QUANTITATIVE ASSESSMENT OF THE SURFACE RELIEF IN FATIGUE USING AFM

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ABSTRACT

Atomic force microscopy (AFM) was applied to study the early stages of fatigue damage in polycrystalline copper and stainless 316L steel. This high-resolution technique allows to study the surface relief evolution from the onset of cycling up to the end of fatigue life. The analysis of the image formation using an AFM tip reveals the lateral distortion of the extrusions and intrusions in case of high extrusions and deep intrusions. The true extrusion height can be obtained in direct observation and the depth of intrusions using replica technique. The evolution of the shape and the height of intrusions allowed to judge the activity of the persistent slip bands (PSBs) during fatigue life. The form of extrusions and intrusions was found in agreement with predictions of recent models of fatigue crack initiation.

KEYWORDS

Fatigue, copper, 316L steel, atomic force microscopy, surface relief, extrusions, intrusions, extrusion growth.

INTRODUCTION

Surface relief that is formed on the surface of crystalline materials during cyclic loading has been studied intensively for one hundred years. One of the first studies of the surface relief evolution due to fatigue loading appeared in 1903 when Ewing and Humfrey [1] published the results concerning the surface changes of the polycrystalline Swedish iron cyclically loaded in reversed bending at room temperature. They used optical microscopy to reveal inhomogeneous distribution of the surface damage. The damage was localised in the bands of intensive slip, much later called persistent slip bands (PSBs). The most important finding was the identification of the first fatigue cracks within the rough surface relief. These observations started the study of the mechanisms of fatigue damage evolution in materials.

Further important stage of the identification of the mechanisms of the surface relief formation represents the discovery of extrusion and intrusion growth by Forsyth [2] in 1953 and the evolution of the general knowledge concerning the internal dislocation structure of the fatigued materials, namely, the discovery of the relation between the dislocation structure of the persistent slip bands and the surface relief [3, 4]. Further step represents the understanding of the role of point defects in cyclic straining and their interaction with dislocations that led to the proposal of new models of surface relief formation and crack nucleation [5, 6].

In order to refine existing models or propose new theories of crack nucleation a detailed knowledge of the surface evolution and early crack nucleation is necessary. The quantitative data on the height and profile of extrusions, on the depth and profile of intrusions and the kinetics of their formation can help to find the details of the fatigue crack initiation mechanisms.

The advancement of our knowledge on the surface relief and damage evolution in fatigue is closely related with the level of experimental techniques. In the early decades of the last century only optical microscope was available. The height of surface roughness can be studied using interference microscopy (e.g. [7-11]). Considerable progress represented the use of transmission electron microscope and the method of plastic replicas (e.g. [12-14]). Even more important step was the use of the scanning electron microscopy allowing to study directly the surface of fatigued materials with high resolution.

In recent years, several new methods based on the scanning electron microscopy were applied: (i) the contamination line technique [15, 16], (ii) the section micromilling technique [17, 18], (iii) sharp corner polishing technique [19-21], and (iv) true replica sectioning technique [20]. The first method has limited resolution and is difficult to apply to all materials. The disadvantage of other methods is the destruction of a specimen. The improvement of the scanning electron microscope using FEG (field emission gun) electron source allowed to increase substantially the resolution of the direct observations of the surface [22].

In the nineties, a few new techniques were adopted to study fatigue mechanisms in metals. Scanning tunnelling microscopy (STM) and atomic force microscopy (AFM) belonging to the family of scanning probe microscope (SPM) techniques became powerful tools because the surface morphology can be observed with nearly atomic resolution. STM, the first invented representative of SPM, has been used for the study of fatigue crack initiation [23-25]. Since STM requires a specimen with high electrical conductivity, AFM started to be applied most frequently for the surface damage observation in cyclic straining. It has been employed to study surface morphology both of material specimens [26-32] and plastic replicas [33]. It was shown that AFM and EBSD (electron backscattered diffraction) represent very useful combination to obtain topographic and crystallographic aspects of fatigue damage [29, 34]. Although three-dimensional measurement of early surface damage was also possible with scanning acoustic microscopy (SAM) [35], the application of this method is scarce due to its low resolution.

In this work a detailed study of the topography of the surface relief at the emerging PSBs in fatigued polycrystalline f.c.c. metals namely copper and 316L stainless steel has been performed with the aim to demonstrate the possibilities and application of this technique to the analysis of the fatigue crack initiation. An importance of quantitative data characterising the intrusion and extrusion profiles and their evolution during the fatigue life is emphasised.

EXPERIMENTAL DETAILS

Two polycrystalline materials, copper and austenitic stainless 316L steel were studied. Copper of 99.99% purity after cold drawing and annealing had the average grain size 50 μm.

The average grain size of austenitic steel was 100 μm. Further details concerning the materials, their mechanical properties and cyclic stress-strain response can be found elsewhere [36, 37].

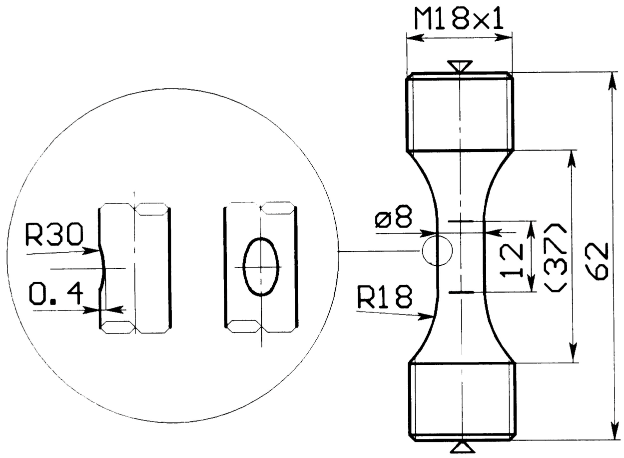

Fig. 1. Shape and size of a specimen (dimensions in mm).

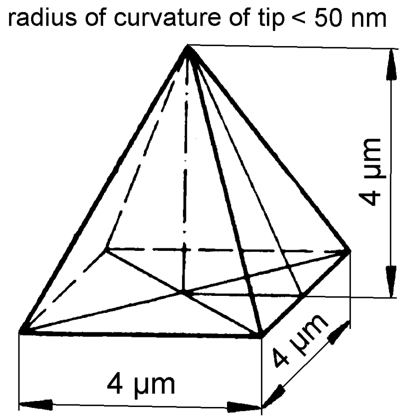

Fig. 2. Geometrical dimensions of an AFM tip.

Cylindrical specimens with threaded ends had a gauge diameter and a length of 8 and 12 mm, respectively (see Fig. 1). The shallow notch in the centre of the gauge length made the study of the surface relief easier. The notch area was mechanically and electrolytically polished in order to achieve a smooth surface. In the case of stainless steel a fine circular marks 400 μm in diameter were engraved on the central part of the polished notch before cycling for easier orientation on the specimen surface. The specimens were cycled in symmetrical push-pull straining with constant plastic strain amplitude. Further information on the testing conditions are given elsewhere [36, 37]. In the study of the surface damage evolution in stainless steel the cycling was periodically interrupted and the specimen was being removed from the testing machine for observation. The systematic surface relief study in the selected grains of the notched area was thus possible.

The details of the surface relief topography were observed in atomic force microscope Accurex IIL (Topometrix). Contact imaging mode in the air was used to obtain constant-force topographic images. V-shaped silicon nitride cantilever with standard pyramidal tip having the radius of curvature smaller then 50 nm was applied. The shape and dimensions of the tip are apparent from Fig. 2. The optical microscope equipped with CCD camera was used to find the appropriate spot inside the circular marks on the specimen surface. Profiles of the PSBs were determined using the implemented software.

RESULTS

Polycrystalline copper

Surface of the polycrystalline copper cycled with constant plastic strain amplitude $\varepsilon_{ap} = 5\times10^{-4}$ for 25 000 cycles (~ 20% N_f) is shown in Fig. 3. Only one slip system has been activated in the grain. The surface slip traces are approximately perpendicular to the specimen axis. The majority of the relief is formed by parallel extrusions but several well perceptible intrusions

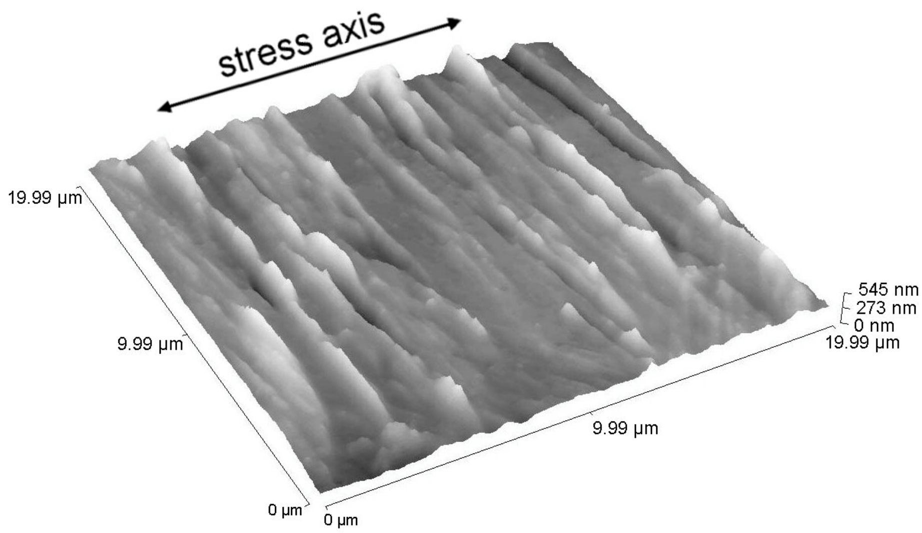

Fig. 3. Surface relief in a grain of polycrystalline copper cycled with constant plastic strain amplitude 5×10^{-4} for 25 000 cycles, AFM.

are also present. The extrusions are band-like and intrusions run parallel to extrusions. The height of extrusions along PSB markings, relative to the original neighbouring surface, is not constant. Maximum extrusion height was 400 nm.

The details of the surface relief in a different grain with the profiles in a section perpendicular to the surface markings (marked by a black line on the surface) are shown in Fig. 4. A band-like extrusion of the individual PSB formed at the grain boundary is apparent in Fig. 4a. The different levels of the non-deformed surfaces of both grains can be seen in sections 1 and 2 in Fig. 4b. In all cases the extrusions are accompanied by parallel intrusions. Some intrusions are

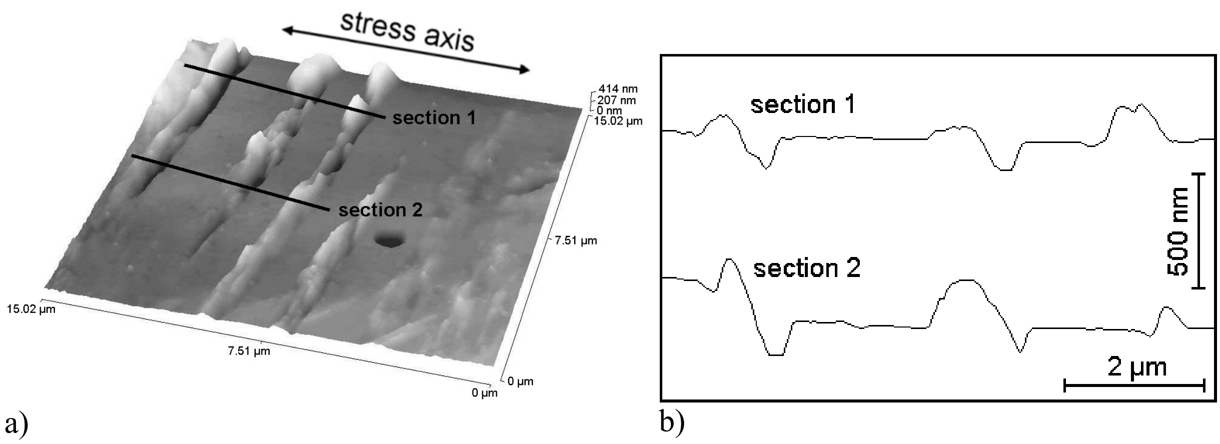

Fig. 4. A detail of surface relief of individual PSB markings in a grain of polycrystalline copper cycled with constant plastic strain amplitude 5×10^{-4} for 25 000 cycles, (a) 3D image, AFM, (b) profiles of extrusions and intrusions in sections perpendicular to the markings (marked in (a)).

only very shallow and thin.

Another important feature of the surface relief in fatigued polycrystalline copper is the variable height of extrusions along the PSB markings. Rather quasi-periodic alterations of extrusions and intrusions superimposed by ribbon-like extrusion characterise the shapes of individual PSB markings (Fig. 4a). Both extrusion and intrusion morphologies display a profile of a non-symmetric triangle (Fig. 4b). The slope of the right side of the triangular extrusion, or the left side of the triangular intrusion with the original surface is smaller than the slope on the opposite side.

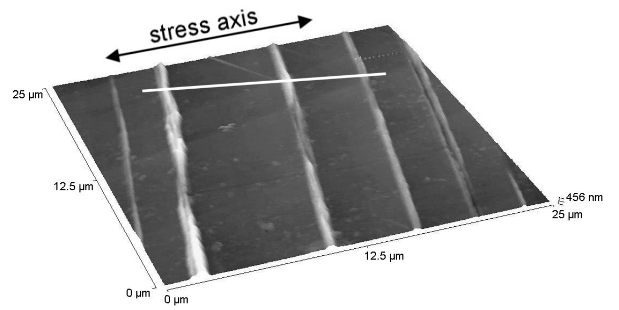

Fig. 5. Surface relief in a grain of 316L steel cycled with $\varepsilon_{ap} = 2\times10^{-3}$ for N = 25 000 cycles (~ 50% N_f), AFM.

Austenitic steel

Surface relief evolution in austenitic stainless steel 316L was studied in a specimen cycled with plastic strain amplitude $\varepsilon_{ap} = 2\times10^{-3}$. Figure 5 shows an AFM micrograph of the specimen surface after 10000 cycles (~ 50% N_f). Several band-like extrusions of different width and one shallow intrusion (the fifth PSB marking from the left) are apparent along the intersection of PSBs with the surface (Fig. 5). The surface slip traces are inclined at an angle 75 degrees to the loading axis. The surface relief evolution of fatigued 316L steel has numerous features similar

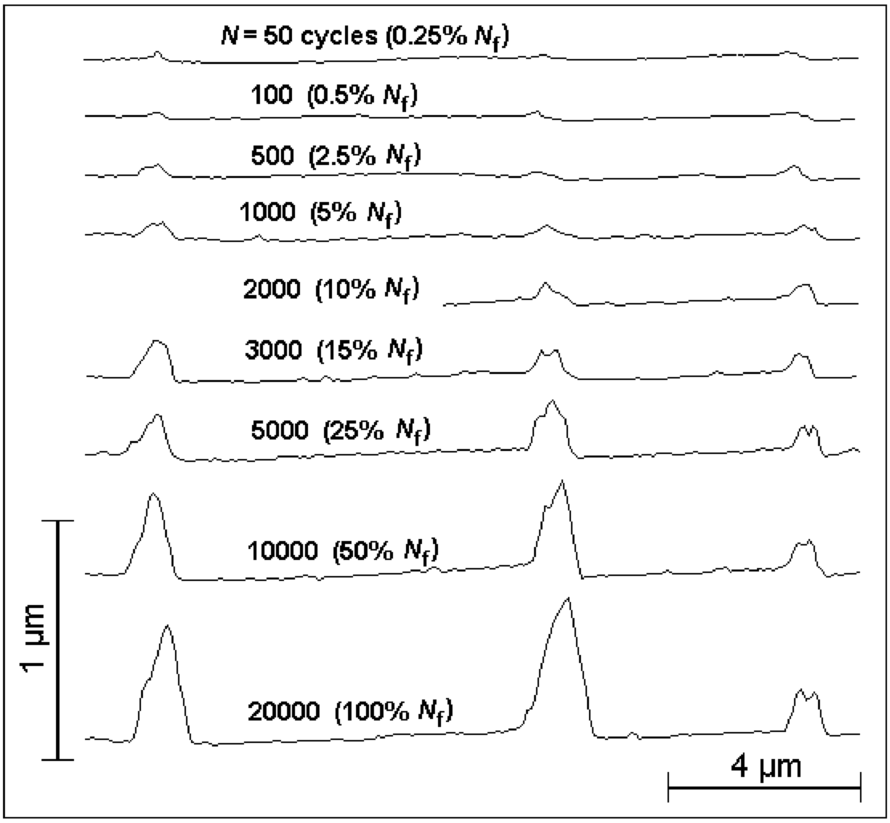

Fig. 6. Characteristic sequence of surface relief profiles in a section marked in Fig. 5 in a grain of 316L steel cycled with $\varepsilon_{ap} = 2\times10^{-3}$.

to those found on polycrystalline copper. However, as discussed later, the intrusions can not be imaged in direct observation of the metal surface and can be revealed only using plastic replica of the surface. The height of the extrusions along the PSB marking does not vary as much as in copper but the changes in height and in width of extrusions are apparent.

The quantitative data on the evolution of the surface relief topography within the whole fatigue life of the specimen are presented in Fig. 6. The surface relief profiles were obtained in a section perpendicular to the surface slip markings. The series of surface profiles in the identical section, shown by a white line for 10 000 cycles in Fig. 5, during the fatigue life of the specimen is shown in Fig. 6. Maximum care was paid to obtain these profiles always at the identical location.

The first elevations of the specimen surface in this particular grain could be detected as early as after 50 cycles. In some other grains the first localised surface relief was detected already after 20 cycles. Already in this early stage the surface relief was formed along the whole slip line running through the grain [32]. The surface relief at 50 cycles has some features resembling unidirectional straining but already at this stage a hill like geometry is present (Fig. 6). While the unidirectional step of the same height is present during the whole fatigue life, the extrusion height and width developed considerably. The width of extrusions increased essentially during the first 500 cycles. In this phase a characteristic hill profile was formed.

In further cycling the height of extrusions increased continuously. The height of the middle extrusion in Fig. 6 was only 19 nm at 50 cycles and at 20 000cycles its height was 562 nm. The growth rate of the left extrusion in Fig. 6 is slightly smaller but the extrusion also grows continuously during the whole fatigue life. It must be stressed that the cyclic straining of the grain was not affected by the presence of a crack in the neighbourhood of the grain [32]. The profiles of well developed extrusions have a shape similar to that observed in copper. It is a triangle whose sides have slightly different slopes.

DISCUSSION

The application of the atomic force microscopy in the study of fatigue damage, namely in the study of cyclic plastic strain localisation that precedes the fatigue crack formation, revealed the possibilities to obtain quantitative data on the surface profiles at a nanometric scale. These data are very important for the verification of the predictions of the proposed theoretical models of surface relief formation and fatigue crack initiation.

In fatigue loaded metals both extrusions and intrusions were found. In polycrystalline copper the intrusions were found either adjacent to the ribbon-like extrusion or inside the PSB marking in quasi-periodic alternations with extrusions. In the case of 316L stainless steel, in agreement with our previous results [29,31], the intrusions were much less frequent. In order to verify our findings concerning the low frequency of intrusions in 316L steel the additional observation using plastic replica of the surface were performed [38]. This method allowed to identify thin parallel intrusions adjacent to wider extrusions. Both surface morphologies, i. e. the ribbon-like extrusions with parallel intrusions and alternating extrusions-intrusions along the PSB marking were predicted by the model proposed by Polák [39].

The profiles of extrusions and intrusions are in most cases triangular. The observed asymmetry of the profile of extrusions and intrusions is due to the inclination of the active slip plane to the surface of the grain. The amount of asymmetry depends on the angle of the active slip plane with the surface and the angle of the PSB markings with the loading axis. These considerations are in agreement with previous findings that also in case of polycrystalline materials the surface relief of PSBs grows in the direction of the Burgers vector of the active slip system [29]. This finding agrees with the models of surface relief formation proposed by Essmann et al. [16,40] and Polák [39].

The discrepancy between the surface profiles of the PSB markings in the identical areas obtained by direct observation of the metal surface and using inverse copy of the surface with the help of plastic replica revealed some distortion of the real geometry with AFM. In order to identify the distortion of the real geometry of the surface the shape of the tip must be taken into account. The vertex angle of our tip (see Fig. 2) was minimally 53.1 degrees and maximally 70.5 degrees. Due to this geometrical limitations the non-distorted images can be obtained only of those extrusions or intrusions whose sides contain the angle less than 55 degrees with the horizontal surface. In case of less developed relief in copper (Fig. 4b) this geometrical condition fulfilled (the maximum side angle was 45 degrees). In case of 316L steel the side angle of extrusions for cycle number above 3000 is over 50 degrees (see Fig. 6). Due to tip geometry the lateral distortion is inevitable. The actual width of extrusions is smaller than as recorded by AFM. The height of extrusions, however, is not affected. Therefore, the true quantitative data about the extrusion growth in cyclic loading can be obtained.

The distortion can be much more important in case of intrusions. Parallel intrusions accompanying extrusions are usually thin and deep. These intrusions can escape the detection using AFM in case of direct observation of metal surface. Using replica technique [38] their depth can be recorded without distortion, however, their actual width will be considerably lower than the recorded one.

The systematic study of the surface relief evolution in 316L steel revealed the characteristic changes of the relief connected with the individual PSBs. The slip steps (accompanied already by a hill-like extrusion), which were observed at the onset of cyclic straining, are typical for unidirectional straining and were also observed by Hunsche and Neumann [17] in early stages of fatigued copper single crystals. The important fact, deduced from the growth of extrusions (Fig. 6), is the continuous growth of extrusions during the whole fatigue life in grains not affected by the presence of cracks. More detailed study of the kinetics of extrusion growth revealed two stages of the growth [32]. The initial growth rate is high ($\sim 8\times10^{-11}$ nm/cycle) and later the growth rate is stabilised to a value that is constant for most of the fatigue life ($\sim 1.6\times10^{-11}$ nm/cycle).

Qualitative and also quantitative data on form of extrusions and intrusions and the kinetics of extrusion growth during the fatigue lifetime of specimens can be reconciled with two recent models of surface relief formation [39, 40] and with the numerical simulation [41] of surface relief evolution based on generation, migration and annihilation of point defect during localised cyclic plastic deformation in PSBs.

CONCLUSIONS

The application of the AFM to the study of early stages of fatigue damage evolution in polycrystalline materials led to the following conclusions:

(i) The high resolution of AFM can reveal qualitative and quantitative details of the surface relief evolution and yield new knowledge on the mechanisms of fatigue crack nucleation.

(ii) The finite geometrical dimensions of the AFM tip can result in distortions of the real surface topography of steep extrusions and narrow and deep intrusions. The combination of direct observation and replica technique can lead to evaluation of these distortions.

(iii) The height of extrusions is truly recorded and important data on the extrusion kinetics were collected.

(iv) The cyclic strain localisation starts in the very early stage of the fatigue life and the PSBs can be active in producing extrusions and intrusions during the whole fatigue life.

(v) The details of the surface geometry of extrusions and intrusions are in accord with predictions of the Polák`s model of fatigue crack initiation.

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