IN-SITU HIGH TEMPERATURE LOW CYCLE FATIGUE STUDY OF SURFACE TOPOGRAPHY EVOLUTION IN NICKEL SUPERALLOY

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Abstract

In-situ Low Cycle Fatigue test (LCF) at temperature 635 °C have been performed in Scanning Electron Microscope (SEM) equipped with Electron Backscatter Diffraction analysis (EBSD) on a small dog-bone-shaped specimen of cast Inconel 713LC superalloy. The aim of the work was to study early stage fatigue damage at high temperature by the observations of the characteristic surface relief evolution and crystallographic characterization changes by EBSD. The detail of slip bands shape was checked by FIB and AFM microscopes. The LCF test was conducted on GATAN stage with pre tilted position and constant stress amplitude of total cycle number of 20. The relief produced in the first cycle determines the other locations of the localized cyclic slip to the primary slip planes (111). The relief was modified in the next cycles but without forming additionally new slip traces in the primary system. Based on EBSD analysis before and after LCF, the orientation of two grains was changed which caused activation of second slip system. The damage mechanism evolution is closely connected with the cyclic strain localization to the persistent slip bands where the fatigue cracks were initiated.

Keywords: In-situ, SEM, high temperature, fatigue, superalloy

1. INTRODUCTION

Precipitation strengthened nickel base superalloys exhibits excellent high temperature strength and hot corrosion resistance and therefore they are used for production of blades and discs of gas turbine engines [1,2]. The critical turbine parts are subjected to elastic-plastic straining due to repeated loading and thermal gradients during start-up and shut-down periods. It is thus important to study low cycle fatigue properties of these materials with the attention to the early damage mechanism linked to the nucleation of the fatigue crack. In previous papers studying low cycle fatigue of Inconel superalloys [3-5] the research have been concentrated on cyclic plasticity, fatigue life and dislocation structure cycled at 23, 500, 700, 800 and 900 °C. Cyclic straining of superalloys at all temperature is accompanied by strain localization that leads to the appearance of surface relief with persistent slip markings (PSMs). The characteristic dislocation structure in the interior grains of superalloys Inconel 713LC (IN713LC) was planar and consisted of numerous very thin nearly parallel persistent slip bands (PSBs). They run parallel to the (111) primary slip plane and intersect both the matrix and γ' precipitates [3]. In recent years considerable attention has been attracted to the study of the early damage mechanisms in crystalline materials, starting with the cyclic slip localization and resulting in the initiation of fatigue cracks and their early growth [6,7]. The aim of this paper is to conduct in-situ low cycle cyclic test at 635 °C of commonly used cast polycrystalline superalloys Inconel 713LC and to study in-situ evolution of the surface relief in the SEM and EBSD. Supplementary information about PSB structure and structure of PSMs is obtained using AFM and EBSD technique.

2. EXPERIMENTAL DETAILS

2.1 Superalloy IN713LC

Material in the present experiment was nickel-based superalloy IN713LC (LC in the name stands for “low carbon”) delivered by PBS Velká Bíteš a.s. (CZ). The superalloy has elevated percentage of Cr in order to enhance corrosion resistance. Its chemical composition is shown in **Table 1**. The macrostructure of the studied material was fully dendritic (see **Fig. 1a**) with average grain size (determined by linear intercept method) around 4.2 mm. It consists of carbides, eutectics γ/γ´ and pores [3]. **Fig. 1b** displays TEM micrographs of typical precipitate microstructure in the material. The average size of the main cuboidal precipitates is 450 nm [3]. TEM study revealed γ´ precipitate volume fraction of 56 % [3].

Table 1. Chemical composition of superalloy IN713LC (in wt.%).

Cr	Mo	C	Co	Fe	Zr	Nb	Al	B	Ti	Ni
11.90	4.57	0.050	0.08	0.19	0.010	1.96	5.75	0.013	0.70	Bal.

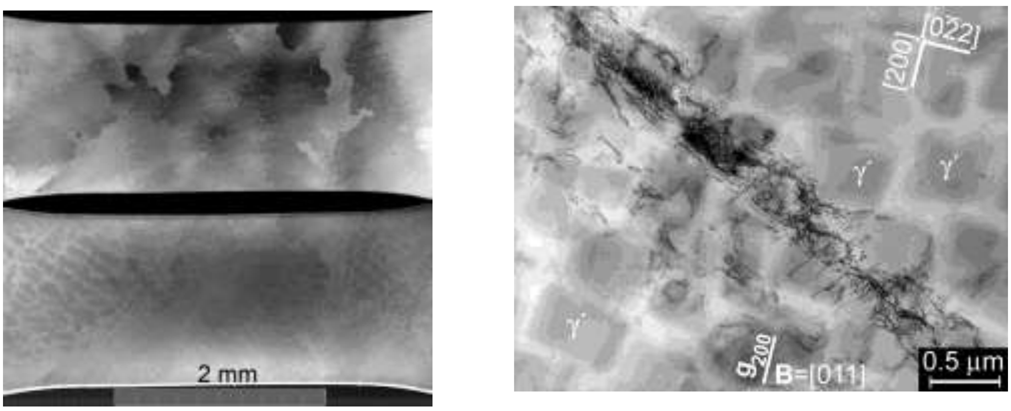

Fig. 1. The microstructure of superalloys IN713LC (a) SEM-BSE (the upper part) and SEM-SE micrographs of the specimen gauge length (the lower part) (b) TEM showing microstructure of the cyclically deformed specimen

2.2 In situ LCF test

Flat specimen (see the gauge in Fig. 1a) had rectangular cross section of 1.4 x 1.64 mm^2 and a gauge length of 2 mm. The specimen was tested and observed in-situ in the vacuum chamber of a scanning electron microscope TESCAN MIRA 3 with FEG cathode using GATAN stage Microtest™2000EW with EH 2000 heated grips tensile-compresion loading stage. The low cycle fatigue test was performed at temperature 635 °C in force control mode. Symmetrical stress cycle with stress amplitude 814 MPa was applied. The saturated plastic strain amplitude corresponding to this stress amplitude evaluated from the cyclic stress-strain curve measured at 700 °C should be 1x10^{-3} [3]. However, due to high initial hardening rate of IN713LC superalloy the initial plastic strain amplitudes could was higher. The displacement rate of the grips was held constant 0.4 mm/min which corresponds to the average strain rate 3x10^{-3} s^{-1}. The grip displacement was monitored by an extensometer. During in-situ deformation SEM micrographs were taken using the four quadrant back-scatter-electron (BSE) and secondary-electron (SE) detectors. Systematic observations of selected locations in the central area of the specimen and also close to the grips were performed when the specimen was unloaded. The total number of cycles was 20 and the specimen was not fractured.

2.3 EBSD and AFM measuring

At the beginnig, during and in the end of LCF test orientation of the grains in the gauge section was evaluated using EBSD detector from OXFORD HKL NordlysMax. And the active slip systems were been identified. The detailed study of the surface topography and its evolution was performed using AFM directly on the metallic specimen surface. The sample topography was obtained by semicontact method. The SPM NT-MDT NTEGRA equipment and NSG10 AFM probes were used. Scanned area was 120x120 μm^2 and 20x20 μm^2. The AFM images were taken after the first cycle and after 20 cycles. After LCF test topography slip traces of the specimen subjected to 20 cycles was checked on cross-sections prepared by focused ion beam (FIB) in TESCAN FIB-FESEM microscope LYRA 3 XMU. The final FIB polishing step was performed at 30kV with probe current 56 pA.

3. RESULTS AND DISCUSSION

During in-situ cyclic loading the force (stress) and the distance between the grips (displacement) were recorded. The plastic strain amplitudes could be thus estimated. Appreciable stress relaxation both in tension and in compression could be observed under the condition of constant displacement rate. Plastic strain amplitudes were higher than in constant strain rate cycling. The plastic strain amplitude in the first cycle was 1.6×10^{-3}. Due to cyclic hardening it decreased and the 20-th cycle the plastic strain amplitude was only 7×10^{-4}. The characteristics surface relief in the central grain oriented for mainly single slip is shown in **Fig. 2a** after the first cycle and in **Fig. 2b** after 20 cycles. Already in the first tensile loading numerous unidirectional slip steps corresponding to the slip on the (111) primary slip planes were formed (see **Fig. 2a**). In the following compression half-cycle the existing slip steps did not disappear but new sharp slip steps (black lines) were observed in several locations. Already after the first cycle very few slip steps corresponding to the secondary slip systems were detected. after twenty cycles the relief is shown in **Fig. 2b.** The comparison with **Fig. 2a** reveals that new primary slip steps were formed and the existing slip steps were modified. The slip steps corresponding to the secondary slip system became more pronounced and new ones appeared. The activation of secondary slip system is due to small rotation of the grain which was measured by EBSD (see **Fig. 3**). This was documented on basic stereographic triangles on **Fig. 3** where is highlighted the position of stress axis (red points) of two central main grains which was shifted to the triangle edges. In this case the stress axis of grains on the triangle edges have the Schmidt factor of secondary slip systems the same as primary. From this reason the activation of the secondary slip systems is more possible [3]. Larger area of the specimen surface in the same location is shown in AFM images in **Fig. 4**. The surface relief after the first cycle (**Fig. 4a**) and that after the 20 cycles (**Fig. 4b**) do not differ substantially. Also the profiles shown in **Fig. 4c** indicate the that the basic surface relief was formed during the first cycle with the highest plastic strain amplitude and further 19 cycles with decreasing plastic strain amplitude introduced only very small changes.More details of the surface topography of the cycled specimen are shown in **Fig. 5**. In the cross section shown in **Fig. 5** the individual slip steps as well as elevations (extrusions) and depressions (intrusions) of the surface can be identified.

(a)

(b)

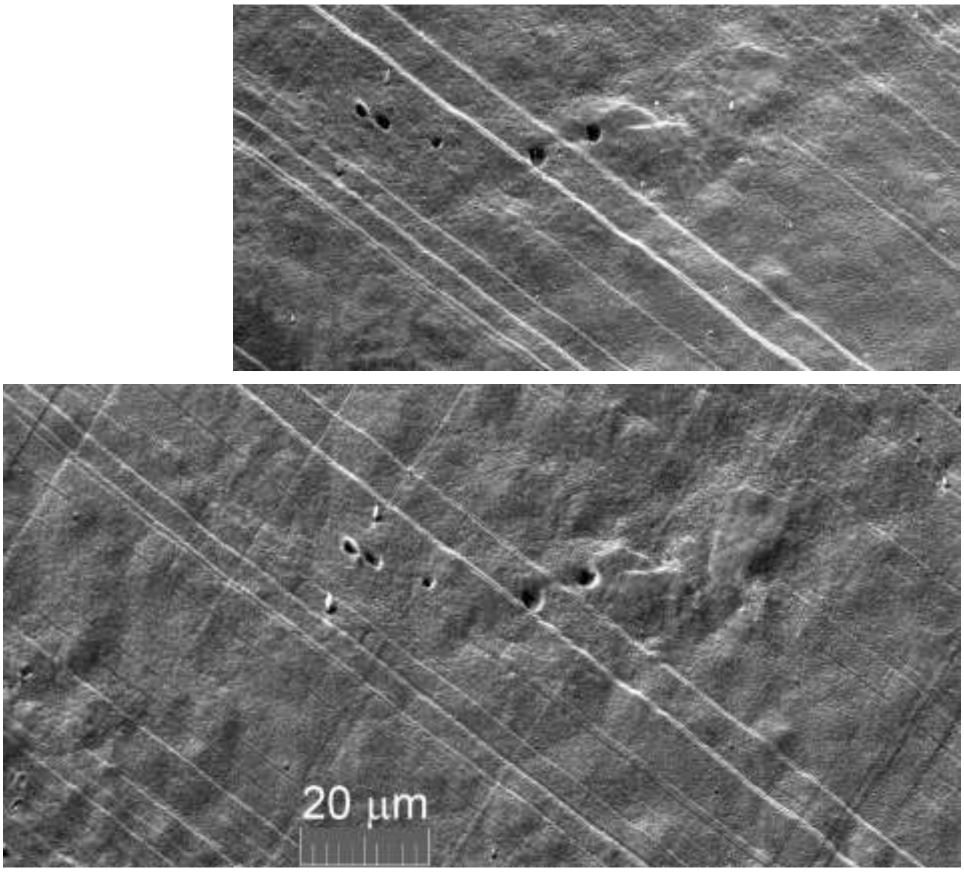

Fig. 2. Characteristics surface relief of the specimen of IN713LC cycled at 635 °C after (a) the first tensile half-cycle and unloading, (b) after 20 cycles. The stress axis is horizontal

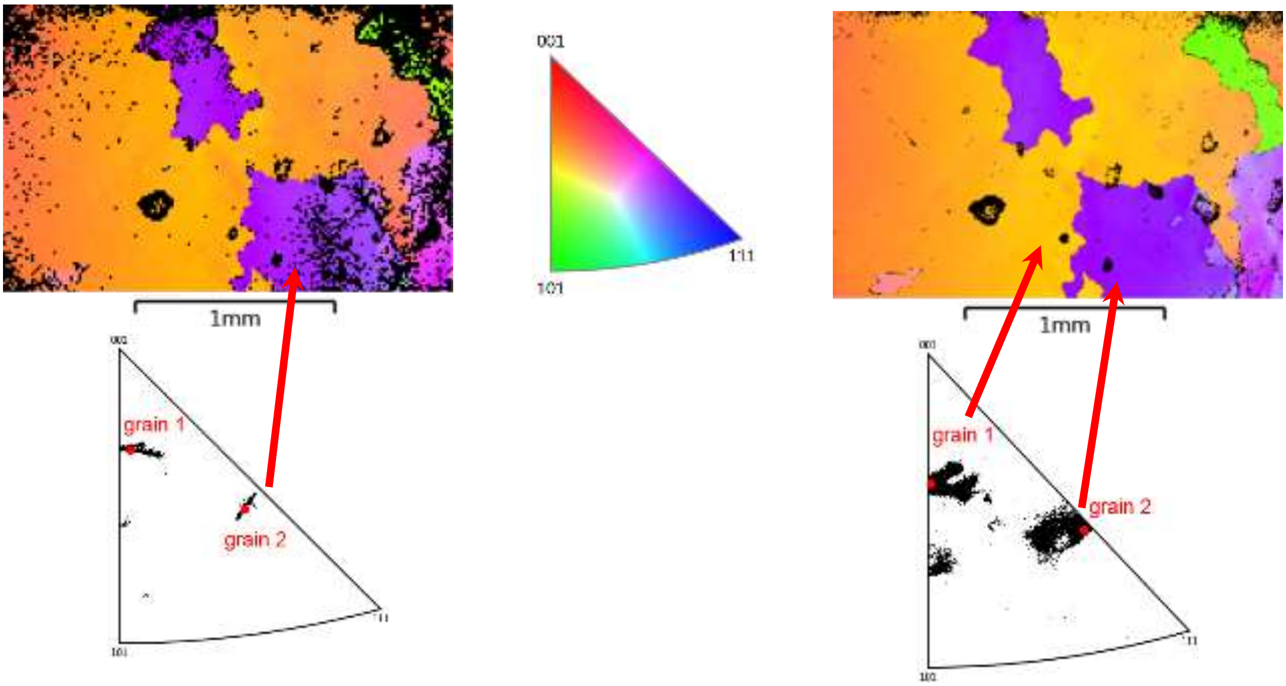

Fig. 3 Results of EBSD measuring before LCF test (left side) and after 20 cycles (right side). The IPF images (upper part) of mainly two grains is calculated for horizontal stress axis. The shift of stress axis of these two grains (red points) are showed in basic stereographic triangles (lower part)

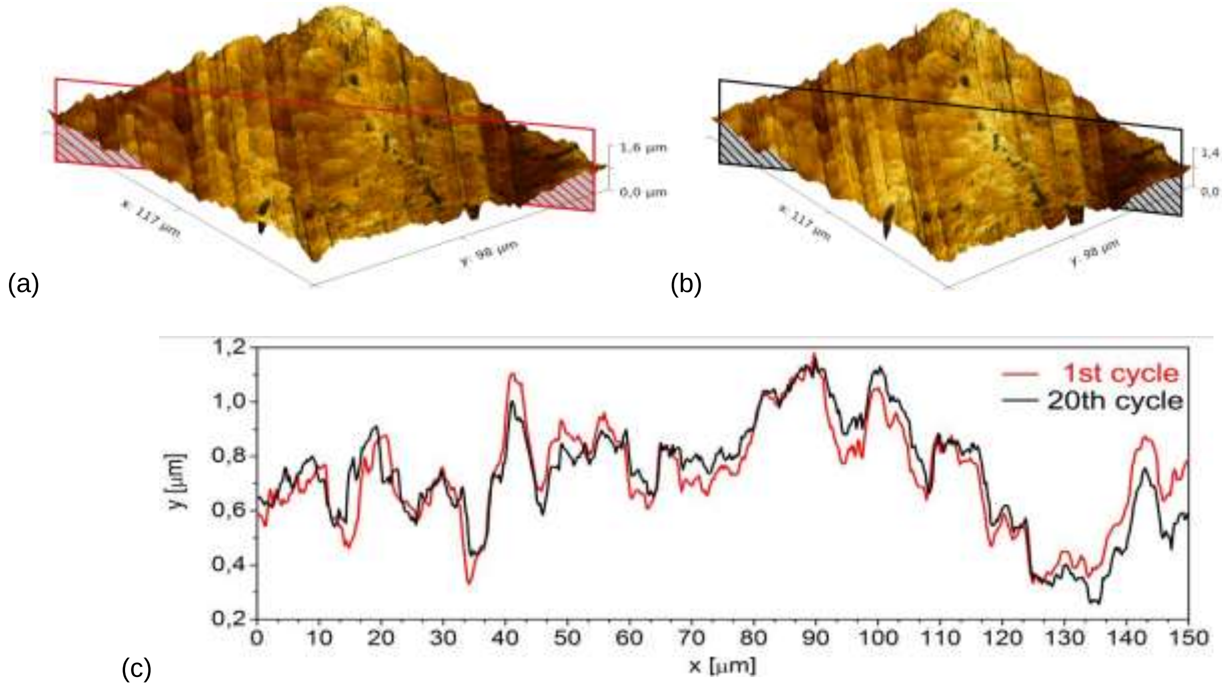

Fig. 4. Three-dimensional AFM image of surface area of the specimen of IN713LC cycled at 635 °C; (a) after the first cycle, (b) after 20 cycles, (c) the profiles along the same surface line both after the first cycle and after 20 cycles

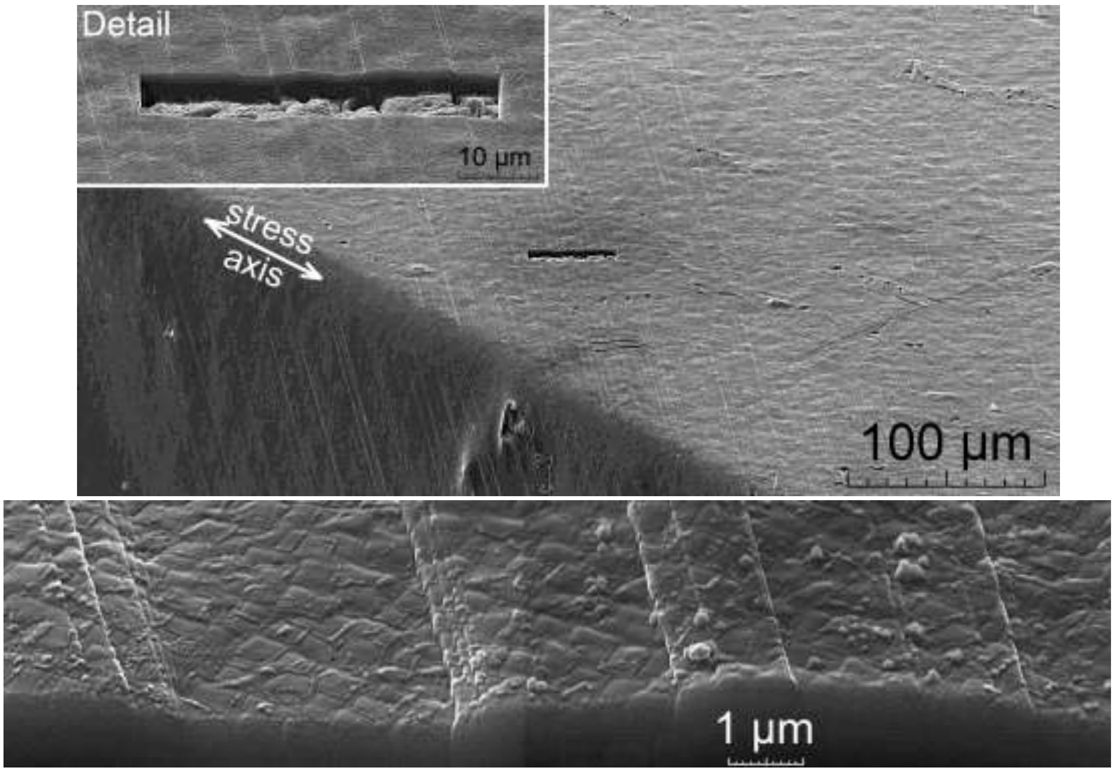

Fig. 5. Typical surface relief produced after 20 cycles (upper part) at 635 °C and detailed cross section (lower part) performed perpendicular to the PSMs

Twenty cycles with stress amplitude applied to the grains of IN713LC superalloy at temperature 635 °C produced early surface relief characterized by alternating elevations and depressions of the surface (see **Fig. 2b**, **Fig. 4** and **Fig. 5**). Since plastic strain amplitude was the highest in the first cycle the basic relief formed during the first cycle did not change substantially during following 19 cycles. Nevertheless, important small changes of the surface relief were identified in several locations and imaged in cross section and AFM profiles (**Fig. 4**). Surface relief shown in **Figs 2** and **5** correspond to the early development of the surface relief typical for cyclic loading.The observed surface relief indicate that unidirectional slip bands formed during the first cycle represent the locations into which cyclic slip during subsequent cyclic loading is localized. The reversibility of cyclic slip is very high (close to 100%) and in general, the appearance of the surface relief has not changed. Only a small but important irreversibility develops in the form of extrusions and intrusions in the surface at locations where PSBs emerge on the surface and where fatigue cracks initiate at larger number of loading cycles.

4. CONCLUSION

The cyclic straining of a specimen of IN713LC superalloy in SEM, in situ observations at 635 °C, AFM and FIB study of the surface relief and EBSD measuring led to the following conclusions:

- In cyclic loading with high stress amplitude the first cycle determines the locations of the localized cyclic slip.
- Both unidirectional and cyclic slip bands intersect the matrix and the precipitates along the primary slip plane (111).
- Rough surface relief produced in the first cycle changes only slightly but the nuclei of PSMs (extrusions and intrusions) are formed already during 20 cycles.
- Rotation of grains during cyclic loading cause the activation of the secondary slip systems along slip planes type {111}.

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