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Near threshold fatigue crack growth in ultrafine-grained copper

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Abstract. The near threshold fatigue crack growth in ultrafine-grained (UFG) copper at room temperature was studied in comparison to conventional coarse-grained (CG) copper. The fatigue crack growth rates \(da/dN\) in UFG copper were enhanced at \(\Delta K \leq 7\) MPa\(\sqrt{m}\) compared to the CG material. The crack closure shielding, as evaluated using the compliance variation technique, was shown to explain these differences. The effective stress intensity factor amplitude \(\Delta K_{eff}\) appears to be the same driving force in both materials. Tests performed in high vacuum on UFG copper demonstrate the existence of a huge effect of environment with growth rates higher of about two orders of magnitude in air compared to high vacuum. This environmental effect on the crack path and the related microstructure is discussed on the basis of fractography observations performed using scanning electron microscope and completed with field emission scanning electron microscope combined with the focused ion beam technique.

1. Introduction

Severe plastic deformation (SPD) techniques, such as equal channel angular pressing (ECAP), have been widely used to obtain ultrafine grain (UFG) metals with high tensile strength and fairly large plasticity at low temperatures. Even though a certain number of studies have been carried out for investigating static and fatigue properties of UFG metals, very few data on fatigue crack growth resistance are available [1, 2]. Particularly, the fatigue crack propagation mechanisms for these materials should be clarified in the near threshold regime since the crack growth plays a major role in the design of safe engineering components and structures.

Reduction in the crack growth resistance with decreasing grain size was reported in Cu, Ni, Ti and Al-alloys [3–7]. This detrimental effect is attributed to the greater availability of grain boundaries in orientation favorable for crack propagation in a material with an UFG microstructure. Much less attempt has been made to identify the intrinsic propagation mechanism by eliminating closure and environmental factors, the only exception being the work by Pao et al. [8] in the case of a UFG Al-Mg alloy.

The current study aims to investigate whether or not the intrinsic propagation mechanism is altered by UFG microstructure. For this purpose, propagation of long fatigue cracks was studied in the mid stress intensity factor amplitudes and in the near threshold range in UFG copper obtained by ECAP. Crack propagation tests were conducted in air and under vacuum at \(R=0.1\) on small CT specimens. The crack growth mechanisms are discussed according to the testing environment based on local fracture surface observations and focused ion beam (FIB) cuts.

2. Materials and methods

2.1. Materials and environments
Commercially pure polycrystalline copper used in this study was annealed at 450 °C for 30 min prior to ECAP processing. The billets were processed in a 90° ECAP die up to 5- and 6-passes upon route C to obtain an UFG microstructure. Compact tension (CT) specimens with a width (W) of 16 mm and a thickness (B) of 5 mm were prepared from the central part of the billets. On these CT specimens the loading direction and notch direction, respectively, were parallel to normal direction (ND) and extrusion direction (ED). Also, CT specimens with the same dimensions as mentioned above as well as standard CT specimen (W=40 mm) were prepared from CG copper (grain size $\bar{d} \approx 50 \, \mu m$). The specimens were pre-cracked under fatigue loading in air at room temperature, at a frequency of 20 Hz and R=0.1 in such a way that the stress intensity factor range $\Delta K_{\text{init}} \approx 8 \, \text{MPa}\sqrt{\text{m}}$ was reached at an initial crack length of $\approx 1 \, \text{mm}$. The used environments were ambient air and high vacuum ($\approx 10^{-6} \, \text{mbar}$).

The overall appearance of grains (size and structure) examined on a FIB cut was rather homogenous after 5-passes and 6-passes and was within the range of ultrafine grain (less than 1 µm).

2.2. Fatigue crack growth testing

Fatigue crack growth tests were conducted on a servo-hydraulic testing machine in load control mode using a sinusoidal waveform signal for loading ($f = 20 \, \text{Hz, R} = 0.1$). The crack length was measured on both sides of the specimens using an optical travelling microscope. The stress intensity factor range was calculated using the ASTM E647 recommendations [9]. Crack closure was measured systematically at each point by lowering the testing frequency to 0.2 Hz and recording the variation of the fatigue cycle load with respect to the displacement measured on the back face of the specimen using numerical data acquisition. Details of the closure determination technique were described in [10].

2.3. Fractography

Observation of fracture surfaces was conducted on a field emission gun scanning electron microscope (FEG-SEM) using secondary electrons with an accelerating voltage of 20 kV. During fractographic analysis, attention was paid to special features on the fracture surface along the crack propagation direction. Also, detailed observations were done beneath the fracture surface on specific positions by performing cuts using FIB technique.

3. Results and discussion

Fatigue crack growth curves in Fig. 1 show relationships between the Fatigue Crack Growth Rates (FCGR) and the stress intensity factor range in the UFG specimen together with the reference data for conventional coarse-grained (CG) copper. It can be seen that material subjected to 5 passes exhibits a considerable increase in the crack growth rates in the range of stress intensity factor below 7 MPa\(\sqrt{\text{m}}\); however, the curves merge together at higher $\Delta K$ values, namely above 7 MPa\(\sqrt{\text{m}}\) (Fig. 1a).

Also, in UFG specimens, a constant deviation of crack growth direction of 22°-24° with respect to the plane perpendicular to the loading direction was observed. It is worth mentioning that according to the ASTM standard [11] for such deviations the same value of stress intensity factor as calculated in mode I loading can be used with an error of about 1%. It is already well-established that the crack closure plays a major role in promoting marked differences between the effective (near-tip) $\Delta K_{\text{eff}}$ and the nominal (far-field) $\Delta K$ of long cracks. The contribution of crack closure has been shown to depend on various factors [12, 13] such as length, size and geometry of the plastic wake along the crack flanks. Crack closure measurements done during the propagation test were used to calculate the effective stress intensity factor ranges as shown in Fig. 1b. It comes out from this figure that the differences observed in the nominal curves between UFG and CG copper are rationalized in terms of the
effective stress intensity factor range $\Delta K_{\text{eff}}$ after crack closure correction. These results are in accordance with those obtained by Pao et al. [8] in the case of a UFG Al-Mg alloy.

Figure 1. Crack growth rate of CG-Cu (black) and UFG-Cu (red) in air at room temperature and $R=0.1$. a) $da/dN$ vs. $\Delta K$ curve, b) $da/dN$ vs. $\Delta K_{\text{eff}}$ curve.

3.1. Fractography
The fracture surface morphology of CG copper tested in air (Fig. 2) is substantially different from that on the UFG copper, particularly regarding the surface roughness. Asperities of several micrometers amplitude filled with matted edges observed in CG copper (Fig. 2a) could induce higher crack closure levels than in the case of the UFG copper which exhibits a very smooth fracture surface (Fig. 2b) under comparable loading conditions.
Figure 2. Fracture surfaces on CT specimens tested in air a) CG at $\Delta K \approx 5$ MPa√m, $da/dN = 1E-9$ m/cycle, and b) UFG-Cu at $\Delta K \approx 5.5$ MPa√m, $da/dN = 4E-9$ m/cycle (arrows show propagation direction).

In Fig. 3 the crack growth curve of 5C specimen tested in air is compared to the propagation of 5C and 6C specimens tested under vacuum. The two curves in vacuum are superposed. The experimental results clearly indicate a huge change in the crack propagation mechanism under vacuum as compared to the one in air, with growth rates in air more than two orders of magnitudes higher than under vacuum. Another important feature is the change in crack paths due to the change of environment as shown on the right-hand side of Fig. 3. Deviation of crack growth direction with respect to the plane perpendicular to the loading direction became greater when the crack grew under vacuum, as can be detected on the lateral surfaces of both specimens on Fig. 3. Meanwhile, whether the crack propagation plane remains constant throughout the thickness of specimen or changes within the distance from the free surface of the specimen is not yet known.
Figure 3. da/dN vs. ∆K curve of 5C (red full colored) specimens tested in air superposed on curves of 5C (red) and 6C (blue) specimens tested under vacuum at room temperature and Z=0.1.

3.2. Microfractography of fracture surfaces obtained under different environments

A comparison of the fracture surfaces of specimens tested in air and under vacuum is given in Fig. 4. The propagation in air, Fig. 4a, seems to be predominantly transgranular, very similar to a conventional stage II crack propagation mechanism. On the contrary, the spongy aspect of the fracture surface under vacuum, over the whole area, cannot be easily referred to some known crack path morphology. However, such a featureless aspect could be in accordance with an intergranular crack path, as can be seen in Fig. 4b. This hypothesis will be explored further (see section 3.3).

A quantitative explanation of the effect of environment together with the grain refinement on the fatigue threshold is not straightforward. Actually the FCGRs in this domain are, in addition to environment and crack closure, strongly influenced by several factors including slip mechanisms in accordance with deformation localization, grain (cell) dimensions, crack branching and deflection.
Figure 4. Fracture surfaces of UFG-Cu specimen tested in, a) air $\Delta K_{eff} \sim 5.5$ MPa$\cdot$m, $da/dN = 4E-9$ m/cycle, and in b) vacuum $\Delta K_{eff} \sim 5.5$ MPa$\cdot$m, $da/dN = 4E-11$ m/cycle [arrows show propagation direction].

Taking into account the fracture mechanisms approach and the real material microstructure, there exists a transition between near threshold regime and the intermediate fatigue stage when the size of the cyclic plastic zone (CPZ) at the crack tip becomes comparable with the grain size [12]. In the case of UFG microstructure the estimated size of the CPZ is much greater than the grain size. Hence, a large number of neighboring grains are involved in the crack tip plasticity even near the threshold. This is illustrated in Fig. 5 on the lateral surface of the UFG specimen. There is an obvious difference between the area in the vicinity of the crack, crack flanks, in air and vacuum (compare two images on the top of the Fig. 5).

The extensive cyclic plasticity and slip activity in vacuum even at the threshold value ($\Delta K \sim 5.5$ MPa$\cdot$m) is remarkable. The existence of well-developed slip bands around the crack witnesses for severe concentration of the deformation under vacuum.
3.3. FIB cuts
In order to investigate the extent of the cyclic plastic deformation at the crack tip, Tescan LYRA 3 XMU FEG/SEM scanning electron microscope equipped with a FIB technique was used for examining the microstructure beneath the fracture surface as well as in the vicinity of the crack tip. For this purpose, the area under study was first covered by a platinum layer to protect the surface morphology from any damage that can be caused during FIB cutting as documented in Fig. 6.

![Image of FIB cuts](image)

**Figure 6.** Positions of FIB observations on the 6C specimen, a) crack flanks in vacuum, b) picture of the crack on the lateral surface of specimen and c) crack flanks in air.

Fig. 7 shows two FIB cuts performed through a crack with nearly the same $\Delta K$ value perpendicular to the plane of crack propagation and to the lateral surface. The microstructures beneath the fracture surface created under vacuum and air are shown in Fig. 7a and 7b, respectively. The UFG structure remains without any changes in the regions far
from the fracture surface; however, the grains adjacent to the fracture surface are visibly coarsened.

![FIB section of a crack grown in, a) air $\Delta K \sim 8.14$ MPa√m, $da/dN = 5.3E-10$ m/cycle, and in b) vacuum $\Delta K \sim 7.6$ MPa√m, $da/dN = 9.5E-11$ m/cycle.](image)

**Figure 7.** FIB section of a crack grown in, a) air $\Delta K \sim 8.14$ MPa√m, $da/dN = 5.3E-10$ m/cycle, and in b) vacuum $\Delta K \sim 7.6$ MPa√m, $da/dN = 9.5E-11$ m/cycle.

The border line between the grain-coarsened area and the UFG microstructure can be easily distinguished. The size of the cyclic plastic zone ($R_{cp} \sim 16$ µm) approximately corresponds to two times the width of the region where grain coarsening occurs. These observations are in agreement with the findings of Vinogradov et al. [14]. The effect can be attributed to the dynamic grain coarsening and development of characteristic dislocations structures observed in low cycle fatigue testing at relatively large strain amplitudes [15, 16].

**4. Conclusions**

From this preliminary study on the influence of the testing environment on the fatigue crack propagation in UFG copper, the following conclusions can be made:

1) In the region of low $\Delta K$ values, the faster fatigue crack propagation in UFG copper in air is explained by a reduced shielding effect of crack closure when compared to the conventional CG copper.

2) At the very low crack growth rates, the fracture surface of the CG material exhibits a higher roughness associated to the intergranular facets which contribute to the shielding effect of crack closure in contrast with the very even and smooth fracture surface in the UFG material.

3) The similitude of the effective crack propagation behavior after closure correction suggests that the same crack growth mechanism (transgranular stage II propagation related to mode I crack opening) operates in both materials regardless of the difference in the grain size.

4) For $\Delta K$ values higher than $7$ MPa√m, the crack growth rates are similar in UFG and conventional copper.

5) A huge effect of air environment in comparison to the high vacuum is put forward in the UFG copper. This effect is accompanied by a strong crack path deviation which is probably related to a shear controlled slip localization and seems to result from an intergranular propagation mechanism.
Observations using a FIB technique revealed a substantial grain coarsening in the vicinity of the crack tip, and crack wakes in the UFG microstructure. Very tortuous and branched crack path under vacuum was very different from a transgranular stage II crack path in air.

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