Application of FIB technique to study of early fatigue damage in polycrystals

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Abstract. Focused ion beam (FIB) technique together with other advanced microscopic techniques were applied to study early microstructural changes leading to crack initiation in fatigued polycrystals. Dislocation structures of persistent slip bands (PSBs) and surrounding matrix were revealed in the bulk of surface grains by electron channelling contrast imaging (ECCI) technique on the FIB cross-sections. True shape of extrusions, intrusions and the path of initiated fatigue cracks were assessed in three dimensions by serial FIB cross-sectioning (FIB tomography). Advantageous potential of FIB technique and its other possible utilization in fatigue crack initiation studies in polycrystals are highlighted.

1. Introduction

Focused ion beam (FIB) technique represents a modern tool which has been used in various areas of materials research for studying or modifying material systems down to nanometre levels (for a review see e.g. [1]). In fatigue damage studies in polycrystals FIB has been used to study environment-assisted crack profiles in pipeline steel [2], the interaction of microcracks with selected interfaces in nickel-based superalloy [3] and in the study of short crack path in dual phase steel [4].

In comparison with the above papers the present work is directed to the study of early stages of fatigue damage, namely to the study of cyclic strain localization leading to fatigue crack initiation. The localization of the plastic strain in persistent slip band (PSB) is followed by the formation of persistent slip markings (PSMs) consisting of extrusions and intrusions. Numerous experimental studies proved the dominant role of PSMs in the process of fatigue crack initiation (for a review see e.g. [5,6]). Study of surface profiles of PSMs in individual cross-sections [7] and cumulative evolution of PSMs on two perpendicular surfaces [8] in 316L steel using FIB technique were performed recently. In this work the first results on dislocation structure of PSBs and three-dimensional (3D) form of PSMs and fatigue crack initiation obtained by FIB in conjunction with other microscopic techniques are reported.

2. Experimental

Flat specimens with a square cross-section of polycrystalline nickel and cylindrical specimens with a shallow notch of 316L austenitic stainless steel were cycled with controlled constant plastic strain
amplitudes to various stages of fatigue life. Central parts of both specimen geometries were mechanically and electrochemically polished before fatigue tests. Further details concerning the materials, specimens and fatigue tests can be found elsewhere [7–9].

The studies with the FIB technique in polycrystalline nickel (specimen IV used in our recent study [9]) were performed on a Zeiss CrossBeam 1540 XB instrument. Dislocation structures were studied by electron channelling contrast imaging (ECCI) technique in high-resolution SEM–FEG (Zeiss Ultra 55 FESEM). For serial cross-sectioning of PSMs in 316L steel (FIB micro-tomography) a FEI Dual Beam Helios NanoLab was used. Thin protective platinum layer was deposited on a small area of interest across PSMs before ion milling. U-shaped trench with one high quality cross-sectional surface perpendicular to PSMs and to the specimen surface was produced by FIB. Subsequently series of surface relief profiles were obtained by periodical removing of material within U-shaped trench parallel with the above high quality cross-sectional surface by FIB using automated software FEI Slice and View. In total, 500 sections were milled (300 sections with 20 nm slice thickness followed by 200 sections with 12 nm milling step).

3. Results and discussion

3.1. Dislocation structure and the depth of PSBs
Recent study of the local cyclic plastic strain within PSBs in nickel polycrystals revealed the broad spectrum of strain amplitudes with the average value around 1.5% [9]. Among factors, which can influence the local plastic shear in PSBs, the depth of PSBs and their dislocation arrangement beneath the specimen surface are of a great importance.

Figure 1a shows a grain of polycrystalline nickel cycled with the plastic strain amplitude \( \varepsilon_{ap} = 5 \times 10^{-4} \) for 30,000 cycles (0.6Nf). PSMs coming from two activated slip systems are apparent in this grain. The EBSD (electron backscattering diffraction) measurements [9] showed that this grain is demarcated by twin boundaries TB1, TB2 and TB3 and high angle grain boundary GB1, as indicated in figure 1b. The calculation of trace angles for the twin boundaries and PSMs both on the specimen surface and in the interior resulted in the construction of a tentative sub-surface profile of this individual grain and the possible PSB depth evaluation – see figure 1b.

To reveal the unknown trace of GB1 and the dislocation structure below the specimen surface combined FIB/ECCI technique was adopted. After finishing the half-cycle experiments [9] additional 2000 cycles with the same \( \varepsilon_{ap} \) were applied to specimen IV followed by a regular FIB cross-sectioning across GB1 (see figure 1b). Adoption of the BSE (back-scatter electron) contrast in SEM revealed the path of the grain boundaries GB1 and TB2 on the FIB cut below the specimen surface (figure 1b).

Dislocation arrangement both on the specimen surface and FIB-cut plane visualized by ECCI technique in SEM–FEG are documented in figure 2a. To improve the quality of ECCI micrographs the specimen was shortly electropolished which resulted in the slight rounding of FIB-cut edges and removing thin surface layer (see figure 2a). Detail of dislocation structure from the central area on the

![Figure 1](image-url)
FIB cross-section plane shows figure 2b. A PSB lamella with ladder structure lying parallel to TB1 is clearly seen. This finding perfectly agrees with the surface relief observation of the PSM going along the intersection of TB1 with the specimen surface (cf. figures 1 and 2b).

3.2. The shape of PSMs and fatigue crack initiation – FIB tomography

After the documentation of the specimen surface 3D form of PSMs in individual grains of cyclically strained 316L steel were studied by FIB either in regular cross-sections or in U-shaped trench using automated milling procedure. Here we will present only the results obtained by the latter way.

Figure 3a shows parts of two surface grains of 316 steel fatigued with $\varepsilon_{ap} = 1 \times 10^{-3}$ for 3800 cycles (8.3%$N_f$). Well developed PSMs in the right grain are equidistantly displaced whereas less developed PSMs in the left grain are present only locally (see the upper left corner in figure 3a). An individual sharp PSM which developed parallel to a thin twin separating these grains (see below) was chosen for the subsequent 3D FIB micro-tomography study. U-shaped trench created for this purpose around the area of interest is shown in figure 3b.

Figure 4 demonstrates an example of results obtained by FIB sectioning. The sectioning started from the lower end of PSM in direction to its centre. The profile of extrusion indicates that PSB is adjacent to a thin twin. The profile of the PSM changes. At the end of PSM only a thin extrusion was present. The extrusion widened and its height increased until in section 360 (figure 4b) the extrusion was well developed. The first indication of the starting crack was in section 390 (figure 4c). The growing crack follows twin boundary and later deviates from it (figure 4e). Figure 4e also reveals thin
intrusion accompanying the extrusion and running along twin boundary. This observation suggests that the fatigue crack started to initiate from an intrusion in the centre of the grain where the PSB depth is most pronounced. Further milling can prove or disprove this hypothesis.

4. Summary

The present work demonstrates that FIB, coupled with other advanced high-resolution techniques (SEM–FEG, ECCI, EBSD), represent an effective tool for systematic studies of the early fatigue damage and fatigue crack initiation in individual surface grains of cyclically strained polycrystals at submicroscopic level. Detailed data on the 3D form of PSMs and the initiation of fatigue cracks can be obtained. Dislocation structures corresponding to surface PSMs (with their possible 3D reconstruction) can be identified and the depth of PSBs (i.e. their length in the active slip direction) beneath the specimen surface can be evaluated. Another promising use of FIB technique in the early fatigue damage studies in polycrystals which has not been checked so far is the preparation of thin foils directly from specimen surface for transmission electron microscopy (TEM) analysis. Experimental data obtained by these modern high-resolution techniques will be invaluable for checking the validity and further advancement of theoretical models of surface relief formation leading to fatigue crack initiation.

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