in crack nucleation under low stress amplitude.

CHAPTER II
LITERATURE REVIEW

Fatigue behavior in metals and alloys refers to the response of the specimen to cyclic loading, usually either monotonic or reversed loading. In general, fatigue behavior involves dislocation multiplication and sliding, slip band and cell formation, GB deformation, and other microstructural changes that lead to crack nucleation and propagation. A typical fatigue life undergoes cyclic hardening, and/ or cyclic softening, fatigue crack nucleation, initial growth on the microcrack scale, propagation as macrocracks and the final failure [1]. Research interest has been largely focused on the early stages, and many mechanisms of fatigue crack initiation have been built, experimentally observed and quantitatively supported.

1. PSB and Extrusion Formation

It is generally agreed that PSB and GB play important roles in crack nucleation and growth. On PSB formation, models were established to characterize their growth. PSB are formed as a result of dislocation movement: under continuously applied cyclic stress, dislocations are rearranged into lower energy configurations through slipping.

Within a PSB, stable edge dislocation configurations, for instance, Tyler lattice, veins, and

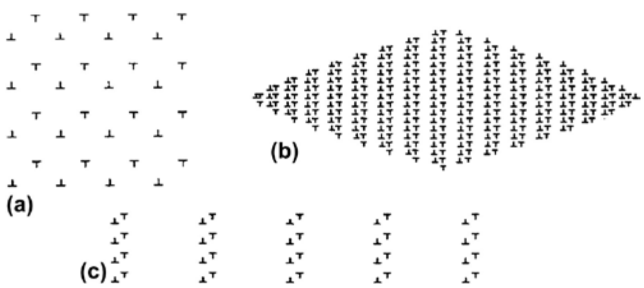


Figure 2: Dislocation structures in PSBs [2]

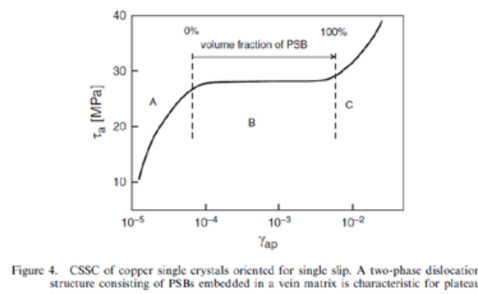


Figure 1: PSB Growth in Fatigue life [3]

ladders (figure 2)[3], involve dipoles resulted from cross slip.

PSB growth starts after a threshold number of cycles, which corresponds to the onset of plastic deformation, and mostly completes in the plateau region on the stress-strain curve, as demonstrated in figure 3 [4, 12]. As pointed out by Lukas and Kunz, PSB growth in polycrystalline materials follow a similar pattern except that the plateau region is not as clearly defined in the σ-ε curve. The beginning of PSB growth occurs after a threshold number of cycles, which a period of rapid growth of PSB height follows. Over the progression of the plastic deformation phase of fatigue life, the number of dislocations saturate in the PSB. As dislocation density increases, an equilibrium minimum mean distance between dislocations is reached, corresponding to the lowest energy configuration [4].

Such configuration can not accommodate more dislocations produced from further plastic deformation, and is resistant to further dislocation movement, causing dislocations to pile up at the end of PSB, where PSB intersects with GBs, which is the well-known and supported dislocation pile-up mechanism [2]. The piled-up dislocations protrude against the neighboring grain, causing extrusions to form at GB-PSB intersections [12]. The EGM model was proposed to characterize this extrusion surface roughening effect: the first phase of which is rapid, and the second growth period is more gradual because it is diffusion controlled. It was found that the extrusion height is proportional to grain size, implying surfaces of larger grains are rougher, and thus more effective stress raisers and more probable preferential cracking sites [5]. It was also suggested by Sangid that PSBs form preferentially at triple points [13]. This claim could be confirmed from SEM images of fatigued high purity Al observed in this study: the largest extrusions and roughest surfaces appear at triple junctions, implying that triple points are among the most favored cracking sites.

The above model was long established and experimentally validated that PSB-GB intersections are among the most common sites for crack nucleation. However, studies have indicated different GB characters as preferential for crack initiation. Twin boundaries, especially those with normal PSBs, were observed to be the most susceptible to cracking from many experiments. This is because twin boundaries are more coherent, and thus of lower energy and act as the highest energy barriers to slip transmission at extrusion sites across grains. In addition, mechanisms such as the surface step plastic incompatibility model, dislocation transfer model, traction model, and elastic incompatibility model were also proposed to elucidate such phenomenon. Finite element analysis was employed to validate the high elastic stress concentration at twin boundaries, and later plastic strain was accounted for in the model as well. It is also observed from experiments with Ni based super alloys that high coincidence lattice site density (low Σ values) GBs are resistant to cracking, while low angle GBs are common cracking sites. The later phenomenon is rationalized by the argument that the similar orientation of the two adjacent grains allows dislocations transmit across GB [3]. It is worth noting that the given explanations are not completely convincing. For instance, since dislocations are allowed to transmit through low angle GBs, no dislocation pile-ups and extrusions are formed, therefore cracks should form at roughed high angle GBs instead, as observed in Zhang and Wang's experiments [14]. Further, the conclusion that low angle GBs and coherent low Σ value GBs and TBs are preferential cracking sites is not a self-consistent one, since low misorientation angle often does not correspond to low Σ value. Σ3 tilt boundaries in the <111> direction in pure Al, for example, is a high angle GB with a low Σ value, as evidenced in Figure 4 [15].

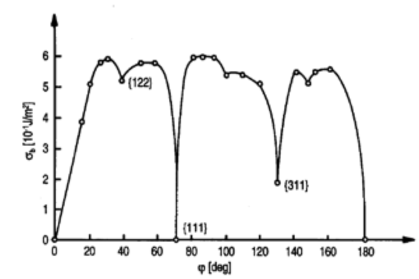


Fig. 2.4. Dependence of the energy of symmetrical <110> tilt boundaries in Al on the tilt angle φ. The indices given in the figure are Miller indices of the corresponding grain boundary planes (see text) (after [14]).

Figure 3: GB Energy Variation with Misorientation [9]

As the high misorientation angle would predict resistance to cracking from this conclusion, its low Σ value indicates the exact opposite according to the same model. In fact, Σ value does not follow the same trend as GB energy either, as GBs of minimum energy occur at various levels of coincidence site density as shown in figure 4, thus the energy approach can not be relied on in providing theoretical support. Various structures on GB surfaces are also observed to be common sites of fracture. Dimples on GB from coalescence of voids within the matrix were often observed on fracturing sites. GB ledges as a result of dislocation pile up, according to Kawabata and Izumi, also favor crack initiation [16]. However, such claim needs to be tested since GB ledges are also formed from heat treatment of the metal specimen, as ledges with the sharpest profile produced in the recovery phase of annealing in pure Al were observed by the author of this thesis. GB ledges are also visible after cyclic deformation, yet it is unclear whether the ledges are formed from plastic deformation in fatigue or the annealing process, so its origin should be examined from dislocation structure under TEM. Overall, GB characteristics favoring cracking still lacks definite conclusion and theoretical explanation, and will be studied in this project.

Orientation of grains and slip systems is also an often researched factor in determining crack nucleation site. Since the highest strain occurs at 45^{o} angle to stress axis, shear bands with the most strain localization and plastic deformation occur in these directions [5]. This theory was experimentally validated with low cycle fatigue of Al7075 when PSB cracking is favored at a temperature around 260 ^{o}C [17]. Therefore, preferential slip system and slip direction should also be investigated in this study, as EBSD analysis will be performed at cracking sites to determine PSB and grain orientations at cracking sites.

While PSB-GB junction is a universally observed fatigue cracking site, other types of crack initiation sites were also reported and studied. Internal cracks, for example, were detected in materials such as polycrystalline Cu, Cu-Zn alloy, and Ti-Fe-O alloy. Microcracks were also found in this study on grain surfaces without extrusion-intrusion deformations [18]. Zhang suggested that intersecting slip bands (SBs) within the grain are responsible for such microcrack development. Microcracks nucleate at points where secondary SBs strike primary SBs, the orientation of which is independent of GB or stress direction, and thus is more common than crack initiation at twin boundaries with parallel SBs [19]. Therefore, it is worth looking for intersecting SBs in Al specimens as well, but it should be noted that this mechanism might not be completely applicable to wavy slip materials such as Al, in which the high stacking fault energy enables cross slip to occur easily and thus intersecting SBs are much more difficult to form. Yokoyama points out another possible mechanism for internal crack formation: internal stress intensification rather than surface roughening by PSBs. At GBs with neighboring recovered and recrystallized grains, strain develops due to the difference in dislocation mobility across the GB. Recovered grains contain more mobile dislocations are thus are softer than recrystallized grains, that is, more plastic deformation can be accommodated in recovered grains. As recrystallized grains have already achieved a low dislocation density equilibrium configuration, yielding occurs first in recrystallized grains and thus a strain incompatibility develops between the yielded and unyielded grain across the GB. Microcracks thus develop in recrystallized grains along GB, which connect and form intergranular fracture [18]. While such argument seems plausible and the microcracks observed in Ti-Fe-O alloy are similar to the ones observed in this study, grain structure should be carefully examined after annealing before fatigue. As the annealing condition was selected for this study to ensure recrystallization and grain growth (see the details in the Methods section), there should be no just-recovered grains in the specimens and other mechanisms should be sought for to explain the development of cracks on smooth inner grain surfaces.

Though rare, a few studies have been performed on external factors affecting the choice of cracking sites. The effects of temperature and strain amplitude were investigated in Li and Marchand's work on low cycle fatigue of Al7075. It was observed that fatigue at room temperature led to cracking only at inclusion particles early in fatigue life, and little localized plastic strain was detected at the cracks. At high temperatures (around 260 ^{o}C), only GB and PSB cracking were found, with GB cracking favored on small grains and PSB cracking favored at 45^{o} to stress axis. This finding is contradictory to the theory that extrusion height is proportional to grain size, indicating preferential cracking at large grains instead. The variation of extrusion height in relation to grain size and cracking density should be noted in this study. At mid-range temperature (180 ^{o}C), however, preferential cracking sites vary with strain amplitude. Under low strain amplitudes, cracks occur at inclusion particles; under high strain amplitudes, cracks develop at GBs. Such variation occurs due to disparity in plastic strain to total strain amplitude. Under low temperatures and low strains at mid-range temperature, most strains are elastic and thus PSBs play no role in crack initiation since it requires plastic deformation. Under such condition, cracks thus initiate at inclusion particles such as intermetallics through bond decohesion under stress. At high temperatures and strain amplitudes, plastic deformation is made possible so the well-known dislocation pile-up mechanism at PSB-GB junctions takes over [17]. A similar distinction in cracking sites is expected under different stress amplitudes in fatigue of high purity Al and other high strength Al alloys under room temperature.

The existing studies paved a solid foundation in the basic mechanism of PSB-GB cracking, however, experimental conclusion and theoretical support on specific microstructural factors remain largely deficient, with many inconclusive or even contradictory. Moreover, many studies performed experimented with Cu and Cu alloys, the conclusions of which are not applicable to Al and Al alloys under most cases due to the huge disparity in stacking fault energy.

2. Dislocation Cell Formation and Transformation

Difference in SFE results in disparity of the ease of cross slip in different materials, and therefore different dislocation structures are preferentially formed during fatigue. Mughrabi summarized a general relationship between the wavy versus planar character of dislocation slipping and dislocation configuration at various strain amplitudes, presented in the following diagram [5]:

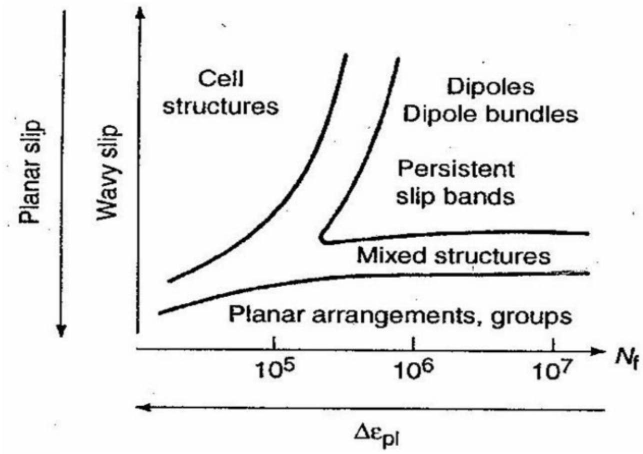


Figure 5: Dependence of fatigue-induced dislocation patterns on cyclic slip mode and fatigue life [5].

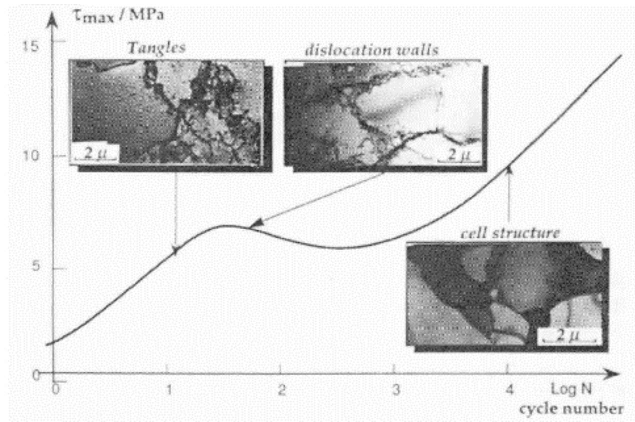


Figure 6: Stages of Fatigue life [1]

For Al, located at the extreme end of wavy slip materials, one would expect to see dislocation dipoles at low strain amplitude, and cells at high strain amplitude. Fougeres further drew correspondence between cyclic stress-strain response, dominant dislocation structure, and number of fatigue cycles in wavy slip materials, as shown in figure 6 [1]. While cell structure evolution is regarded to correlate with hardening and softening behaviors during the progression of fatigue life, the case presented in figure 6 is not agreed upon by researchers. Many research have devoted to cell structure investigation in pure Al in either monocrystalline or polycrystalline form, and some typical results are presented as follows.

The work done by Feltner and Laird in 1967 is considered classical and lays the foundation for almost all subsequent studies. While a main goal of their work is to investigate the effect of temperature and pre-treatments such as annealing and cold working on the cyclic response of Cu and Cu-7.5%, only relevant parts on dislocation structure characterization in the relatively wavy slip material, Cu, under various strain amplitudes are presented here. From experiments, low strain amplitudes are associated with prismatic dislocation loops and cell boundaries comprised of dislocation dipoles. At higher strain amplitudes, the cell sizes are smaller, with more irregular cell walls and more tangled dislocations, as well as few dislocation loops [20]. The initial hardening fatigue response to low strain amplitude (defined as fatigue life $N_f > 10^6$) is analogous to that of stage I in unidirectional stress-strain test, characterized by easy glide. Dislocations tend to form prismatic loops and straight dipoles, especially parallel to <112> in FCC metals. Dipole density increases with total strain, and stress saturation can be achieved as dipoles accumulate even in wavy slip materials. For materials with more wavy character, a higher strain hardening rate dictates, corresponding to a faster generation of dislocations. At the end of hardening stage, stress saturates as dislocation dipole density reaches a certain level. While the constantly reversing strain tend to produce flip-flop motion of elongated prismatic loops, the tangled dislocation dipoles resist such movements, achieving a stage of constant energy and stress level. As for the later softening region, dynamic recovery is achieved through reverting cells to dipoles in a dislocation annihilation process, possible only at stresses above a critical value. For high strain amplitudes, where $N_f < 5\times10^4$, the hardening response is similar to stage II in unidirectional tests. Multiple slip systems are activated, with high dislocation entanglement, rapidly transforming into cells. Dislocation density increases with both strain amplitude and total accumulated strain. For large enough strain amplitudes, the specimen directly enters stage II, with cells formed in the first cycles. This hardening and stress saturation procedure can be explained with the dislocation cell shuttling mechanism, where dislocations are bowled to form loops at the cell walls toward cell interior [21]. This is verified by experiment from TEM imaging of dislocation cells in pure polycrystalline Al by Madhoun et al [10]. Complete reversion to dipoles is unnecessary for softening under high strain amplitude, as cross slip can enable a slight rearrangement of dislocation cells, which in turn causes softening. The authors note that a slower softening rate is associated with more wavy materials, as annihilation of dislocations is not as easy given the high freedom to cross slip. As for intermediate strain amplitudes, which are associated with $N_f \approx 10^5$, long walls of dislocations are formed with high density of dislocation dipoles in the wall, which can be seen as a preliminary stage of cells [6].

However, note that pure Cu was taken as wavy slip material, and Cu-7.5% Al alloy was used as planar slip material in this study. As cross slip is even more immensely favored in Al than in Cu, one should be cautious in applying their conclusion in fatigue of "wavy slip Cu" to that of pure Al.

Besides dipoles, tangles, and cells, rumpled and coarse slip bands were observed on the surface, and Videm and Ryum also observed similar structures on the surface of pure Al polycrystals during fatigue [11, 22]. In Videm's experiments, these slip bands (SBs) appear together with coarse PSBs. SBs and PSBs, respectively, form their own macrobands in various directions and transverse each other. During the softening stage, dislocation density is observed to increase in PSBs, and some SBs grow more prominent than others. Even after polishing, further fatigue reproduces these coarse SB patterns. Cracks are observed to nucleate always at GBs, a result of strain concentration in clustered PSBs in macrobands impinging of GBs [22]. Similar phenomena is observed in Li et al.'s experiments, where SBs gradually cover the entire surface, and deformation band II's replaced by locally distorted SBs. They rationalized this phenomenon in the framework of the hardening process, stating that distorted SBs produces lattice rotation between the macrobands and the matrix, causing strain hardening. Extrusions and intrusions are found on the surface along these bands, and cracks thus nucleate and propagate along the macrobands [7]. As PSBs and cells are both important and prevalent dislocation structures formed during fatigue, their relationship and possible interactions is a critical one, especially on the grounds of crack nucleation, which is the result of dislocation configurations and movements. In Wilsdorf and Laird comprehensive analysis of the fatigue process, dislocation veins, PSBs and cells are subsequent stages of dislocation structure in fatigue as the stress increases (material hardens) under constant strain amplitude. The alignment of dipoles in veins transforms the structure to PSBs, and the onset of multiple slip system activation marks the transfiguration from PSBs to cells. This transforming process of dislocation structure allows a lower dislocation density and thus energy in each new structure, while further cycling concentrates the strain in the newly developed configuration [23, 24]. Therefore, according to this theory, in very wavy materials such as pure Al, cracks only form after cell structures have completely replaced PSBs, reached maximum entanglement in the cell walls and minimum dislocation density in cell interiors, which is contrary to many other experimental findings. Fujii et al.'s research found that different dislocation structures develop under different strain amplitudes. Loop patches are found under low strain amplitudes ($< 10^{-4}$), dislocation walls turn into labyrinth structure for medium amplitudes ($5\times10^{-4} \sim 3\times10^{-3}$), and cells form very early for high amplitudes ($> 3\times10^{-3}$) [25]. Li et al. found simultaneous existence of PSBs and cells, with PSBs only on the surface, and cells being the most common structure [7]. This seems to be consistent with Feltner and Laird's view, who mentioned an unspecified association of the PSB at surface with inner dislocation cells, as cells are found in the interior of the specimens, beneath a certain spacing under the surface slip bands [6, 20]. However, plenty of researchers claim that no PSBs or any SB and labyrinth structures are formed in fatigue of Al [10, 11].

The evolutionary path of cells is also a hotly debated topic. Some researchers found cells forming from the first cycle, and even at very low strain amplitudes [7, 10, 11], while some claim that cells are only formed towards the end of the first hardening stage, or earlier only under low cycle fatigue conditions [6, 20]. Most researchers agree that during hardening, the cell walls reduce in thickness as the loose dislocations entangle and increase in density over accumulated total strain. Dipole clusters are commonly found, and loop structures are gradually replaced by cells. Multiple slip systems are activated during this process, and dislocation accumulation is accompanied by lattice rotation at the cell walls [7, 10, 11]. Reported morphology of cells in subsequent stage, however, differs. Mohamed detailed a change in shape to more equiaxed [11]. Suresh mentions a constant cell size and misorientation from saturation to failure [8]. The Mesh-length theory of hardening, based on mean free path of dislocations, indicates that the cells only shrink to a critical size despite the continued increase in total strain. Further shrinkage is prevented by cross slip of screw dislocations and climb of edge dislocations, which only contribute the entanglement in the cell walls rather than changing the cell size, which is verified in Madhoun's observations [10].

While cell structures have been extensively observed and studied in pure Al specimens, a comprehensive theory on its formation and evolution is still lacking due to the disparate experimental results, in contrast to the consistency found in PSB-GB extrusion formation mechanism, with only disputes on relatively minor points. The correlation between cells and potentially possible PSBs or SBs on surfaces of fatigued specimens is a critical one, especially concerning crack nucleation, yet experimental findings are far from consistent, and no one has attempted to offer a hypothesis on this topic.

CHAPTER III
METHODS AND MATERIALS

The 1mm thick 5N high purity Al plate (>99.99% Al) is cut into dogbone samples with gage length 2cm, and gage width 4mm. The samples are mechanically polished and annealed at 400^{o}C for 1 hour, and quenched in water. Previous research on GB ledge formation in relationship to annealing conditions indicates that heat treatment at this medium temperature and time yields the most regular grain surfaces, mostly free of lumps, dimples and other structures that may interfere with fatigue response. Fatigued tests are run on the annealed samples with Instron 5848 tensile tester at various stress amplitudes above and below the yield strength with the same R ratio of 0.1 and frequency of 1 Hz. For SEM sample preparation, liquid Ga is applied on the fatigued specimens at 30~40^{o}C and the specimen is allowed to sit in Ga for 4~5 hours on the hotplate for the Ga to completely penetrate into the GBs. Such temperature is selected based on the phase diagram of Al-Ga as shown in figure 7. Then, excessive Ga is removed with wipers and the Al grains are broken apart by tweezers, based on experiments done by Patterson [26]. SEM imaging is done with Hitachi SU8010 Field Emission (FE) SEM with secondary electrons. TEM samples are cut from the specimen with a razor blade, mechanically polished and jet electropolished with a 90% ethanol, 10% perchloric acid solution, and analyzed in a FEI Tecnai F30 TEM at 300kV.

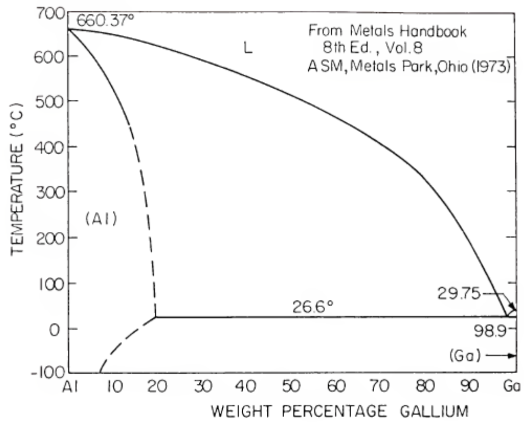


Figure 7: Al-Ga phase diagram [26]

CHAPTER IV
RESULTS

Uniaxial tensile tests of the high purity Al dogbone samples gives the following true stress-strain curve (Figure 8). Deviation from linearity, indicating accumulation of plastic strain, starts below 30 MPa. Based on this data, stress controlled fatigue tests were run, which are summarized in table 1:

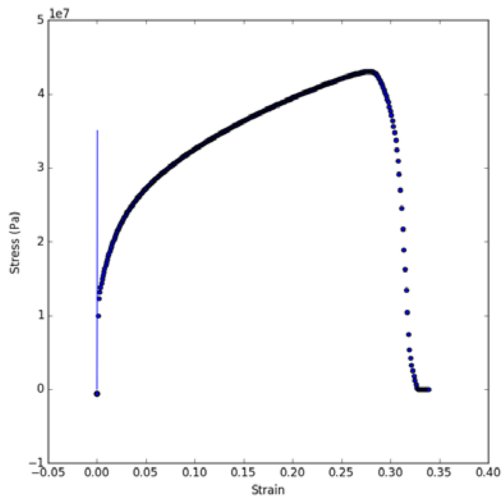


Figure 8: True stress-strain curve from uniaxial tensile test

Table 1: Fatigue test parameters

Speciment No.	Load (N)	Stress amplitude (MPa)*	Number of cycles	Failure
1	14~140	3.15~31.5	6957	Yes
2	8~80	1.8~18.0	235600	No
3	14~140	3.15~31.5	1000	No
4	14~140	3.15~31.5	3000	No
5	14~140	3.15~31.5	3000	No
6	14~140	3.15~31.5	7792	Yes
7	10~100	2.27~22.7	5000	No
8	10~100	2.27~22.7	691080	No

*Stress amplitude is engineering stress, calculated with A_0 = 4.4 mm^2

1. Grain Boundary Morphology Observation with Scanning Electron Microscopy

With SEM, it is observed that GBs are hardly deformed under low stress amplitude even after many number of cycles while GB deformation occurs extensively under high stress amplitude early in fatigue life. Figure 9 represents a typical triple junction and grain surfaces obtained under in high cycle fatigue (specimen #2, 8~80N). The grain surfaces are very smooth; both the GBs and triple junction are sharp, exhibiting similar morphology as that directly after annealing. Severe deformation occurs at GB very early in low cycle fatigue. Figure 10 shows the uneven GB and ragged triple junctions formed after 1000 cycles at 14~140N, and similarly after 3000 cycles (Figure 11).

Extrusion formation at GBs are observed with SEM. No extrusions have been observed on the high cycle fatigue sample. For specimens subjected to 14~140 N stress, extrusions are observed on GBs after 3000 cycles, but not after 1000 cycles. From Figure 10, only general GB deformation occurred, and no concentrated regions of extrusion-instrusion pattern have been found. Such patterns, however, begin to appear in Figure 11, particularly in the area highlighted in the blue box, where the “hills and valleys” are of greater height than the generally deformed surroundings. More examplar extrusions have been observed in the same specimen, fatigued at 14~140N after 3000 cycles. Figure 12 shows a group of SEM micrographs of extrusion formed on GBs in this specimen, preferentially at triple points, with the right hand side image showing a close up view of the extrusion region in the left image in Figure 12 (b). Such extrusion structure exists prevalently across GB triple junctions.

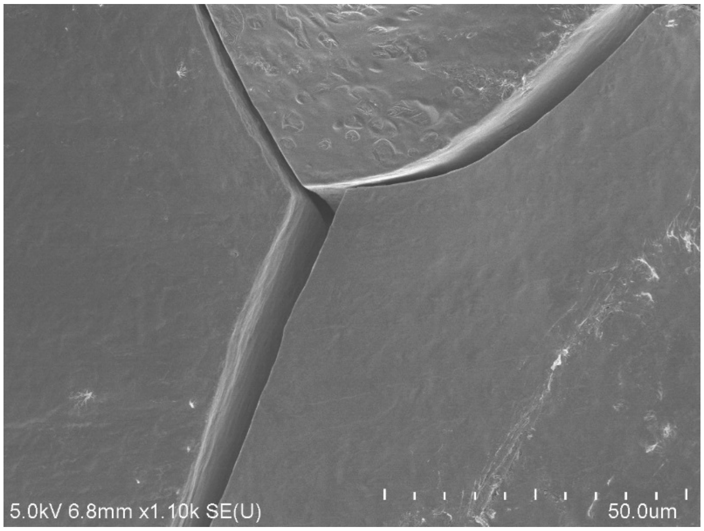


Figure 9: GB morphology after 235600 cycles at 8~80 N

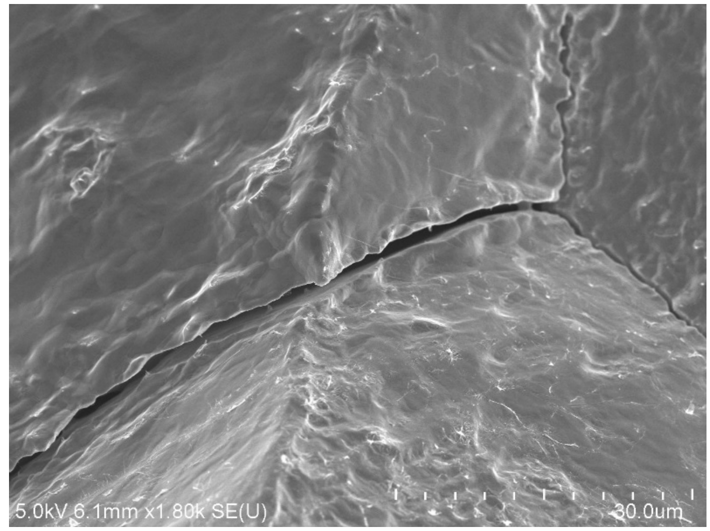


Figure 10: GB morphology after 1000 cycles at 14~140 N

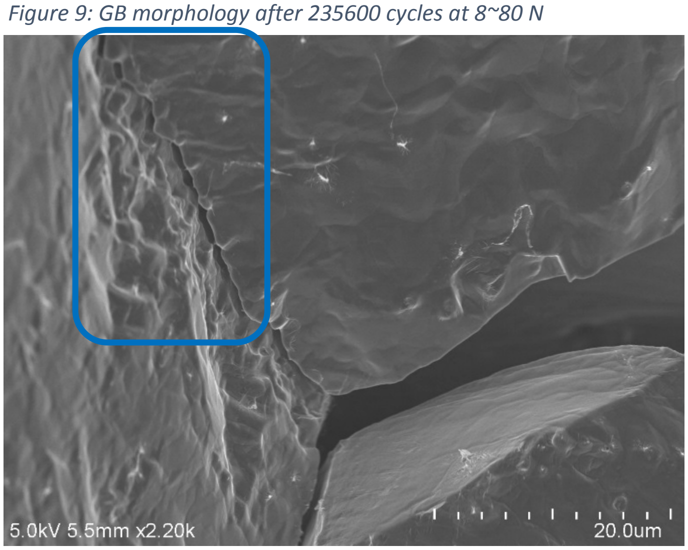


Figure 11: GB morphology after 3000 cycles at 14~140 N

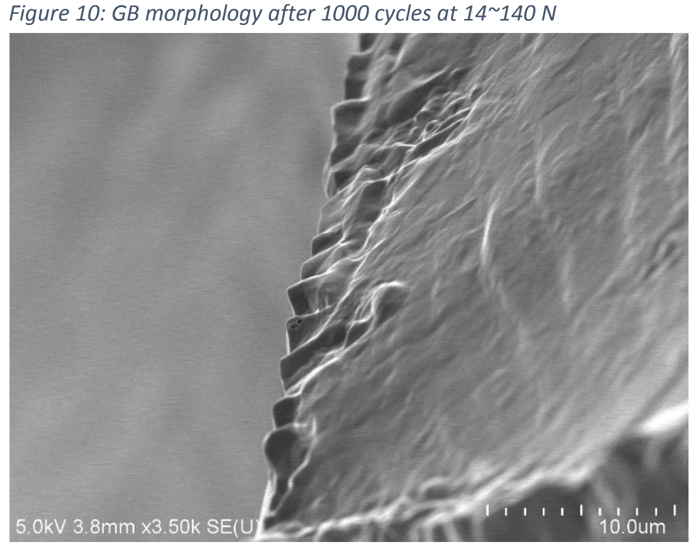


Figure 12 (a): Extrusions at triple junction after 3000 cycles at 14~140 N

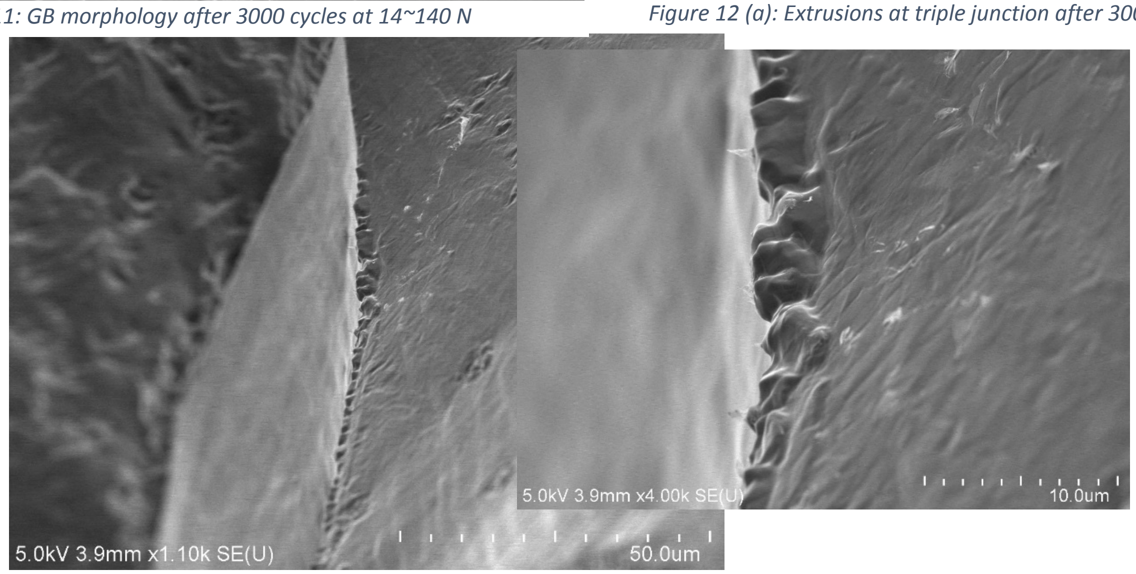


Figure 42 (b): Extrusions at triple junction after 3000 cycles at 14~140 N

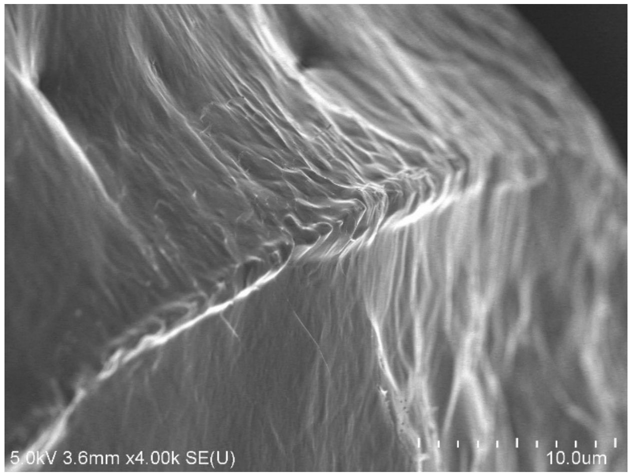


Figure 12 (c): Extrusions at triple junction, forming a ridge, after 3000 cycles at 14~140 N

As for stress amplitudes in the middle range, structures similar to extrusions are found, but their morphology are not as distinct as those in Figure 12. After 5000 cycles at 10~100 N, most GBs are very sharp, as shown in Figure 13. Though very rare, some concentrated deformation profile was observed at a few triple points, for example, the region shown in Figure 14, possibly a preliminary stage of extrusion formation.

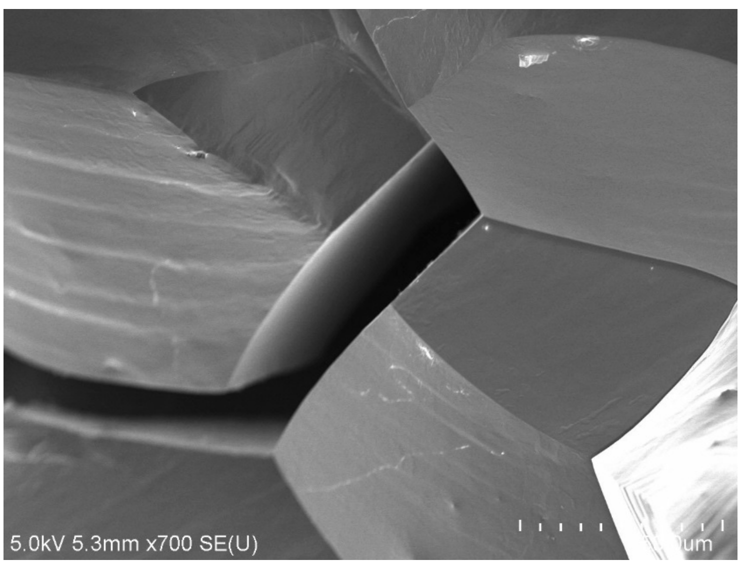


Figure 13: GB morphology after 5000 cycles at 10~100 N

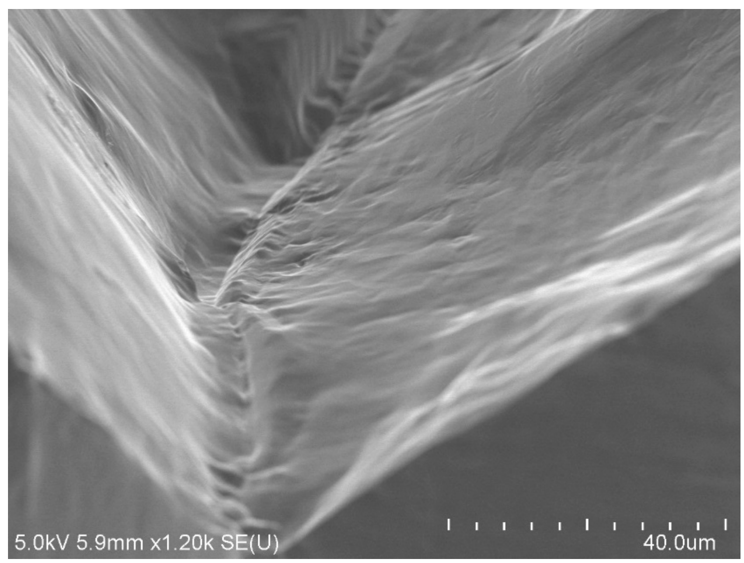


Figure 14: A shallow profile of region of concentrated deformation after 5000 cycles at 10~100 N

The specimen subjected to 10~100 N stress and cycled after 691080 cycles displays large scale GB deformation, as demonstrated in Figure 15. Some GB surfaces, however, remain mostly smooth. Extrusion structures are very rare, but more distinct than that in Figure 14. These extrusions are generally more irregular and have lower heights than the completely formed extrusions in specimen #4 and #5 (14~140 N, 3000 cycles). Almost all these extrusions are formed at triple junctions, the preferential dislocation pile-up site.

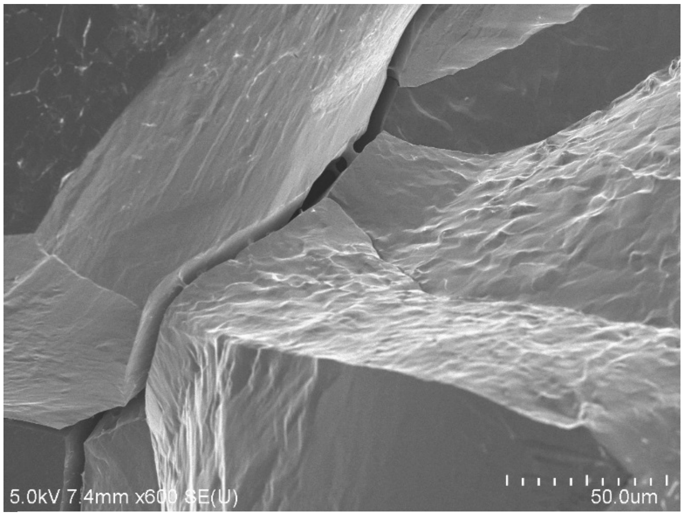


Figure 15: GB morphology after 691080 cycles at 10~100 N

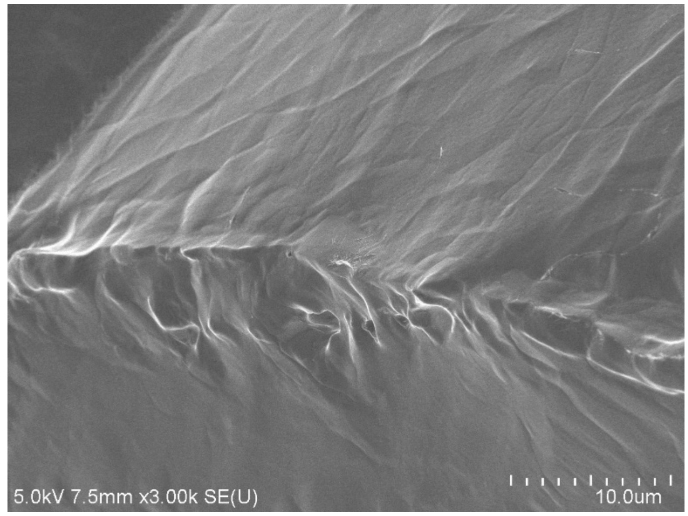


Figure 16 (a): Extrusions starting to form at GB after 691080 cycles at 10~100 N

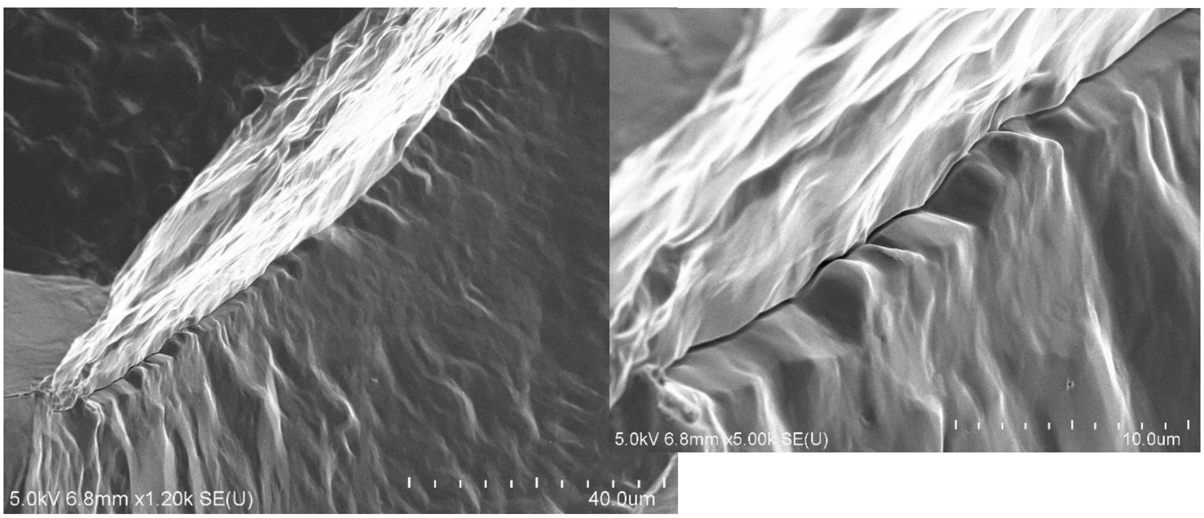


Figure 16 (b): Extrusions starting to form at GB after 691080 cycles at 10~100 N

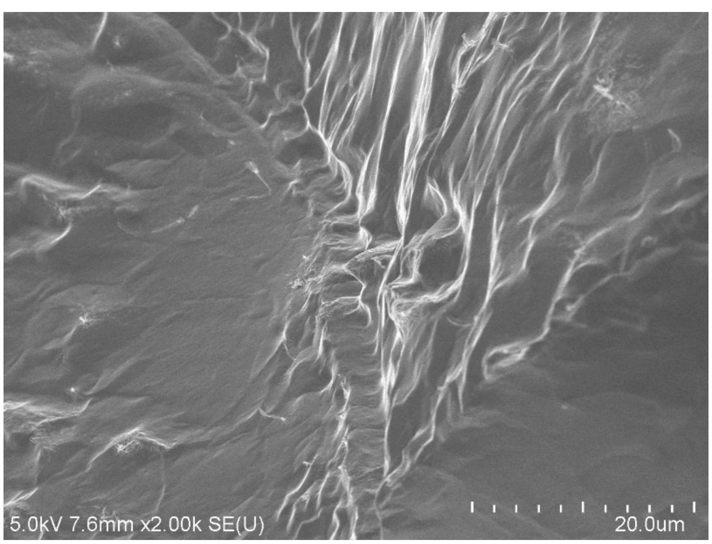


Figure 16 (c): Extrusions starting to form at GB after 691080 cycles at 10~100 N

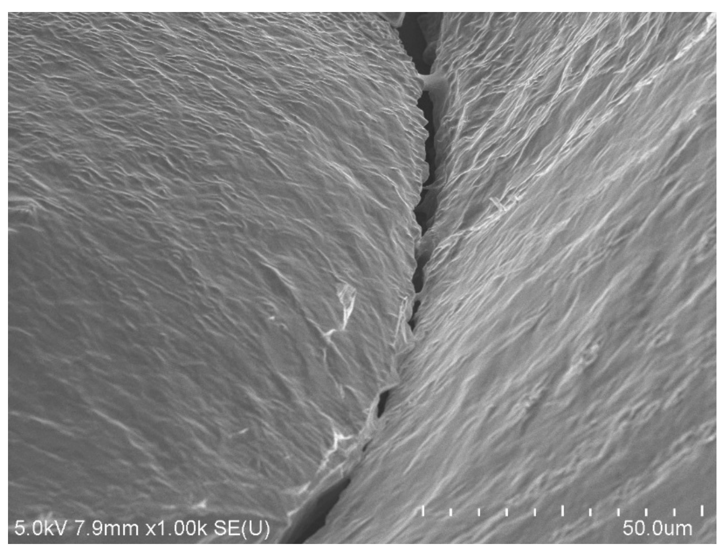


Figure 16 (d): Extrusions starting to form at GB after 691080 cycles at 10~100 N

Cracks are only observed in low cycle fatigue specimens (14~140N) after 3000 cycles, where extrusions have fully developed. Some cracks are observed to nucleate at points where extrusion on GB triple junction emerges at free surface, as shown in figure 17, in which the left side is the edge of the specimen surface. The crack propagates along the line of extrusions on the GB inwards, forming a intergranular crack.

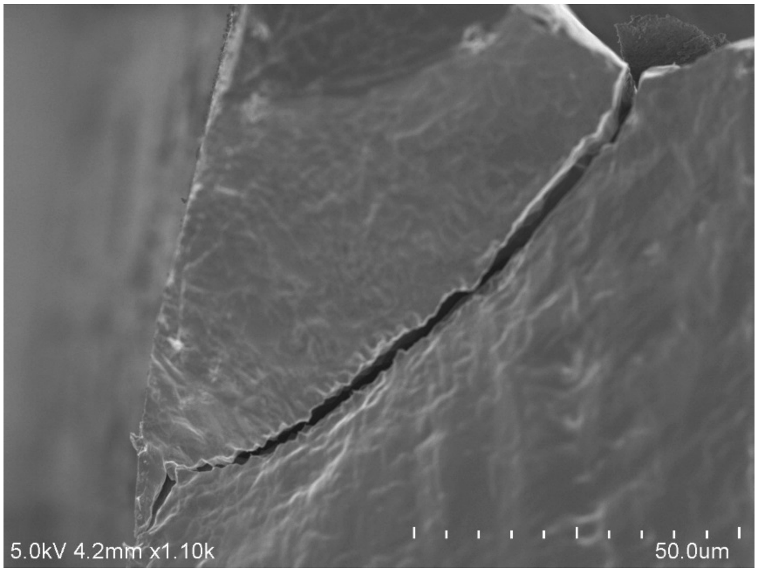


Figure 17: A crack nucleating at and propagating along GB extrusions

Other types of defect structures, possibly related to cracking, are observed for high cycle fatigue. "Microcracks" of length on the scale of a few microns are found on inner grain surfaces, as in Figure 18, possibly resulted from migration and condensation of voids. Similar structures are found in low cycle fatigue, but such structures are of little influence in crack nucleation and propagation, as the cracks formed at extrusions are much larger in size: comparing Figure 17 to Figure 19, the crack at the extrusions are more then ten times longer than the inner microcrack clusters. Though these microcracks develop earlier than extrusions, which are not present after only 1000 cycles, extrusions predominates subsequent defomation and cracking mechanism once they are formed. Under medium stress amplitude,

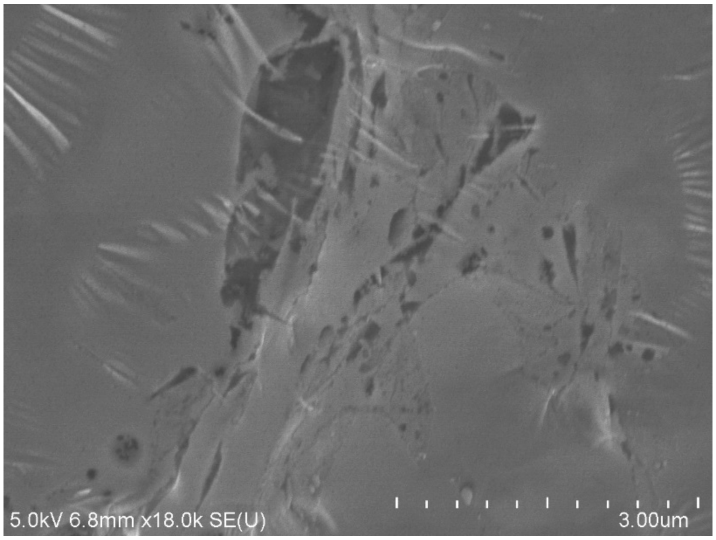


Figure 18: Microcracks on inner grains in high cycle fatigue, after 235600 cycles at 8~80 N

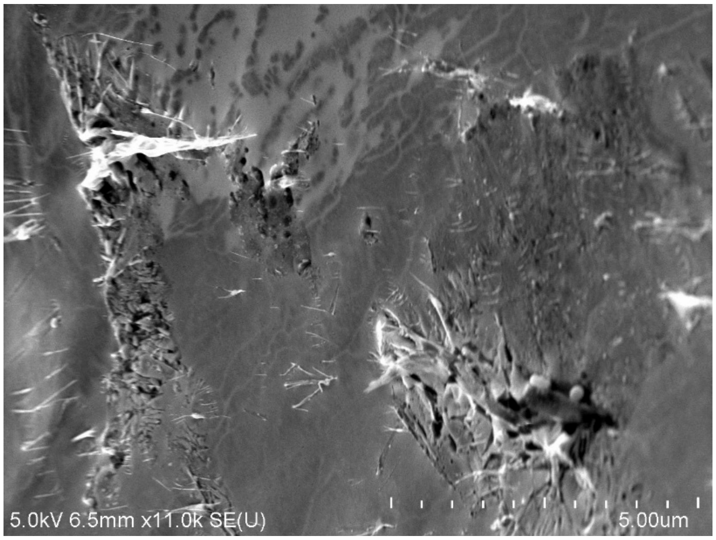


Figure 19: Microcracks on inner grains in low cycle fatigue, after 1000 cycles at 14~140 N

structures possibly related to voids and suspiciously similar to microcracks are found, also of similar sizes, as shown in Figure 20 and 21.

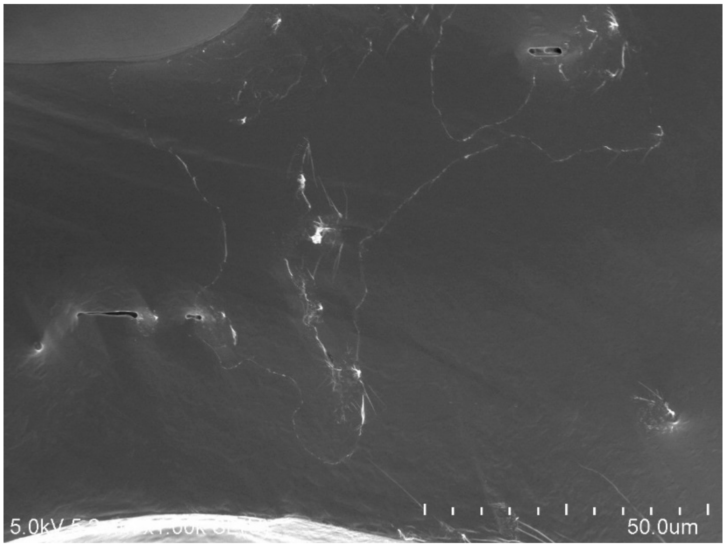


Figure 20: Void/microcrack-like structures after 5000 cycles at 10~100 N

Figure 21: Void/microcrack-like structures after 691080 cycles at 10~100 N

2. Dislocation Structure Observation with Transmission Electron Microscopy

TEM is used to observe dislocation structures in these fatigued samples. Dislocation cells are found resulting from low cycle fatigue. Cycled at 14~140N for 3000 cycles, dislocation cells of the size of several microns are formed, as shown in Figure 22. Scanning-transmission electron microscopy (STEM) indicates some interaction between dislocations and GBs, but no slip bands or dislocation dipole structures are found, as shown in Figure 23.

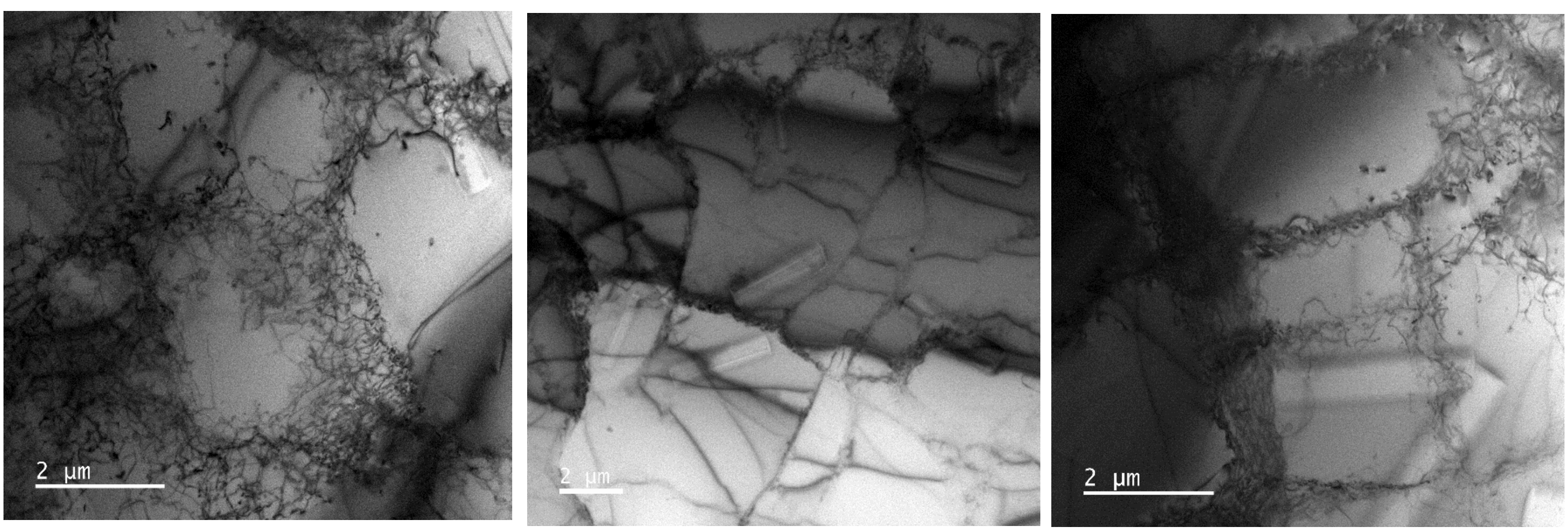
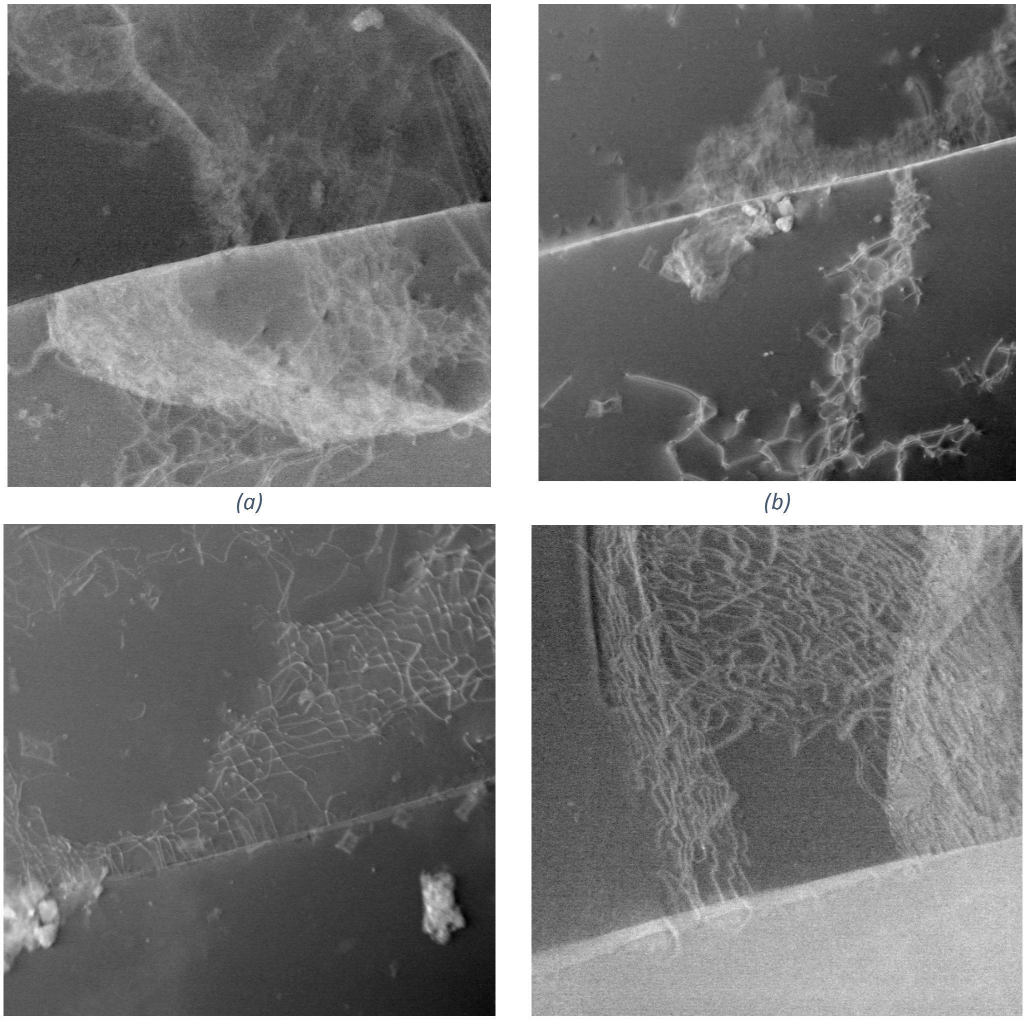


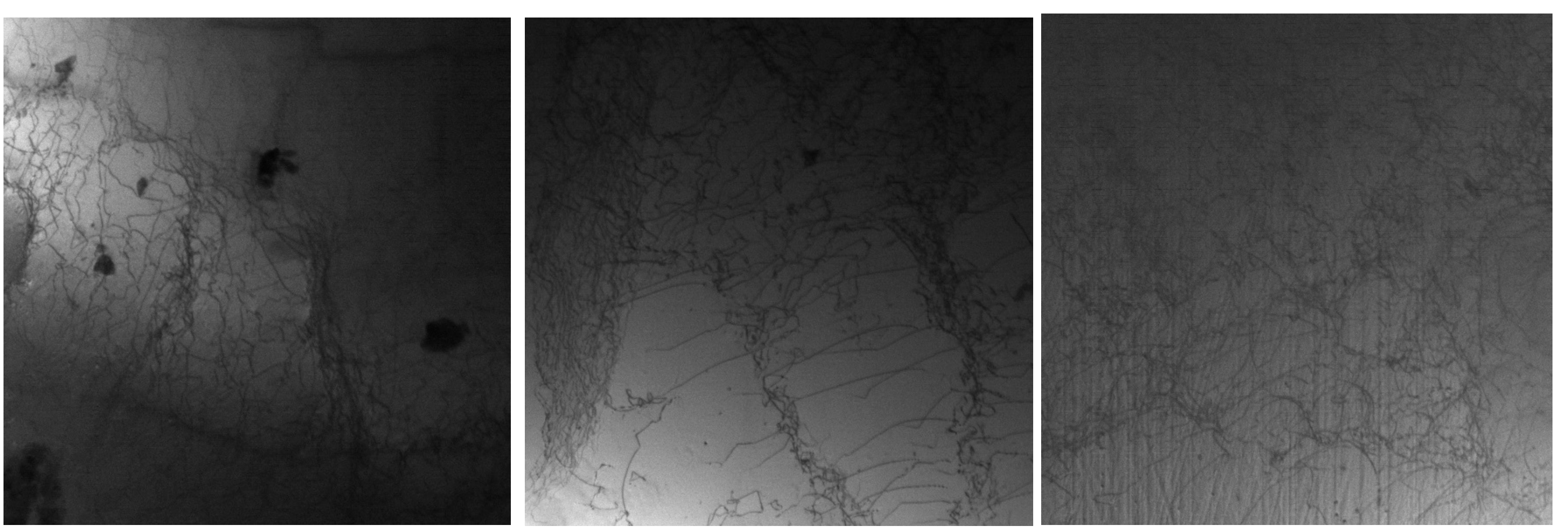
Figure 22: Dislocation cells formed under low cycle fatigue (3000 cycles at 14~140N)



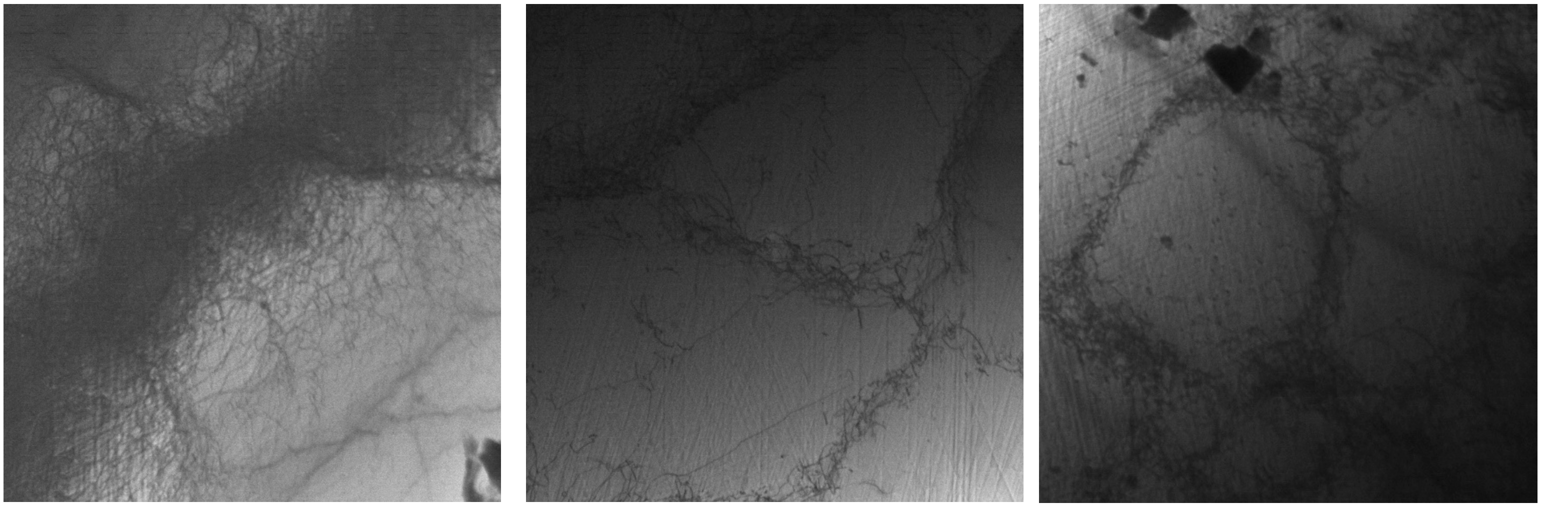
(a) *(b)* *(c)* *(d)*

Figure 23: (a)-(d) STEM images of dislocation-GB interaction from low cycle fatigue (14~140N after 3000 cycles). Dimension of the images are: (a) and (b): 3.56 x 3.56 µm; (c) and (d): 2.51 x 2.51 µm

At medium stress amplitudes, dislocations cells have also been found. Compared to those under low cycle fatigue, more dislocations exist within the cell, and the cell walls are less entangled in terms of



(a) *(b)* *(c)*

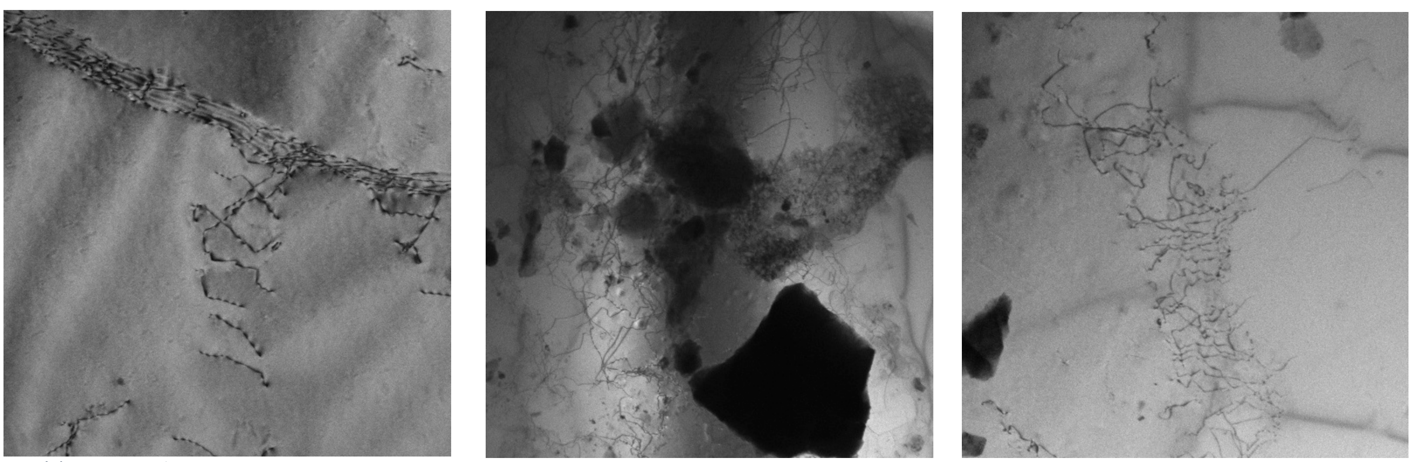


(d) (e) (f)

Figure 24: Dislocation structures formed after 5000 cycles at 10~100 N, 7.87 x 7.87 µm for all images. (a) and (b): Dislocation cells with relatively high dislocation density within cells; (c) and (d): relatively random dislocation distributioin; (e) and (f): larger dislocation cells with low dislocation density on cell walls.

dislocation arrangements. Some regions formed cells larger in size, others show no particular arrangement of dislocations (Figure 24).

In specimens subjected to high cycle fatigue, dislocation density is significantly lower, even after a much larger number of cycles. No recognizable structures have been observed (Figure 25).



(c) Image dimension: 3.16 x 3.16 µm *(a) Image dimension: 9.28 x 9.28 µm* *(b) Image dimension: 3.94 x 3.94 µm*

Figure 25: TEM micrographs of high cycle fatigued specimen: after 235600 cycles at 8~80 N.

CHAPTER V
DISCUSSION

Extrusions have been identified as a site for fatigue crack initiation in low cycle fatigue, whereas no extrusions are found in high cycle fatigue. Therefore, a critical stress amplitude close to the yield strength of high purity Al may exist for extrusions to form. Extrusions are preferentially formed at triple points along GBs, and their appearance correspond fairly well to the evolution of dislocation cells within the grains. At low stress amplitude (8~80N), with maximum stress below yield point, no dislocation cell structures are formed. Other proposed structures, such as dipole walls, labyrinth, PSBs and SBs, are not found, either. The overall dislocation density is too low to form such structures resulted from concentrated plastic strain. While the structure in Figure 25(a) resemble that of GB ledge, which may act as dislocation sources [27], and is indeed interacting with, either emitting or acting as a sink for, dislocations, the dislocation structure present does not provide a large enough misorientation to be characterized as a GB. The nature of such structure is still unknown; since GB ledges can be formed during annealing and from deformation [16, 27, 28], such structures will be further investigated with TEM, combined with SEM observations on ledges on grain surfaces. Cells of early stage, according to previous researches [6, 8-10, 23, 25, 29], are formed around 5000 cycles under 10~100 N, the middle range stress amplitude close to the yield strength. These cells are characterized by low level of entanglement in cell walls, presence of dislocations within the cells, larger cell sizes, and relatively thick and not well-defined shape of cell walls. The presence of random dislocation tangles also indicate that only the beginning stage of cell formation has been reached, since if strain concentration is completely accomplished with the cell structures, all dislocations would be arranged into dense entanglements along the cell wall to maximize their mean free path, according to the Mesh-Length theory of hardening [10, 23]. The corresponding extrusion structures observed with SEM are also not fully developed, with irregular and relatively shallow height distribution in addition to their very rare presence. Under low cycle fatigue, however, extrusions are prevalent across GBs, especially preferentially formed at triple junctions, with clear, ragged morphology and are effective stress raisers. Cracks have been observed to nucleate on the surface where extrusions on GB emerge, the point of highest energy and stress concentration. Intergranular fracture is preferred since cracks can easily propagate along extrusions and intrusions on grain surfaces. Dislocation cells may play a role in the process of extrusion formation and crack nucleation, as they form in patches that are closely packed across the whole grain. Dislocation density within the cell boundary is significantly higher than those formed in lower stress amplitudes, while that inside the cells are significantly lower, both indicating a later stage in cell formation. The mechanism of cell formation and the dislocation structures from which cell evolved, however, are unclear as no other structures have been observed. However, from the STEM image (Figure 26) of cells formed under high stress amplitude (14~140 N, after 3000 cycles), dislocations in some cell boundaries appear more regularly packed than others in terms of orientation and spacing. From such discrepancy, it may be inferred that one type of arrangement precedes the other, and this process have to be investigated in subsequent work. Other types are defect structures are also found in low cycle fatigue, such as the microcrack-like voids in Figure 19. These structures are grow and connect faster, resulting in larger sizes than those formed in low cycle fatigue (Figure 18) due to the larger stress amplitude and thus faster plastic strain accumulation. However, compared with extrusions and cracking at extrusions (Figure 12 and 17), they are an order of magnitude smaller. Therefore, cracking at extrusions is the dominating fatigue crack initiation mechanism in low cycle fatigue, that is, at stresses higher than the critical stress amplitude for extrusion formation.

A critical number of cycles is also required for extrusions to form under low cycle fatigue. This is demonstrated by the lack of extrusions after the first 1000 cycles, compared to the extensive appearance of extrusions after 3000 cycles at the same stress amplitude (Figure 10 and 12). This critical number of cycles is a function of stress amplitude: more cycles are required to form extrusions at lower stress amplitudes. At the stress amplitude of 10~100 N, GBs are almost perfectly sharp after 5000 cycles, while extrusions only start to form at around 691080 cycles, a large rise in threshold compared to the 3000 cycles for 14~140 N fatigue. No quantitative conclusions have been drawn on the relationship between threshold number of cycles, fatigue life, stress amplitude and critical stress amplitude, and this will also be investigated in future work.

For high cycle fatigue, extrusion formation is not possible, so alternative crack initiation mechanism takes over. Condensation of voids into microcracks is the most dominant defect structure observed under low stress amplitudes. With movement of vacancies facilitated by dislocation climb under cyclic deformation, these microcracks grow and connect with adjacent ones, finally reaching a critical size with stress at crack tip sufficient to enable subsequent propagation and final failure. Since such process requires diffusion, it is relatively slow and thus the fatigue life is very long compared to those terminated by cracking at extrusions. As these microcracks are formed on interior grains, transgranular cracks develop, as opposed to prevailing intergranular cracking at extrusions in low cycle fatigue.

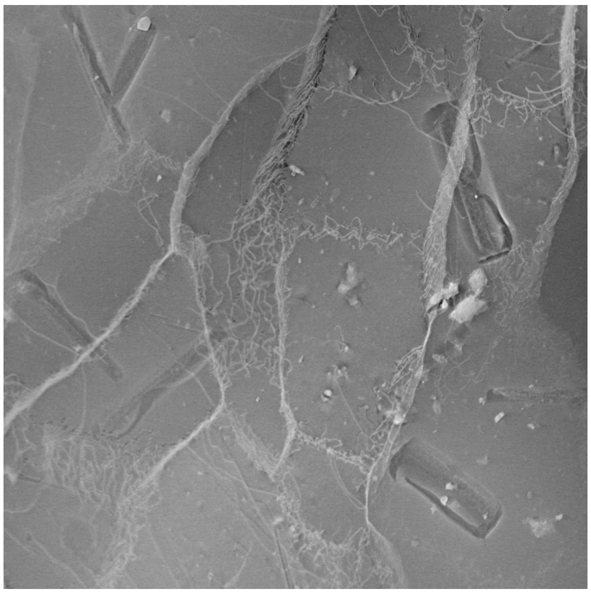


Figure 26: STEM image of dislocation cell structures after 3000 cycles at 14~140 N. Image dimension: 10.9 x 10.9 µm.

CHAPTER VI
CONCLUSION

High purity Al is subjected to fatigue at various stress amplitudes above, below and around its yield point, and the specimens were studied with SEM, TEM, and STEM at various points in fatigue life. The following conclusions can be drawn:

1) Low cycle fatigue is dictated by crack initiation at extrusions, preferentially at triple junctions. Resulting fracture are predominantly intergranular, typically nucleating near surface and propagating inwards along extrusions. High cycle fatigue is controlled by microcrack formation from void condensation, with much lower growth rate on inner grains, leading to transgranular cracks.
2) A critical stress amplitude and threshold number of cycles exist for extrusion formation, above which cracking at extrusions dominates fatigue failure, and below which microcracks govern high cycle fatigue life.
3) Dislocation cell formation is correlated with extrusion formation in low cycle fatigue. Cells form only above a threshold stress amplitude, and occur in the progression of fatigue life comparable to extrusion development. In its evolution, cell boundaries exhibit differing levels of orientation and spacing regularity, evolve towards greater entanglement within cell walls and lower dislocation density in cell interior.
4) Dislocation density in high cycle fatigue is significantly lower than that of low cycle fatigue, and assume relatively random configurations. No other structures of dislocations have been observed, including dipole walls, PSBs, SBs, labyrinth reported in literature.

Much work is still needed to develop a relatively complete theory on fatigue crack initiation in pure polycrystalline Al. The critical stress amplitude for extrusion formation, with its relationship to the yield strength will be investigated. It will also be correlated with the threshold number of cycles for extrusion formation, and its relationship to fatigue life. The role of GB ledges and microcracks are still largely unclear, and needs to be examined with SEM at various points in fatigue life. The path of evolvement for dislocation cells needs to be clarified, and we attempt to bridge dislocation cell structures observed in TEM with extrusion and crack formation observed in SEM to explore the role of dislocation cells in extrusion formation and crack development. Future work will also include another aspect of fatigue: strain controlled fatigue. We will replicate test conditions of previous research on dislocation cell, and employ the same approach with combined SEM and TEM observations to complete the theory on cell formation and fatigue crack nucleation mechanisms.

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