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USE OF ATOMIC FORCE MICROSCOPY IN ASSESSING SURFACE DAMAGE DUE TO FATIGUE

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ABSTRACT

Strain controlled fatigue tests were conducted on polycrystalline copper at $\Delta\varepsilon/2$=0.161% and 0.255% to various fractions of fatigue life. Scanning electronic microscope (SEM) and atomic force microscopy (AFM), a relatively new tool that readily provides high resolution digitized images of surface features, were used to describe and quantitatively characterize the evolution of surface deformation during fatigue. To quantify surface deformation, a parameter γ^{irrev} is defined that is a measure of the local slip irreversibility at the surface. This parameter applies to any type of surface deformation feature, is independent of the size of the fields of view and yields information on the distribution of surface strains over the specimen gage length. From this formalism, a criterion for crack nucleation is proposed using actual AFM surface strain measurements.

KEYWORDS

Damage, Fatigue, Copper, AFM, SEM, Extrusions, Protrusions, Slip-bands

INTRODUCTION

Although engineering models for predicting fatigue life are available, damage evolution that leads to the formation of a macro-crack is difficult to predict, because no easily measurable parameter uniquely describes the state of damage during this stage. The objective of this study is to quantitatively describe the evolution of the surface features that develop in copper polycrystals up to crack nucleation. Because fatigue crack nucleation is a surface phenomenon, fatigue damage is better characterized by changes at the surface rather than by alterations in the interior of the material and, therefore, scanning electron microscopy (SEM) and atomic force microscopy (AFM) were chosen as analytical tools to perform this study. AFM is a relatively new technology that provides high-resolution three-dimensional images of the surface and digitized information about the surface topography providing accurate and quantitative measurements of the surface features.

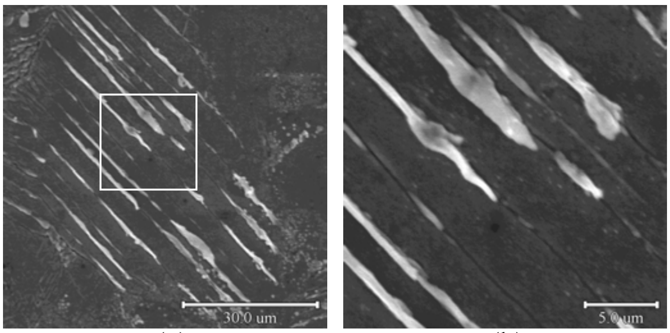
EXPERIMENTAL PROCEDURE

Strain controlled fatigue tests were performed on high purity C101 grade polycrystalline copper (OFHC). The fatigue tests were performed on standard axial specimens with a surface preparation that consisted of a combination of mechanical and electrochemical polish. The grain size of copper was estimated by mean intercept length method at about 40 μm after a heat treatment of one hour at 500°C. The fatigue tests were conducted using a triangular waveform at a strain rate of 0.005 s^{-1} and a stress ratio R=-1. The strain amplitudes enforced on specimens during the fatigue tests were 0.161% and 0.255%, and yielded fatigue lives of 75,900 cycles and 6,900 cycles, respectively. Several specimens were tested at each strain amplitude and all but one test was arrested prior to failure to allow the observation of surface topography by AFM at fractions of the fatigue life of about 0.5 and 0.9. Failure was declared once a macroscopic crack was observed at the surface. The fatigue tests were followed by sectioning of the specimens for post-test observations by SEM and AFM. The AFM used in this study was an Aris-3500 with a long range scanning module METRIS-3070. About twenty scans were analyzed along the length and at several positions around the circumference of each specimen. The collection of AFM images was submitted to a verification procedure described elsewhere [1] to ensure that the area covered by the AFM scans was representative of the whole surface.

RESULTS AND DISCUSSION

Characterization of Surface Damage

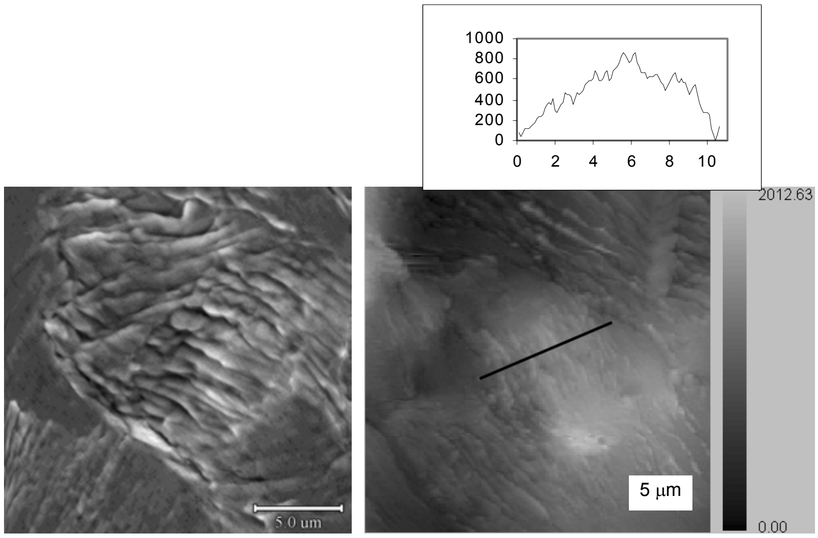
The most common occurrence of surface deformation in cyclically loaded polycrystalline copper is in the form of slip bands regularly distributed within grains. The slip bands in specimens tested at 0.161% strain amplitude, had heights that varied from 30 nm and 900 nm, Figure 1. Slip bands were observed at both applied strain amplitudes, although towards the end of the fatigue life in 0.255% strain amplitude tests, protrusions became the dominant form of surface damage. According to Suresh's definition [2], a protrusion is a surface upset many μm in height, where a macro-PSB, tens of μm wide and containing tens of matrix and/or PSB lamellae, emerges at the free surface. This definition applies to single crystals, where PSBs span across the entire width of the sample and clusters of similarly oriented slip bands can form over the whole surface. Protrusions with characteristics such as reported by other research groups for copper single crystals [3,4] were obviously not observed in this study with 40 μm grains, but AFM measurements showed that a considerable bulging of the surface (about 1 μm) took place across the width of some PSBs that had covered a significant portion of a grain, Figure 2. The typical width of the protrusions was between 10 and 20 μm, creating a height to width ratio smaller than measured in single crystals. This is consistent with findings on copper single crystals by Hunsche and Neumann [4] that showed a decrease in the height of the protrusions with a reduction of the thickness of the single crystals, explained by availability of less material to produce the upset. In the present study, the term protrusion is used when PSBs occupy the greater part of a grain and the density of the slip bands forming the PSB has reached a high enough level that no band of matrix is visible between the slip bands.



(a) (b)

Figure 1: Typical appearance of slip bands at the surface in large grains in polycrystalline copper cyclically loaded at a strain amplitude of 0.161%. (b) magnified picture of the boxed area in a.

At 0.255% strain amplitude, the average height of slip bands at N/N_f=0.25 is 108 nm, which shows that a significant growth occurs early in the fatigue life and then increases to 247 nm at failure. The latter compares well to the average height of 271 nm obtained at failure for the 0.161% strain amplitude, a fact that actually supports the theory by Essmann et al. [5] that relates the surface roughness to point defect formation in the bulk, which is limited by the size of the grains in polycrystals. As a result, the growth of slip bands in

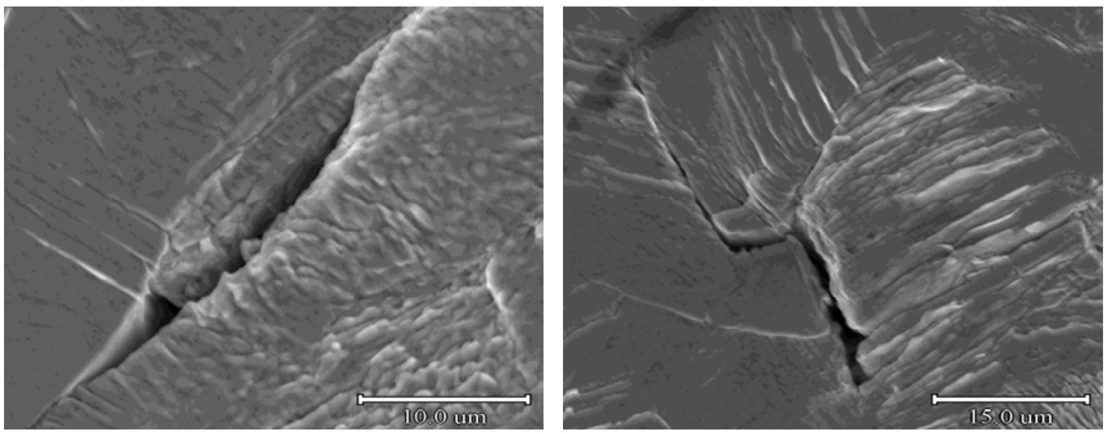


(a) (b)

Figure 2: Example of a protrusion in polycrystalline copper formed during fatigue at a strain amplitude of 0.255% (a) SEM photo and (b) AFM photo. The loading axis is along the vertical and the gray scale to the right of the AFM photo refers to the height of the features.

polycrystalline copper with grains on average 40 μm in diameter seems to saturate at a height of about 250 nm.

Fatigue crack nucleation in copper specimens at both strain amplitude occurred systematically at grain boundaries in regions with significant surface deformation, Figure 3. More specifically, in almost every case, cracks initiate at the boundary between one grain with considerable surface upset and another grain that does not show much trace of surface deformation.



(a) (b)

Figure 3: Typical intergranular cracks in different specimens of Cu cycled at different strain amplitudes. Cracks tend to form on boundaries between grains that show considerable surface deformation and those that are relatively free of deformation.

Criterion for Fatigue Crack Nucleation

The potential of high resolution devices capable of quantitatively describing the surface topography, such as scanning tunneling microscopy (STM) and atomic force microscopy (AFM), was discovered in the early 90's and used for accurately measuring the height of surface features in various materials [6-12]. Quantitative parameters were defined from these measurements to describe the state of surface deformation, such as the average slip distance [6] or the ratio of the average height to the average spacing between the surface features [7] and root mean square (RMS) height of the surface [9]. A direct relationship exists between these parameters and the average accumulation of surface plastic strain, which was exploited in these studies to determine the irreversibility of slip at the surface. Although, the use of average values of surface parameters is correct to describe the general state of surface damage, the extension of their use to the determination of the onset of the nucleation of fatigue cracks, which is by nature a heterogeneous process, is beyond the capabilities of such parameters. Indeed, microcrack nucleation is always preceded by a local concentration of surface deformation and only the local cyclic plastic strain at this site is representative of fatigue damage, not the average deformation over the entire surface.

This study attempts to remedy the above shortcoming by characterizing the entire distribution of surface displacements instead of just the average value. In addition, the irreversible surface deformation parameter defined in this investigation to quantify surface deformation is not limited to the height or height to spacing ratio, but combines measures of length, height and number per unit area of the surface features and is therefore able to accommodate different types of surface deformation features. Furthermore, it does not rely on spacing between slip bands, which makes it also applicable to surface features that develop individually as was the case with one of the materials studied here. The irreversible surface deformation parameter is defined as follows (a detailed derivation is provided in [13])

$$\gamma^{irrev} = \frac{\sum_{j=1}^{m}\left(\sum_{i=1}^{n} 2|\delta_i| a_i^{norm}\right)_j}{\sum_{j=1}^{m} L_j} \tag{1}$$

where δ is the height or depth of a surface feature, a^{norm} is its length normalized relative to the size of the field of view (FOV), L is the length of the FOV, n is the number of slip bands per FOV and m the total number of FOV. The numerator consists, therefore, of the total amount of normal deformation at the surface, while the denominator represents the total length of the fields of view, which is effectively the gauge length.

When considering the entire surface of a specimen, equation (1) provides an average value of γ^{irrev}, which is not pertinent to the study of the onset of fatigue crack nucleation, because the latter is the result of local surface deformation. Therefore, the measured average values of γ^{irrev} are not reported here, but can be found elsewhere [13]. On the other hand, the following analysis of the distribution of γ^{irrev} can provide valuable information on the onset of fatigue crack nucleation.

Due to the high level of heterogeneity in fatigue surface deformation, it is virtually impossible to predict where the fatal crack will nucleate and monitor changes in topography at that precise location. However, even though the local maximum of γ^{irrev} cannot be directly measured, by statistically characterizing the distribution of surface damage in a large number of FOVs, it is possible to predict the amount of damage in the region with maximum local surface damage, even though that specific region is not directly included in the FOVs. It was found that the distribution of γ^{irrev} resembles a Gaussian distribution where only a few regions have extreme amounts of surface upset (high or low) and the major portion of the surface has a local amount of damage that is close to the sample average [13]. The distribution is in fact adequately modeled by a continuous normal distribution function, which has the advantage that it can be easily integrated and has an area under the curve equal to one, corresponding to 100% of the specimen's surface. Figure 4 shows the distribution of γ^{irrev} with the horizontal axis representing ranges of γ^{irrev} and the distribution indicated on the vertical axis.

Since the normal distribution curves of Figure 4 reveal the extreme values of irreversible surface deformation reached in some regions, these plots directly provide information on the advancement of surface upset in the highly deformed regions. Thus, this data can be used to predict when the material reaches a critical state that will trigger the nucleation of a fatal crack. This analysis is however better performed on the cumulative version of these plots shown in Figure 5, which is simply the integral of the plots in Figure 4. The vertical axis on the left indicates the portion of the surface that contains amounts of irreversible surface deformation between zero and the amounts indicated on the horizontal axis of the graph. Conversely, the vertical axis on the right provides the fraction of the surface that has developed amounts of irreversible surface deformation larger than the amount indicated on the horizontal axis.

The previous AFM and SEM observations clearly showed that fatigue cracks nucleate in regions with significant surface deformation. Therefore, a criterion for crack nucleation may be defined in terms of a critical *local* value of γ^{irrev} necessary for crack nucleation. This critical value of γ^{irrev} can be determined from AFM measurements by measuring the value of γ^{irrev} of FOVs that contain a crack nucleus. Once known, this value can be traced on the normal distribution curves of the irreversible surface deformation, as shown by the vertical lines shown on Figure 5. It was found that the levels of local surface deformation necessary to trigger the nucleation of a fatigue crack are similar between the low and the high amplitude tests in stainless steel but they differ between the two strain amplitudes in copper [13].

It is important to note that the criterion described above does not act like a "failure or no failure" switch that would imply that a crack is always nucleated once the critical value of surface deformation is achieved in a specific location. On the contrary, certain portions of the surface may develop levels of surface deformation well beyond the critical value without nucleating a fatal crack, as observed in all failed specimens and in some specimens tested to 90% of the life. The actual fraction of the surfaces that has reached or exceeded the crack nucleation criterion is indicated by the ordinate of the intersection of the distribution curves with the vertical line that specifies the criterion. Now, if one postulates that the likelihood of nucleating a crack increases with the fraction of the surface over which the critical level of surface deformation is exceeded, the distribution curve of the irreversible surface deformation then provides an estimation of the probability for crack nucleation.

CONCLUSIONS

From the SEM and AFM analyses of the surface deformation of cyclically loaded polycrystalline copper, the following conclusions were drawn.

Extrusions are the principal surface deformation features in copper tested at 0.161% and 0.255% strain amplitudes, with the development of protrusions later in the fatigue life at the higher strain amplitude. Slip bands reach on average 250 nm in height at both strain amplitudes, which tends to indicate that the growth of slip bands saturates at this level in copper with a grain size of 40 µm. In all tests, fatigue cracks nucleated at grain boundaries and seemed to be driven by the strain mismatch between adjacent grains. From the digitized description of the surface topography, a procedure was developed to quantitatively assess surface damage at any given point in the fatigue life.

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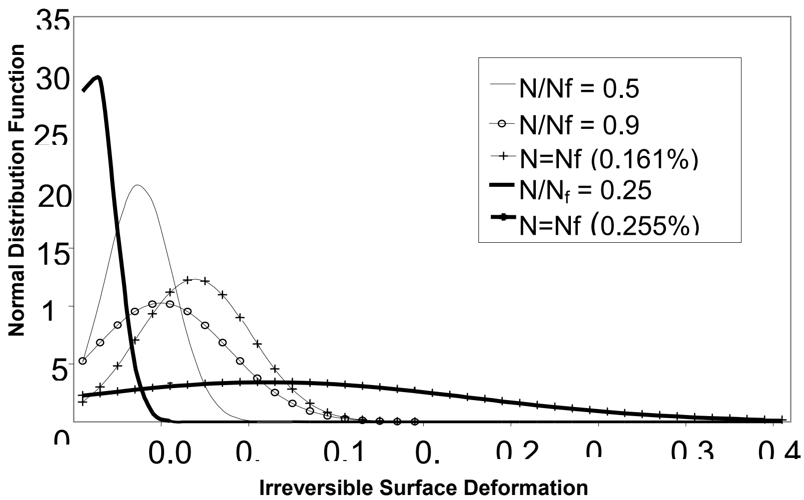

Figure 4: Representation of the distribution of the irreversible surface deformation at the surface of fatigue specimens using the normal (Gaussian) distribution function for polycrystalline copper (at 0.161% and 0.255% applied strain amplitude).

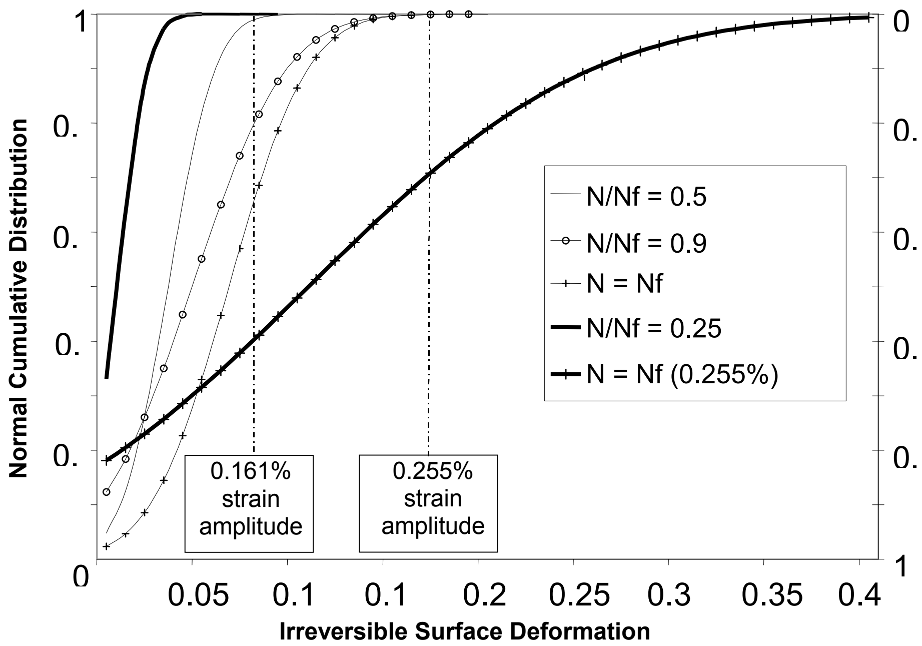

Figure 5: Polycrystalline copper (tested at 0.161% and 0.255% strain amplitude). The right axis corresponds to one minus the left axis, and the vertical dotted lines represent the critical values of the irreversible surface strain.

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