GRAIN BOUNDARY MORPHOLOGY EVOLUTION IN LOW CYCLE FATIGUE OF HIGH PURITY ALUMINUM

Xueqiao Wang[1], Wade Lanning[1], Christopher Muhlstein[1], Josh Kacher[1]

[1]Georgia Institute of Technology (School of Materials Science and Engineering, 771 Ferst Dr NW, Atlanta, GA 30332, USA)

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Abstract

In this paper, an approach for characterizing the evolving grain boundary morphology state of Al under fatigue loading conditions is demonstrated. Although grain boundaries have often been correlated with fatigue crack initiation, most past studies on fatigue-induced microstructure variations have been confined to surface or thin-film based characterization, leaving the internal morphological evolution of the material unknown. This study employs a method of Ga-induced liquid metal embrittlement (LME) to preserve the grain boundary (GB) morphology for scanning electron microscopy (SEM) characterization at various points in fatigue life. This enables in-depth characterization of the GB structure at different stages of a material's fatigue life. To demonstrate this new approach, the evolving GB topographical state and the associated dislocation configurations were investigated at different stages of low cycle fatigue life in high purity Al. GB deformation structures such as ledges, extrusions and voids were observed with SEM. Dislocation structures were characterized by transmission electron microscopy (TEM). It was found that the formation of ledges, extrusions and dislocation cells occur at approximately the same time and that their formation is sensitive to the stress amplitude, with GB ledges and triple junction extrusions forming after the first few cycles when loaded at high stress amplitudes.

Introduction

Past studies of fatigue damage accumulation in face centered cubic (FCC) metals and alloys have shown that under certain loading conditions, damage initiation occurs via surface roughening through strain localization at persistent slip bands (PSBs) and irreversible slip accumulation at surfaces [1, 2]. Under cyclic loading, dislocations constantly undergo multiplication and mutual annihilation. For screw dislocations or components, this process is mostly reversible due to the ability to cross slip; for edge dislocations, however, a surface step is generated irreversibly once they glide to the surface. Edge components are therefore built up along slip directions near the surface, forming persistent slip bands (PSBs). The dislocations emerged at the surface roughen the surface topography, causing extrusions and intrusions to form. Early experiments on copper (Cu) single crystals corroborated this theory, and later studies on Cu polycrystals and alloys demonstrated that the same mechanism applies to grain boundary (GB) roughening, with fatigue damage tending to accumulate at GBs (Fig. 1) [2-5].

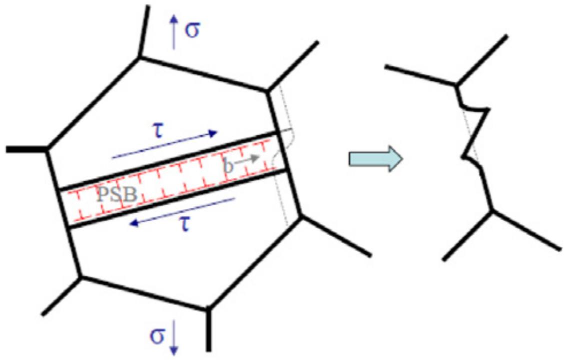


Figure 1. PSB-GB interaction: irreversible slip and dislocation pile-up at GB causes GB roughening by extrusion formation. Image modified and used with permission from [5].

As for research on dislocation structures in fatigue of Al, little consensus is reached on the mechanism. Based on past studies on strain controlled fatigue, it is generally agreed that cells constitute the major dislocation structure; under larger plastic strains, dislocation cell size is smaller and cell boundaries are thinner and have higher dislocation density [6-9]. Typically, monocrystalline Al undergoes a hardening-softening-hardening behavior, with dislocations evolving from tangles, to walls, and finally to cells **Error! Reference source not found.**[10]. The evolution of dislocation structure was also found to depend on strain amplitude, though there exists much conflict among reported phenomena [6, 9, 11]. A saturation stress has also been observed in many studies, where the cell size and misorientation stay constant as the cell morphology changes, in a process of equilibrium between work hardening and dynamic recovery [12]. It has been proposed that the saturation stress is dependent on grain size through a Hall-Petch relationship [9].

A persistent challenge in understanding fatigue damage accumulation and failure initiation has been the difficulty in resolving the internal GB structure as a function of fatigue loading. This has led to difficulties in correlating roughened grain surfaces (e.g. extrusions and intrusions) to cell structures in wavy slip materials [2, 3, 5, 11, 13]. In this work, we demonstrate an approach to resolve GB structures at various stages of low cycle fatigue damage by using a Ga-induced liquid metal embrittlement (LME)-based approach.

LME has been used in past studies to separate individual grains in polycrystalline Al and Al-based alloys. Applying liquid Ga to the surface of Al allows for penetration of Ga into Al GBs, which weakens or breaks the bonds across GBs, inducing brittle intergranular fracture under very low applied force. In an early study, liquid gallium (Ga) was applied to polycrystalline Al to study the grain shape and size distribution in correlation to processing parameters and grain growth kinetics [14]. Other studies have employed Ga-induced LME to facilitate brittle intergranular fracture to study the degree of GB sensitization by quantifying the size and spacing of β-phase (Mg_2Al_3) particles in 5xxx Al alloys and their effect on intergranular and stress corrosion [15]. These studies demonstrated that LME by Ga on Al-based samples is a robust method to preserve both composition and topography of GBs.

In this study, the LME approach was used to preserve deformation microstructures on GB at various points in fatigue life, thereby facilitating the direct observation and characterization of GB surface morphology as a function of cyclic loading conditions. TEM-based analysis was also used to correlate the morphology changes with changes in the underlying dislocation structures. This approach is expected to be applicable to understanding damage under a wide range of loading conditions such as high cycle fatigue or creep deformation.

Methods and Materials

Commercial 99.999% high purity Al plates of 1mm thickness were cut into dogbone samples with gage length 2 cm, and gage width 4 mm. The samples were mechanically polished with 1200 grit SiC paper, annealed at 400°C for 1 hour, and quenched in water. Previous research on GB ledge formation in relationship to annealing conditions indicated that heat treatment at this temperature and time yields the most regular grain surfaces, mostly free of surface features that may interfere with observation of fatigue response. As a proof of concept, stress controlled fatigue was used. Stress-controlled tension-tension fatigue tests were run on the annealed samples using an Instron ElectroPlus 3000 tensile tester at loads of 3.5—35MPa (14—140N) as well as 2.5—25 MPa (10—100N) and frequency of 1 Hz. The peak load was increased linearly with time to 140 N over the first 20 cycles. To expose the grain boundaries for SEM characterization, liquid Ga was applied on the fatigued specimens at 30-40°C and the specimens were allowed to sit in Ga for 4-5 hours on the hotplate. This time and temperature were found to be sufficient for intergranular fracture to occur with minimal applied force, suggesting complete penetration of the Ga into the GBs. Excessive Ga was removed with wipers and the Al grains were broken apart by tweezers. SEM imaging was done with Hitachi SU8010 Field Emission (FE) SEM with secondary electrons at 5kV and 10mA. TEM samples were cut from the specimen with a razor blade, mechanically thinned with 1200 grit SiC paper, electro jetpolished with a 90% ethanol, 10% perchloric acid solution, and analyzed in a FEI Tecnai F30 TEM at 300kV using bright field TEM and dark field scanning (S)TEM imaging conditions. A tensile test was performed at a displacement rate of 25 μm/s on the same high purity Al specimens as those used in fatigue tests, and displacement data was collected with an extensometer.

Results and Discussion

From the tensile test, a true stress-strain curve as shown in Fig. 2 was generated. As can be seen, the maximum stress applied during cyclic loading, 35 MPa, is well into the plastic regime, and thus significant plastic deformation was expected in every loading cycle.

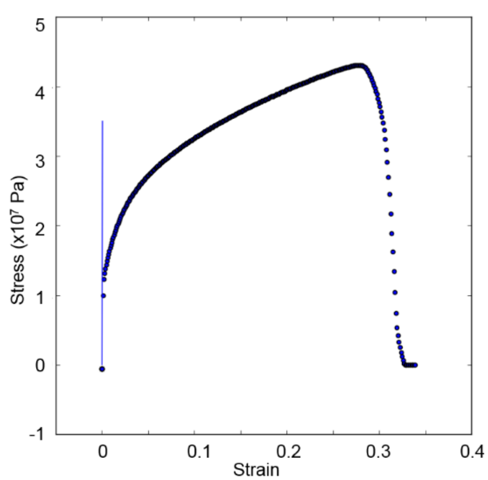
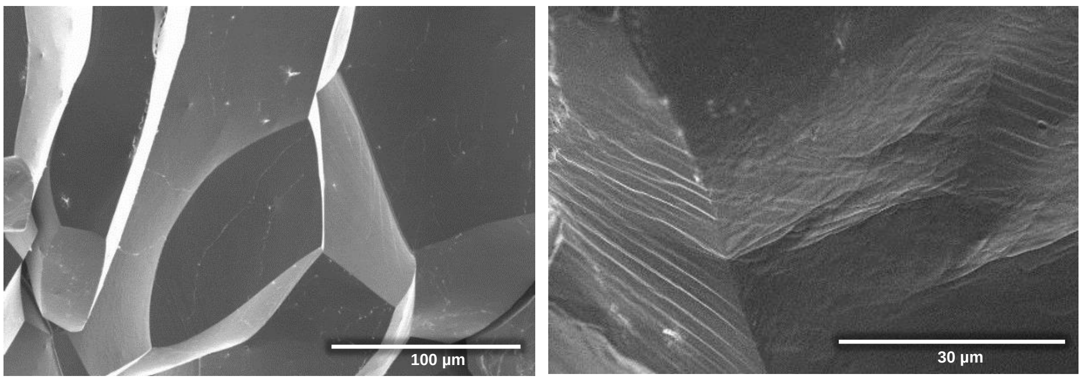


Figure 2. Stress-strain curve obtained from tensile test of the same specimens used for fatigue tests. The yield point is around 12 MPa.

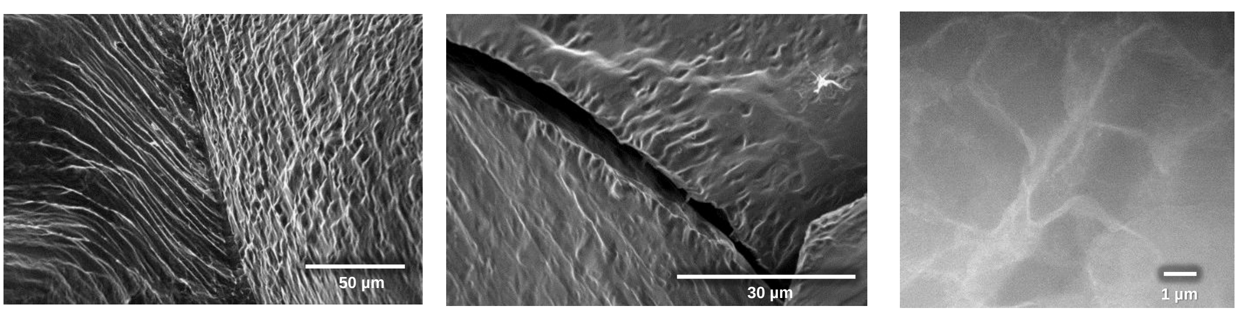
After annealing, GBs were almost completely free of surface features, with only a few patches of GB ledges apparent (Fig. 3). These ledges are presumably due to grain growth during the annealing process.

To establish the approximate fatigue life of the material under the given loading conditions, 3 samples were fatigued to failure. This was found to occur at 2157, 7792, and 6957 cycles. Post-failure analysis showed that the internal grain surfaces become extensively deformed, with ridge-like structures forming on both the GB surfaces and triple junctions (Fig. 4a-b). TEM analysis showed that the dislocation structures at failure included well defined cells (4c). The cells have very dense cell walls and relatively small sizes of approximately 2-3 μm in diameter.



(a) (b)

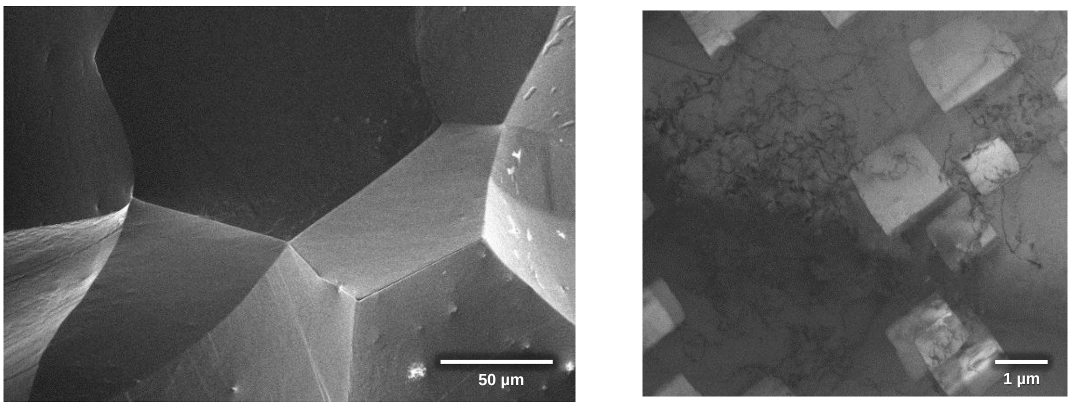
Figure 3. SEM images of the as-annealed GB morphology after liquid Ga embrittlement showing (a) smooth, featureless GBs representative of the majority of the observations and (b) limited areas of ledges formed on the GB faces.



(a) (b) (c)

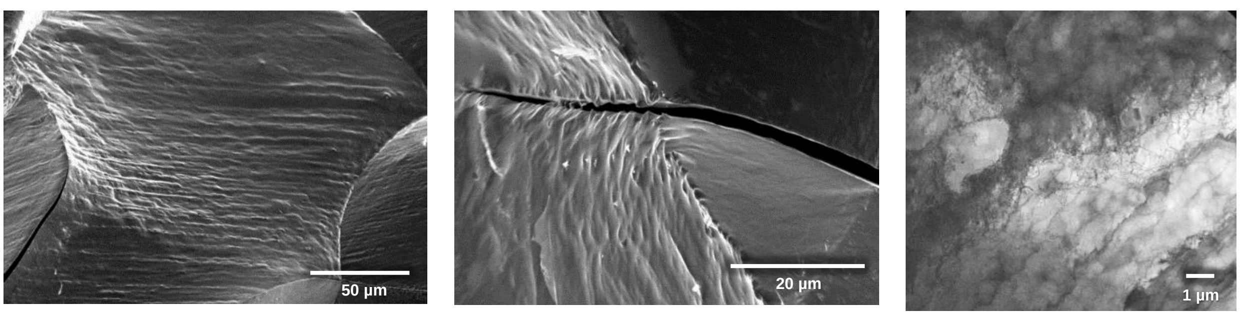
Figure 4. SEM images of (a) GB covered by ledges and (b) extrusions at triple junctions after failure. (c) Well defined dislocation cells after failure under low cycle fatigue, imaged by STEM.

The evolution of fatigue life was tracked with specimens characterized after 1, 100, and 1,000 cycles at the same stress amplitude. After 1 cycle, the grain structure mostly resembled that of the annealed samples, with relatively featureless GBs and triple junctions (Fig. 5a). Morphological changes from the as-annealed conditions were only very rarely observed at triple junctions, while almost all grain surfaces are perfectly smooth. TEM analysis of the samples showed mostly randomly scattered dislocations without well-defined structures (Fig. 5b). After 100 cycles, deformation-induced ledges appeared across many facets of GBs, and ridge structures formed preferentially at triple junctions, where deformation ledges intersect at the triple junctions (Fig. 6a-b). However, TEM analysis showed that the dislocation structures remained ill-defined, with the majority randomly distributed in loose tangles, and only a few cells beginning to form (Fig. 6c). This is at an early stage of cell formation, where entangled dislocation form partially connected cell boundaries, and have not yet formed closed cells. After 1,000 cycles, deformation on the grain surfaces become more extensive and irregular. Linearly packed ledges evolved into more random clusters of lumps (Fig. 7a), and ridges at triple junctions were still found to be prevalent (Fig. 7b). Cell structures were also found to form, though the dislocation density and degree of entanglement within the cell was lower than what was seen in the samples cycled to failure (Fig. 7c).



(a) (b)

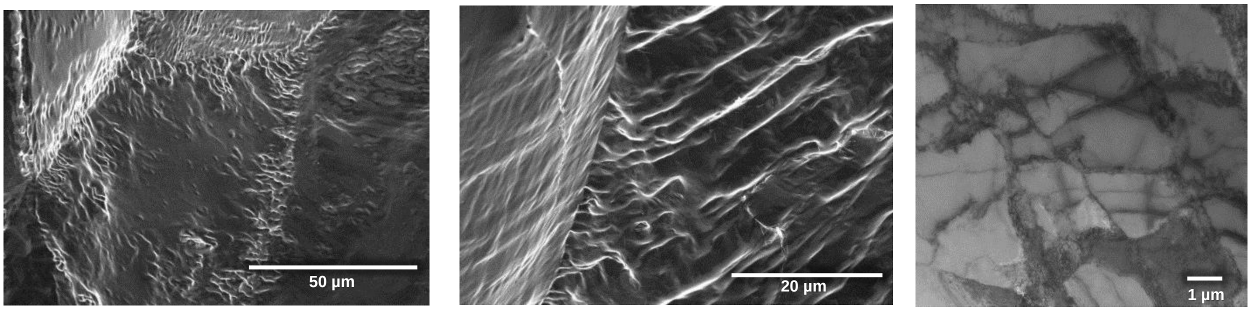
Figure 5. After 1 cycle, (a) SEM image of GB morphology, similar to as-annealed condition and (b) bright field TEM image of randomly entangled dislocations



(a) (b) (c)

Figure 6. After 100 cycles, (a) SEM image of GB structure showing extensive deformation ledge and lump formation, (b) SEM image showing extrusions forming preferentially at triple junctions, and (c) Bright-field TEM image of early stages of dislocation cell formation.

These results show that GB ledges and extrusions form before dislocation cells become saturated under low cycle fatigue, where the maximum stress exceeds yield point. The bowed out dislocations may easily impinge at GBs as well as on other cell walls, causing GB roughening and dislocation cell evolution to occur simultaneously.



(a) (b) (c)

Figure 7. After 1000 cycles, (a) SEM image of GB morphology showing extensive irregularly shaped lumps on GBs, (b) SEM image of triple point extrusions, and (c) bright field TEM image of well-defined dislocation cells formed.

Fatigue tests were run at a different amplitude in order to verify that the LME approach demonstrated here is capable of resolving differences in deformation structures due to different loading conditions. Due to time constraints, the fatigue life at the lower loading amplitude was not determined, but the dislocation structures and GB morphology were characterized at 5,000 and 691,080 cycles (Fig. 8). As can be seen, the results differ significantly from what was observed under higher stress amplitudes, suggesting that the LME-based approach is sensitive to variations in deformation-induced structures.

At 5000 cycles, dislocation cell structures were first observed (Fig. 8a). Most dislocation cells formed in this early stage were observed to have elongated shapes, and contain dislocations bowing out and stretching across the grain interior. GB morphology at this stage still mostly resembled that of the as-annealed condition, while some roughening was observed at triple junctions (Fig. 8b). Later, after 691080 cycles, dislocation cells appeared more equiaxed with higher dislocation density in the cell walls (Fig. 8c). A significant number of dislocation bow-outs were still observed in the cell interior, suggesting that cell refinement via dislocation impingement was in progress. Correspondingly, more well-defined extrusions were observed to form at triple junctions at this stage; however, GB ledges were not observed under this lower stress amplitude (Fig. 8d). This difference in GB morphology evolution from two different stress amplitudes suggests that Ga-induced LME can effectively prevent undesirable deformation to GB morphology during sample preparation and thus allows for imaging of intergranular topographical features at different points in fatigue life under different loading conditions.

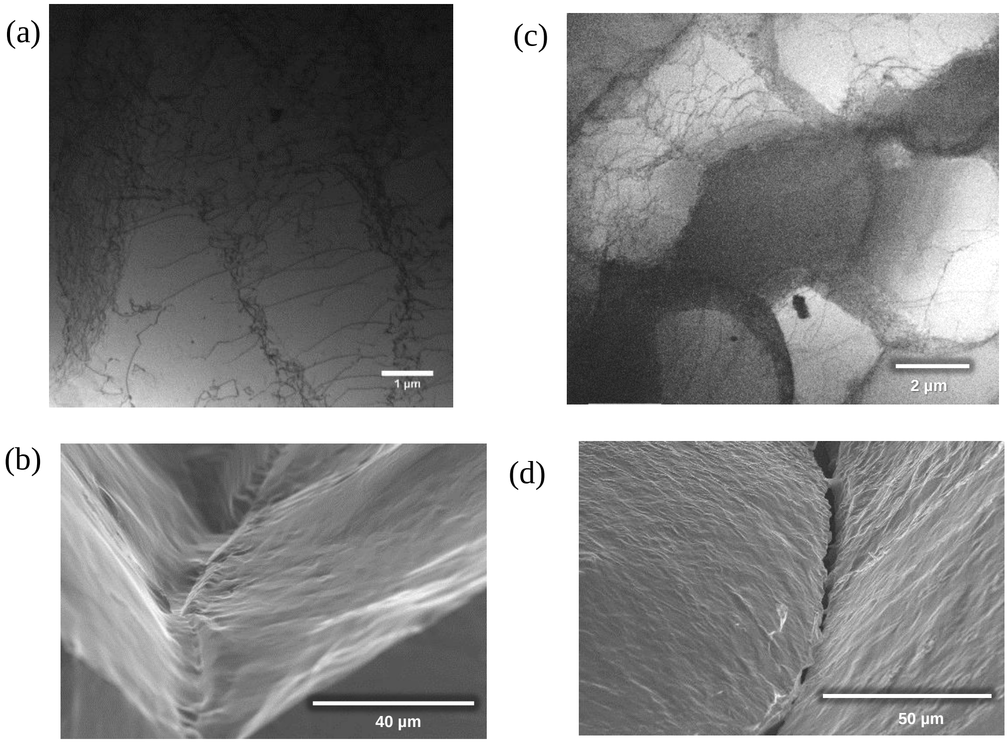


Figure 8. Dislocation structure and GB morphology under higher cycle fatigue with 2.5—25 MPa loading. (a) TEM bright field image of an initial stage of dislocation cell formation after 5000 cycles. In the cell interior, dislocations bowed out from cell walls, and in the cell boundaries, dislocation density was low, (b) SEM image of initial roughening at triple junctions after 5000 cycles, (c) TEM bright field image of more equiaxed dislocation cells formed via cell subdivision. Bowed out dislocation were still prevalent in cell interior after 691080 cycles, and (d) More well-defined extrusion formed at triple junctions after 691080 cycles.

Conclusion

Ga-induced LME is shown to be an effective method to preserve GB morphology for mechanistic studies of microstructure deformation under cyclic loading conditions. Under stress controlled low cycle fatigue of high purity Al, it was seen that GB deformation occurs very early in fatigue life, with ledges forming on GB faces and at triple junctions in the first 5% of fatigue life. Dislocation cell formation also occurs rapidly during this stage, and later cell structures evolves gradually by increasing dislocation density in cell boundaries. Deformation-induced GB structures became more pronounced at later stages of fatigue with the density of surface features increasing to cover nearly all GBs and triple junctions. This evolution process is dependent on stress amplitude, and occurs much more slowly for high cycle fatigue. For all stress amplitudes tested, dislocation structure and GB morphology evolution tend to proceed in a synchronized pattern. The earliest stages of cell formation were observed at the same stage of fatigue life as ledge formation at grain boundaries and triple junctions. Future plans include a systematic study of GB morphology evolution under strain-controlled high cycle fatigue tests.

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