ELECTRON MICROSCOPIC ANALYSIS OF FATIGUE CRACK TIPS IN AN AL-CU ALLOY

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Abstract

To elucidate the micro-mechanism of fatigue crack growth (FCG), this study conducts a direct analysis of the crack tip using several electron microscopic techniques. Particular attention is paid to FCG within a precipitation-hardened alloy, specifically aluminum (Al) - 2.4% copper (Cu). A small specimen containing the target crack tip is cut from a fatigued bulk specimen, and the two-step ion beam polishing method is applied to analyze its cross-section. It was found that FCG is strongly related to the formation of a dislocation cell structure, the size of which depends on the applied stress intensity factor range, ΔK.

Key words: Fatigue crack growth, Crack tip, Electron microscopy, Aluminum alloys, Precipitates

1 Introduction

The fatigue of metals has been a leading cause of machinery failures since the industrial revolution drastically increased the demand for metals as the main material. In modern metallurgy, discussion of the fatigue property of metals centers around the cyclic-stress (strain) response, that is, the hardening or softening behavior of the matrix prior to failure is of central interest[1)]. Conversely, from a mechanical engineering perspective, the estimation of failure cycles (N_f), directly related to the behavior of a fatigue crack, takes precedence[1)-2)]. When a fatigue crack is nucleated after a certain number of incubation cycles, it progressively extends into the material cycle-by-cycle until N_f is finally reached. The fatigue crack growth (FCG) is therefore expressed as an advance per cycle, $\mathrm{d}a/\mathrm{d}N$ (unit: m/cycle), where a and N denote crack length and number of cycles, respectively. Understanding the microscopic processes at the crack tip, namely, how $\mathrm{d}a/\mathrm{d}N$ is determined and influenced by various factors, is crucial. This will not only reveal the cause of fracture but will also provide valuable

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insights into the development of alloys with enhanced FCG resistance. In this study, the authors directly investigate the micro-/nanoscale phenomena around fatigue crack tips in an aluminum (Al) alloy using several electron microscopy techniques. The methodology effectively reveals some aspects of FCG not previously addressed in other studies.

2 Materials and Methods

The material used in this study was an Al alloy containing copper (Cu) as the main alloying element (see Table 1 for its chemical composition). Figure 1 presents information of the material and specimen. The ingot of the Al-Cu alloy was first homogenized (470°C × 6 h) and die-extruded to form a rod shape (ϕ23 mm). The rod was then solutionized (520°C × 1 h) and water quenched. Subsequently, the rod was naturally aged at room temperature (RT) for more than 500 days. The pole figure (Fig. 1(b)) shows that, as a result of the extrusion, the alloy has a clear texture oriented in the <100>/<111> directions. The average grain size measured in this normal plane was 12.5 μm. The magnified image (Fig. 1(c)) reveals needle-shaped precipitates whose typical length and diameter are 100 to 300 nm and 5 to 15 nm, respectively. These precipitates, so-called "Guinier-Preston (GP) zones", are known to be preferentially formed in the <100> directions. A tapered dumbbell specimen shown in Fig. 1(d) was machined from the rod. A small blind hole (see inset "A") was drilled at the gauge center position, which was used as a crack starter. Fatigue tests were performed with a cantilever-type rotary bending machine (GIGAQUAD™ YRB200, Yamamoto Metal Ltd.) at a

Table 1 Chemical composition

Element	Si	Fe	Cu	Mg	Mn	Zn	Ti	Al
Wt.%	0.10	0.20	2.41	0.04	0.65	<0.01	0.03	Bal.

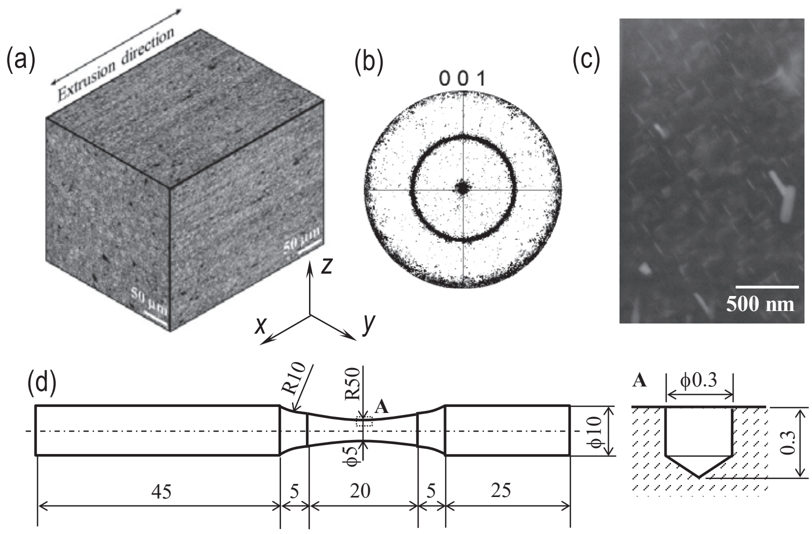

Fig. 1 Material and specimen information: (a) three-dimensional montage of alloy micro-structure; (b) {100} pole figure evaluated on the plane normal to extruding direction (i.e. *yz* plane); (c) micrograph of fine precipitates (GP zone); (d) dimension of fatigue specimen (unit: mm).

frequency of 50 Hz. The stress amplitude, σ_a, at the gauge center was controlled by the bending load applied to the lever end. The specimen temperature was kept at 150°C in ambient air. FCG behavior was intermittently recorded by a digital optical microscope (VHX-5000, Keyence Ltd.). With the recorded crack image, da/dN was calculated.

Figure 2 shows the crack tip sample preparation procedure employed in this study. Following an interruption of the fatigue test, the crack tip region of interest had to be carefully taken out from the deepest interior of the specimen. This is due to the fact that the slip behavior around a surface crack tip does not necessarily represent that of the material interior. The columnar gauge part was therefore cut by a diamond saw to reveal the cross-section of the crack (Fig. 2(a)). The saw-cut surface was then polished by an argon (Ar) ion beam to eliminate saw damage and reveal the original crack-induced deformation (Fig. 2(b))[3)-5)]. At this point, a relatively wide area around the target crack tip was analyzed by the electron backscatter diffraction (EBSD) technique. Then, a relatively small area around the target crack tip was subjected to a gallium (Ga) ion beam process for preparation of a thin foil sample (Fig. 2(c))[6)]. The foil sample was observed by a high-voltage transmission

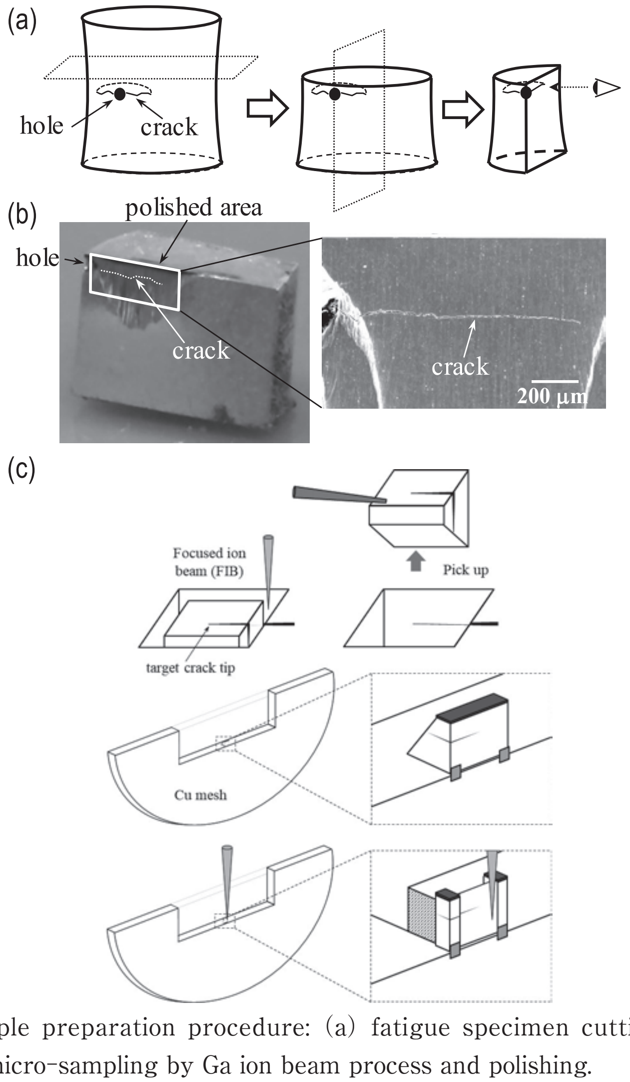

Fig. 2 Crack tip sample preparation procedure: (a) fatigue specimen cutting; (b) Ar ion beam polishing; (c) micro-sampling by Ga ion beam process and polishing.

electron microscope (HVTEM: JEM-1000K RS, Jeol Ltd.) and a scanning-TEM (STEM: JEM-2100F HK, Jeol Ltd.) equipped with an energy-dispersive X-ray spectroscopy (EDS) system, both of which belong to the High Voltage Electron Microscope Laboratory in Nagoya University, Japan. It should be noted that the combination of the two different ion beam processing methods enables the characterization of the *same* crack tip at different scales.

3 Results and discussion

Figure 3 shows an example of the FCG observation conducted by optical microscopy. Fatigue cracks are observed to initiate from the brim of the hole after 10,000 cycles. These cracks, which are separated in the initial stage, coalesce later and grow into the specimen interior in a semi-circular shape, as shown in the schematic illustration. The crack generally maintains mode I growth, i.e., crack plane is normal to the loading direction. This growth morphology was basically the same, irrespective of the applied σ_a level. To facilitate a quantitatively meaningful discussion, the value of the stress intensity factor range, ΔK, at the deepest point "C" is calculated with the following formula[7]:

$$\Delta K = \frac{2.2}{\pi} \sigma_a \sqrt{\pi a / 2} \tag{1}$$

where a is the crack length measured on the specimen surface. Crack tips with different ΔK values are compared hereafter.

Figures 4 and 5 show micrographs of the *same* crack tip (ΔK=1.86 MPa·m$^{1/2}$, $da/dN \approx 10^{-8}$ m/cycle) observed by different microscopy techniques. The data shown in Fig. 4 was obtained

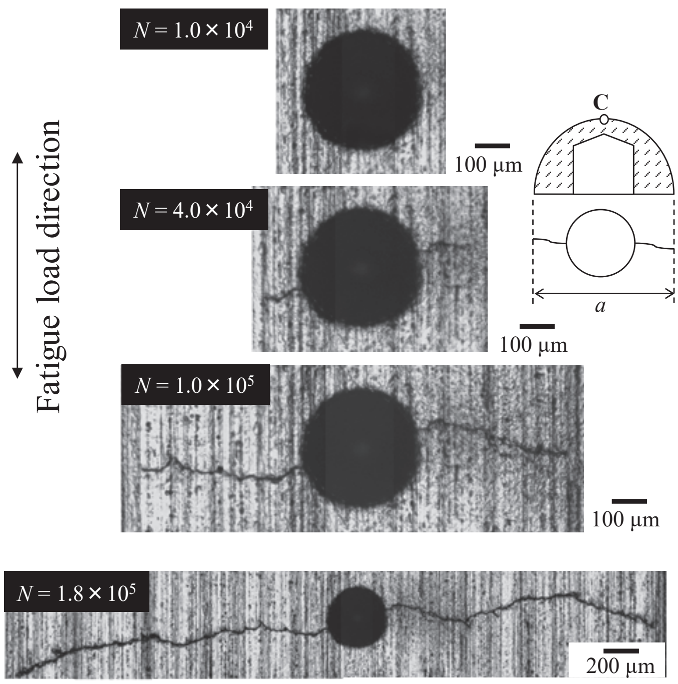

Fig. 3 Example of fatigue crack growth (FCG) behavior observed on the specimen surface by optical microscopy. Test condition: 150 ° C, air, σ_a = 120 MPa. The schematic illustration shows the semi-circular crack morphology in the specimen interior.

before the micro-sampling process shown in Fig. 2(c) was carried out. The crack appears to basically traverse the original elongated grain structure. The crack tip region accompanies a cellular structure whose size is clearly smaller than that of the original grains. These cells (termed "subgrains" in other studies[8]) are presumed to have been formed by the locally intense cyclic stress (strain) field. Further observation by TEM (Fig. 5) reveals interesting

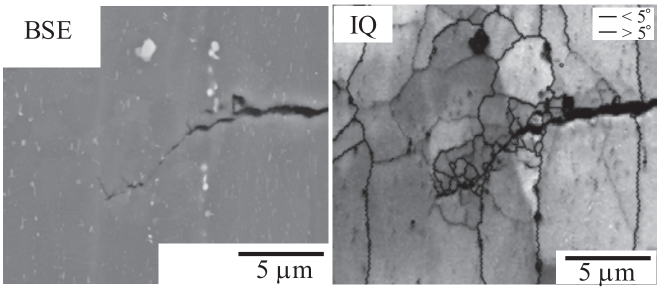

Fig. 4 Micrographs of a crack tip with a relatively low ΔK value (ΔK=1.86 MPa·m$^{1/2}$, da/d$N \approx 10^{-8}$ m/cycle). Backscatter electron image (left) and image quality (IQ) map obtained by the EBSD technique. Note that the boundary lines shown in the IQ map are categorized into two classes depending on the magnitude of misorientation angle.

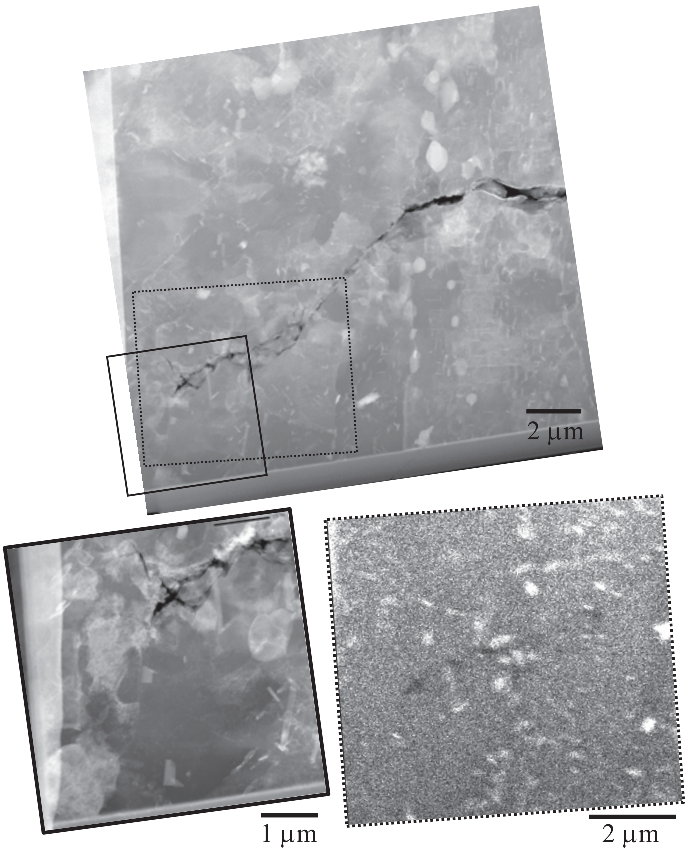

Fig. 5 HVTEM micrographs of a crack tip corresponding to Fig. 4. Dark-field image of overall area (upper) and enlarged image (lower left). The STEM-EDS elemental map shows the distribution of Cu (lower right, white-colored particles in the map).

points. Cells (approximately 1–2 μm in size) are formed along the crack, or, more precisely, the crack appears to propagate ***along*** the boundaries of these cells. The original distribution of the GP zones is altered: many of them are reformed into sparsely distributed coarse precipitates, or are seemingly dissolved and disappear from the matrix. This is probably due to the temperature-time (aging) effect together with severe dislocation cutting imposed around the crack tip.

Figures 6 and 7 present another set of micrographs obtained from a crack tip subjected to a much more severe mechanical condition (ΔK=5.72 MPa·m$^{1/2,}$ da/d$N \approx 10^{-7}$ m/cycle). The crack tip shows branching but tends to grow horizontally. The cells are more distinctly formed and closely packed around the tip than the lower ΔK case. TEM clearly confirms that the crack tip is located at a cell boundary and the cell size is much smaller. The distribution of Cu also appears to be related to the formation of cells, with a tendency to agglomerate at the cell boundaries. Alternatively, it may be more appropriate to state that cells are initially bounded by the pre-formed Cu precipitates. This is because the diffusion rate of Cu in Al matrix, estimated to be 6.4×10^{-11} m/s at 150°C, is expected to be significantly smaller than the cell forming rate (estimated to be closer to the product $f \times$ da/dN=5×10^{-6} m/s, where f is the load frequency, 50 Hz).

The above observations suggest that the FCG in the present Al-Cu alloy is closely related to cell formation, regardless of the applied ΔK level. It should be noted, however, that the observed cell size is actually larger than the apparent da/dN. This discrepancy can be understood in one of the following ways. One hypothesis is that the effective dimension of the crack tip unit event (i.e., opening by plastic blunting) is simply smaller than the cell size. The other hypothesis is that the crack growth does not strictly occur every cycle (i.e., it is essentially intermittent at the microscale). Unfortunately, the validity of these hypotheses cannot be confirmed by the present ex situ method. Regardless, there is no doubt that the addition of Cu, or presumably any alloying elements, strongly influences the FCG rate of Al alloys through the formation of a local cell structure at the crack tip.

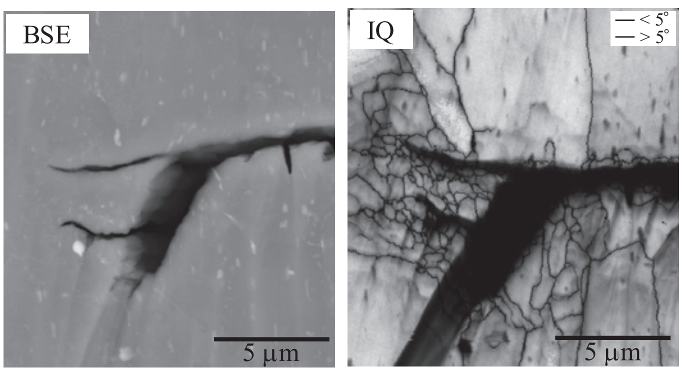

Fig. 6 Micrographs of a crack tip with a relatively high ΔK value (ΔK=5.72 MPa·m$^{1/2}$, da/d$N \approx 10^{-7}$ m/cycle). Backscatter electron image (left) and image quality (IQ) map obtained by the EBSD technique. Note that the boundary lines shown in the IQ map are categorized into two classes depending on the magnitude of misorientation angle.

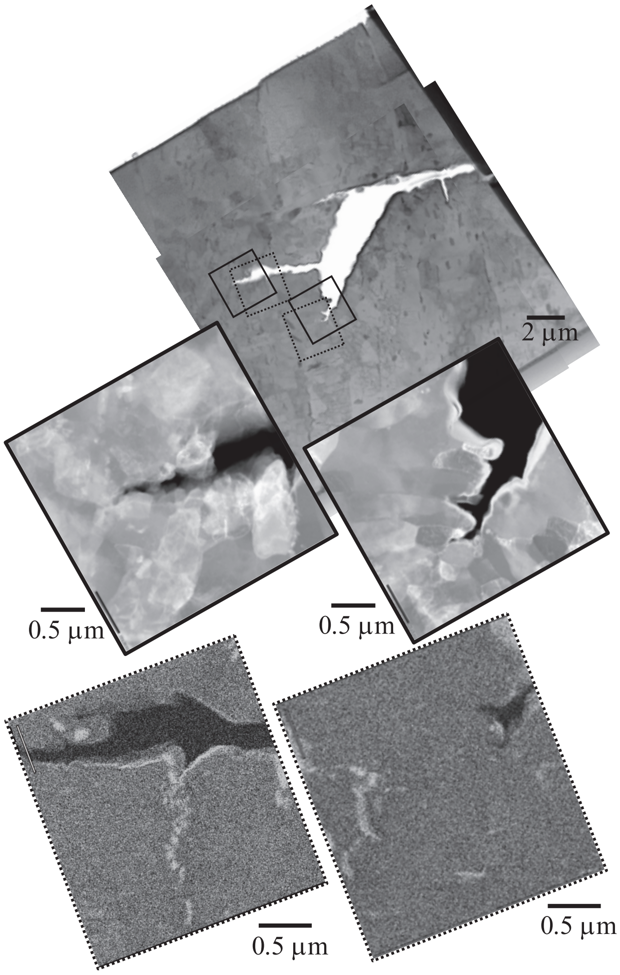

Fig. 7 HVTEM micrographs of a crack tip corresponding to Fig. 6. Bright-field image of overall area (upper) and enlarged dark-field images (middle). The STEM-EDS elemental maps show the distribution of Cu (lower).

4 Summary and conclusions

In this study, the microscopic mechanism of fatigue crack growth (FCG) in a precipitation-hardened Al-Cu alloy was investigated through the direct observation of crack tips using several electron microscopic techniques. The findings revealed that FCG is closely related to the formation of a local cell structure at the crack tip that was accompanied by the reformation of the original Cu precipitates (GP zones) into coarser ones. The applied ΔK level strongly affected the local cell size and the distribution of precipitates. Interestingly, the cell size was not directly correlated with the apparent FCG rate, da/dN (m/cycle). Ultimately, the analytical methodology introduced in this study can be broadly applied to various materials and crack growth phenomena, contributing significantly to the understanding and enhancement of the life properties of materials subjected to harsh mechanical conditions.

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