

Growth behavior of small surface cracks in coarse and ultrafine grained copper

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Abstract

Since fatigue life of a plain specimen of ductile metals is controlled mainly by the propagation life of a small surface crack, to clarify the growth behavior of a small crack is crucial to the safe design of smooth members. However, little has been reported on the growth behavior of small surface cracks in ultrafine grained (UFG) metals. In the present study, stress-controlled fatigue tests for coarse grained (CG) and UFG copper were conducted. The surface damage evolution during cyclic stressing was observed by optical microscopy, and the growth behavior of a small surface crack was monitored by a plastic replication technique. The physical background of fatigue damage for CG and UFG copper was discussed from the viewpoints of the initiation and growth behavior of small surface cracks.

Keywords: equal channel angular pressing, copper, ultrafine grain, fatigue, small surface-crack.

1 Introduction

Copper has been widely used as a base material in various electronics-based industries. Recent developments in electronic apparatus however mean that



higher strength/ductile materials are now required to meet these needs. To improve the strength of copper requires the development of highly alloyed Cu materials. Alloyed Cu however, possesses inherently lower conductivity compared to their unalloyed counterparts. Therefore, in order to overcome this inherent shortcoming associated with conventional Cu alloys, the development of pure Cu with submicron grained structure is currently being explored by employing a severe plastic deformation principle, such as the ECAP (equal channel angular pressing) processing [1–4].

For applying the ultrafine grained (UFG) copper to the components of machines and structures, fatigue damage should be clarified. Since the fatigue life of machine components and structures are mainly controlled by the growth life of a fatigue crack, the crack growth behavior should be clarified for the design of safe machine components and structures. Recently, the growth behaviors of millimeter-range cracks in UFG metals were studied for compact-tension [5–7] and single edge-notched specimens [8,9]. On the other hand, the fatigue life of smooth specimens is approximately controlled by the growth life of a small surface crack. Nisitani and Goto [10], Goto and Nisitani [11] and Goto and Knowles [12] showed that the crack growth life from an initial size to 1 mm accounted for about 70% of the fatigue life of plain specimens of many conventional grain-sized metals. This means that the growth behavior of small cracks must be clarified to estimate the fatigue life of plain members. For both coarse grained (CG) and UFG copper, to the authors' knowledge, there has been little reported in the literature on the growth behavior of a small surface crack [13].

In the present study, stress-controlled fatigue tests for CG and UFG copper were conducted. The surface damage evolution during cyclic stressing was observed by optical microscopy, and the growth behavior of a small surface crack was monitored using a plastic replication technique (PRT). The physical background of fatigue damage was discussed from the viewpoints of the initiation and growth behavior of small surface cracks.

2 Experimental procedures

The material used was 99.99% pure oxygen-free copper. Before the ECAP process, the material was annealed at 500°C for 1hr. In ECAP die, the inner and outer angles of channel intersection were 90 and 45°, respectively. Repetitive ECAP was accomplished by Bc route (after each pressing, the billet bar was rotated around its longitudinal axis through 90°). Four passages of extrusion resulted in an equivalent shear strain of about 3.9 [14]. MoS₂ was used as lubricant at each pressing, and the pressing speed was 5 mm/sec. After ECAP, the microstructure and mechanical properties were studied. Transverse cross sections of ECAP processed bars were cut to prepare specimens for transmission electron microscopic observation. Specimens were mechanically polished to a thickness of 100 µm, then subjected to twin-jet electropolishing. The solution was 200ml CH₃OH plus 100 ml HNO₃. The jet thinning was conducted at –30°C.



Round bar specimens of 5 mm diameter (Fig. 1) were machined from the annealed and ECAP processed bars. Although the specimens had a circumferential notch ($\rho = 20$ mm and $c = 0.25$ mm, where ρ = radius and c = depth of the notch), the strength reduction factor for this geometry was close to one, and therefore the specimens could be considered plain specimens. Before testing, all the specimens were electropolished to remove about 25 μm from the surface layer in order to facilitate observations of the surface state.

All tests were carried out at room temperature using a rotating bending fatigue machine operating at 3000 rev/min. The surface damage evolution during cyclic stressing was observed by optical microscopy. The measurement of crack length was conducted using a PRT. The crack length, l , is a length measured along the circumferential direction of the surface. The stress value referred to in this paper is that of the nominal stress amplitude, σ_a , at the minimum cross-section.

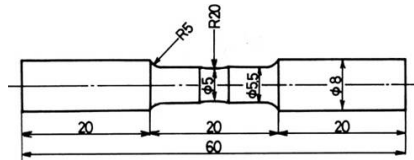


Figure 1: Shape of the specimen.

3 Experimental results and discussion

3.1 Microstructure and mechanical properties of the materials

Figure 2 shows typical microstructures for CG and UFG copper. Grain size of CG copper was about 100 μm . For UFG copper, refinement of structure is evident. Namely, granular grains with average size of 300 nm are formed, and grain boundary (GB) areas involve a high population of dislocations. The SADP (selected area diffraction patterns of the center area, 1 μm in diameter) consists of rings of diffraction spots, showing that GBs have high angles of misorientation. The fraction of high angle GBs might be relatively low [15]. In addition, the GBs had lost their sharpness and exhibited a 'spotty' contrast and broad contours, suggesting non-equilibrium GBs contain random networks of GB dislocations [16].

The change in heat flow value was measured using a differential scanning calorimeter in a nitrogen environment with a ramping rate of 1.67°C/min up to 500°C. The measurements of the heat flow value showed that the changes in the heat flow value (*CHFV*) after the 4th pressing of ECAP were *CHFV* = 0.85 J/g. The rationale for the heat-flow measurement is that the heat-flow of pure copper would be solely controlled by the generation and/or annihilation of dislocations and GBs during ECAP processing, unlike the other alloys with either precipitation hardening or solid solution hardening mechanism. As might be expected, the *CHFV* of fully annealed copper is almost zero. The large value of

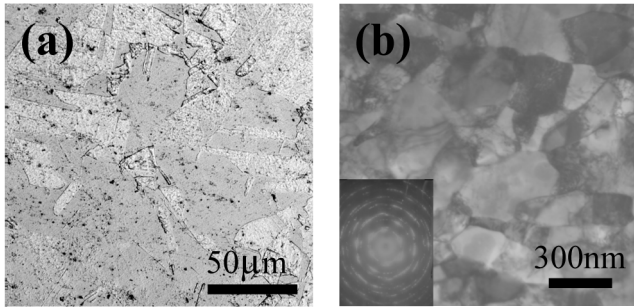


Figure 2: Microstructure of the materials; (a) CG copper, (b) UFG copper.

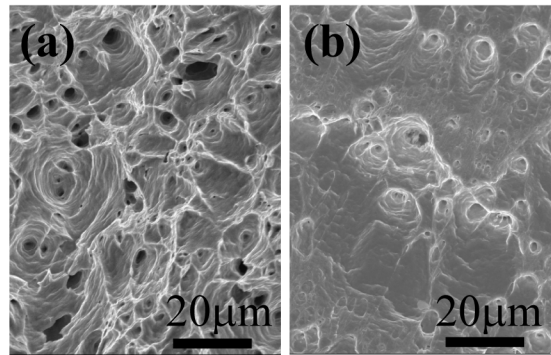


Figure 3: Fracture surface of tensile specimens; (a) CG copper, (b) UFG copper.

CHFV after the 4th pressing of ECAP means that redundant strain energy related to high-density dislocations is stored in the material, showing instability of the microstructure.

The mechanical properties of an annealed bar were 183 MPa tensile strength, 64% elongation, and a Vickers hardness number of 63. After eight passages of ECAP, their values changed to 438 MPa, 28%, and 141, respectively. The relationship between the Vickers hardness number, H_V , and the number of pressings showed that a dramatic increase in H_V ($H_V = 63$ to 128) occurs from the first pressing. However, the subsequent increases become milder, followed by a saturation trend after the 3rd pressing. Figures 3(a) and (b) show the post-fracture surface for tensile specimens of CG and UFG copper, respectively. The fracture surface of CG copper shows many dimples. The sample processed by ECAP shows a flat fracture surface, including a couple of dimples, compared to the fracture surface of annealed sample. There is no significant difference in the dimple size between two samples, in spite of the two orders of magnitude difference in grain size. Nevertheless, the dimples are shallower for the UFG copper because of its decreased ductility.

3.2 Formation behavior of fatigue damage

Figure 4 shows the S-N curve of CG and UFG copper. For UFG copper examined under stress controlled testing, the enhancement in fatigue life is obvious [17–19]. The degree of enhancement is sharply increased with increasing stress amplitude. In the long-life field in excess of $N = 10^7$ cycles, however, the fatigue life to failure of UFG copper tends to coincide with that of CG copper.

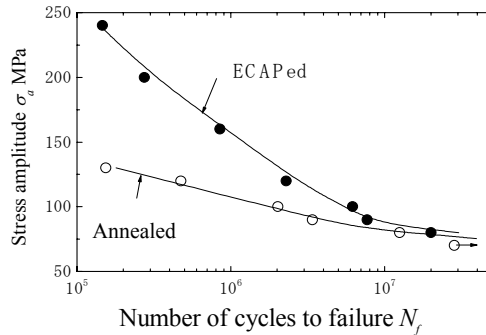


Figure 4: S-N curve.

Figures 5(a) and (b) show the typical surface damage of a post-fatigued specimen for CG and UFG copper, respectively. For CG copper, slip bands were formed along limited slip planes within a small number of grains and the length of slip bands was nearly equivalent to the grain size. In contrast, the surface of UFG copper was covered by a high population of damaged area. Figure 5(c) is SEM micrograph of the highlighted area in Fig. 5(b), showing protrusions and intrusions [20]. Surprisingly, those sizes are larger than the grain size.

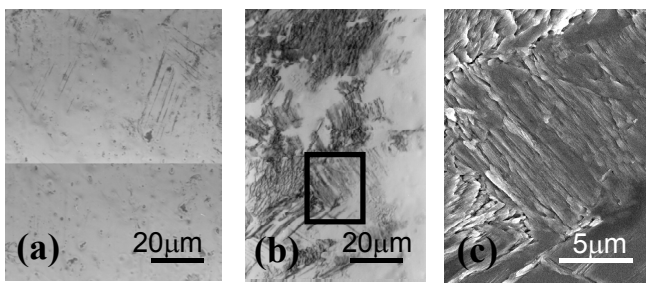


Figure 5: Surface morphology of post-fatigued specimen at $\sigma_a = 120$ MPa; (a) CG copper ($N_f = 4.739 \times 10^5$), (b) UFG copper ($N_f = 3.75 \times 10^6$), (c) SEM micrograph of the highlighted area in (b).

Figure 6 shows the formation process of surface damage at $\sigma_a = 120$ MPa for UFG copper, where the number of cycles to failure, N_f , was $N_f = 3.75 \times 10^6$. It is

found that there is a pronounced time lag in the onset of a significant enlargement of the damaged areas. In addition to this, the whole surface observations of the specimen indicated that the damaged regions were formed at an early stage of cycling, and that the number and area of these regions slowly increased with further cycling up to a specific number of cycles, depending on the material and stress amplitude. Once this specific number of cycles had been exceeded, both the number and area of the damaged regions showed a significant rise. The number of cycles performed prior to the start of this remarkably large extension in damaged regions was about half of fatigue life ($N \approx 1.7 \times 10^6$).

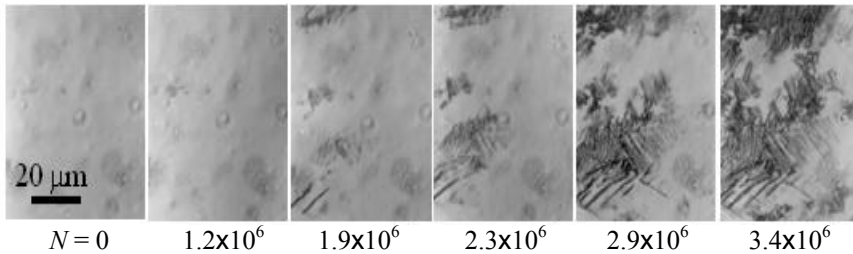


Figure 6: Change in surface state of UFG copper during repeated stressing of $\sigma_a=120\text{MPa}$ ($N_f=3.75 \times 10^6$).

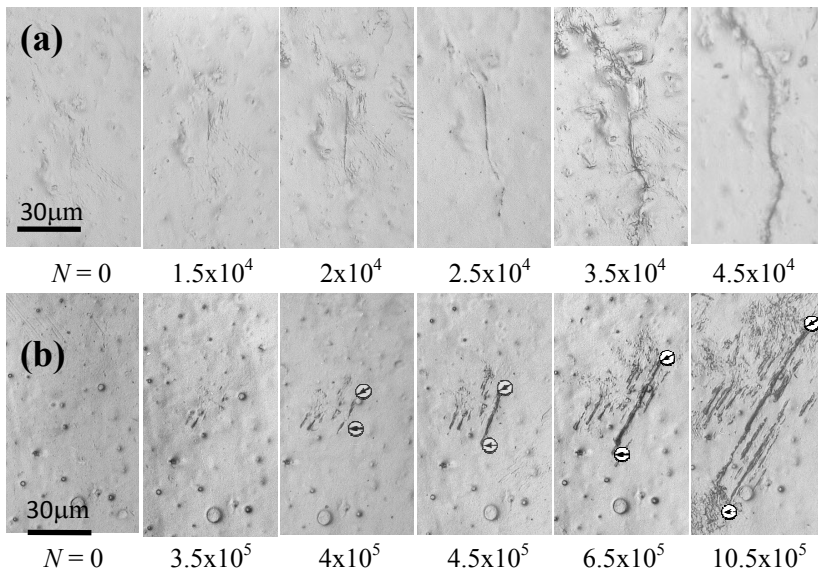


Figure 7: Change in surface state around a major crack observed by a plastic replication technique; (a) CG, $\sigma_a = 130\text{MPa}$, $N_f = 1.535 \times 10^5$, (b) UFG, $\sigma_a = 120\text{MPa}$, $N_f = 2.3 \times 10^6$.

Figure 7 shows initial growth behavior from a major crack, which led to the final fracture of the specimen, monitored by PRT. At an early stage of cycling,

for CG copper, slip bands were formed within a grain. A grain size crack was initiated from slip bands at an about 20% of fatigue life. For UFG copper, a 30 μm length crack was initiated at about 20% of fatigue life.

3.3 Growth behaviour of a small crack

Figure 8 shows the crack growth curve ($\ln l$ vs. N relation). The relationship for UFG copper can be approximated by a straight line independent of a stress amplitude. For CG copper, however, the relationship depends on a stress amplitude. Namely, the growth curves at a stress above $\sigma_a = 100$ MPa are nearly represented by a straight line. At a stress below $\sigma_a = 90$ MPa, a linear relation of the growth curve nearly holds for a crack length in excess of $l = 0.3$ mm, whereas, each plot for $l < 0.3$ mm deviates upward from an extension of the linear relation for $l > 0.3$ mm.

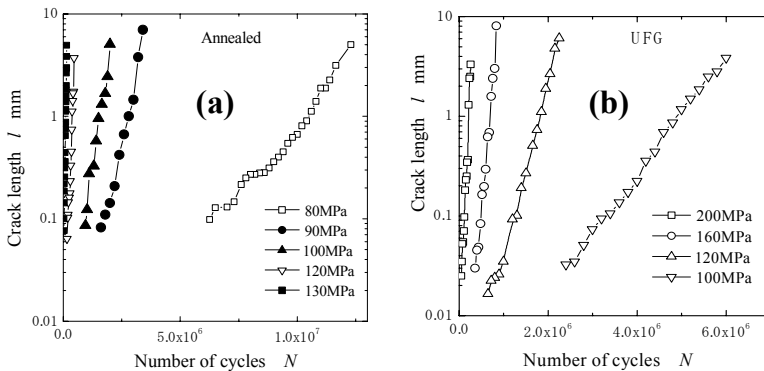


Figure 8: Crack growth curve ($\ln l$ vs. N relation); (a) CG copper, (b) UFG copper.

The linear relation shown in Fig. 8 means that crack growth rate (CGR, dl/dN) should be proportional to l at a constant stress amplitude. Figure 9 shows the CGR versus crack length relation. The CGR for CG copper is nearly proportional to the crack length ($dl/dN \propto l$) with the exception of the CGR below $dl/dN = 10^{-6}$ mm/c. For UFG copper, however, the $dl/dN \propto l$ relation nearly holds even for extremely low CGR range ($dl/dN < 10^{-6}$ mm/c) in spite of some fluctuating plots around $dl/dN = 10^{-8}$ mm/c. To compare the growth resistance of a small crack between CG and UFG copper, the relations at $\sigma_a = 80$ and 120 MPa for CG copper (Fig. 9(a)) were redrawn by dashed lines in Fig. 9(b). The comparison of the relation at $\sigma_a = 120$ MPa for both copper indicates that CGR of CG copper is about three times larger than that of UFG copper. In addition, relation at 80 MPa for CG copper is nearly equivalent to that at 85 MPa for UFG copper. These are different from the growth behavior of other ECAPed Al and steel alloys, in which finer microstructures exhibit higher growth rates [5, 6, 8]. However,

experiments conducted by other alloys were for a long crack initiated from artificial notches. This means that applied stress amplitudes were sufficiently small and a crack propagated under the small scale yielding condition. In the present fatigue tests, the stress amplitudes were excessively large compared to stresses applied to other alloys. For CG copper, especially, the ratios of stress amplitude to tensile strength, σ_a/σ_{ts} , were 0.43 and 0.66 for $\sigma_a = 80$ and 120 MPa, respectively. Accordingly, the crack at $\sigma_a = 120$ MPa propagates under large scale yielding, showing accelerated CGR compared to UFG copper with higher tensile strength.

The dependency of CGR on stress amplitude was studied and it showed that dl/dN is nearly proportional to σ_a^n at a constant crack length. The value of n was about 7.5 and 4.4 for CG and UFC copper, respectively. Considering the stress dependency and the relation $(dl/dN \propto l)$ in Fig. 9 together, thus, the CGR of the present copper is uniquely determined by a term $\sigma_a^n l$, namely small-crack growth law (SCGL) defined by equation (1) holds [10, 21];

$$\frac{dl}{dN} = C_1 \sigma_a^n l \quad (1)$$

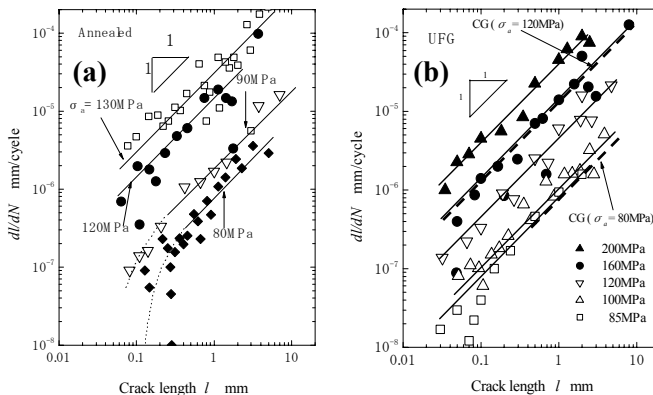


Figure 9: Crack growth data (dl/dN vs l relation); (a) CG copper, (b) UFG copper.

Figure 10 shows the dl/dN versus $\sigma_a^n l$ relation. It is evident that the SCGL is available for estimating CGR of a small surface crack propagating with a growth rate above 10^{-6} and 10^{-7} mm/c for CG and UFG copper, respectively. A dashed line in Fig. 10(b) is the relationship obtained from UFG copper processed by twelve pressing cycles of ECAP (grain size: 300 nm). There is no significant difference of the relationship between UFG copper with four and twelve ECAP process cycles.

Many researchers have reported that the growth rate of fatigue cracks can be unified in terms of the stress intensity factor range ΔK . Here, the parameter ΔK is

the effective parameter for a crack when the condition of small scale yielding at a crack tip is satisfied. The value of ΔK for an infinite plate with a crack is given by the equation $\Delta K = \Delta\sigma(\pi a)^{1/2}$. This equation indicates that the stress range has to be higher for a small crack in order to get the same growth rate as for a larger crack. Therefore, when a sufficiently small crack propagates at a finite growth rate (for example, 10^{-6} to 10^{-3} mm/c) the condition of small scale yielding is not usually satisfied. Thus the growth rate of a small crack cannot be determined uniquely by ΔK . Nisitani and Goto showed that a term $\sigma_a^n l$ is effective parameter to determine the CGR of a small crack in many ductile materials. Here, n is a material constant. The expression $\sigma_a^n l$ ($n = 3$) was first proposed by Frost and Dugdale [22]. They applied it to comparatively large cracks in which the condition of small scale yielding nearly holds. Now $\sigma_a^3 l$ can be considered as an approximation for ΔK , whereas $\sigma_a^n l$ in the present study is a parameter for crack propagation under large scale yielding. Moreover, an effective and convenient method based on a SCGL in which the effect of mechanical properties is partly considered has been proposed for predicting the fatigue life of smooth members of many CG metals [23, 24]. The prediction was in good agreement with the experimental results. Thus, Fig.10(b) means an applicability of SCGL to the evaluation of fatigue life of UFG metals.

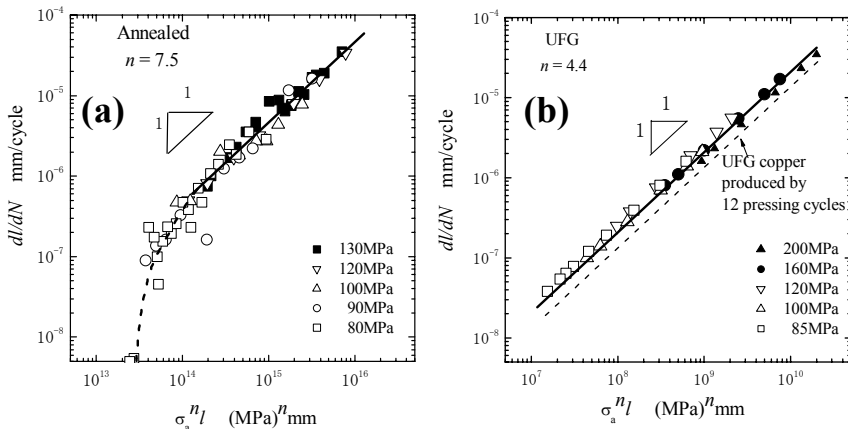


Figure 10: dl/dN vs. $\sigma_a^n l$ relation; (a) CG copper ($n = 7.5$), (b) UFG copper ($n = 4.4$, dashed line indicates the relation for UFG copper processed by 12 pressing cycles).

4 Conclusions

The main results of the present study can be summarized as follows:

- (1) The enhancement in fatigue life due to ECAP was remarkably large in the short-life regime, yet not so pronounced in the long-life regime, as reflected by the fatigue strength of UFG copper at $N = 10^7$ cycles, σ_w , which coincided with that of fully annealed CG copper.



- (2) For CG copper, a major crack, which led to the final fracture of the specimen, was initiated from slip bands with a dimension of grain size. For UFG copper, it was initiated from intrusions with a dimension of over ten times grain sizes. For both copper, the crack growth life from an initial size to fracture accounted for about 80% of the fatigue life of plain specimens.
- (3) At a high stress amplitude ($\sigma_a > 100$ MPa), growth rate of a small crack of CG copper was larger than that of UFG copper. At $\sigma_a = 80$ MPa, however, CGR of CG copper was nearly equivalent to that of UFG copper. In CG copper, the ratio of $\sigma_a = 100$ MPa to tensile strength was about 0.55. In a range of $\sigma_a > 100$ MPa, thus, the crack in CG copper propagates under large scale yielding, showing accelerated CGR compared to UFG copper with higher tensile strength.
- (4) The growth rate of a small crack cannot be unified in terms of the stress intensity factor range ΔK , but it was uniquely determined by a term $\sigma_a^n l$. Here, n is a material constant and the value of n was about 7.5 and 4.4 for CG and UFG copper, respectively. For CG copper, the term could estimate the growth rate of a crack propagating with $dl/dN > 10^{-6}$ mm/cycle. For UFG copper, the term was applicable to a crack with an extremely low growth rate ($dl/dN > 10^{-7}$ mm/cycle).

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