Extrusion and intrusion evolution in cyclically strained cast superalloy Inconel 738LC using confocal laser scanning microscope and AFM

K Obrtlík, M Juliš, J Man, T Podrábský and J Polák

1 Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Žižkova 22, 616 62 Brno, Czech Republic
2 Institute of Material Science and Engineering, Faculty of Mechanical Engineering, BUT, Technická 2, 616 69 Brno, Czech Republic

Abstract. Surface relief within persistent slip markings (PSMs) was studied using confocal scanning laser microscopy (CSLM) and atomic force microscopy (AFM) in cast nickel base superalloy Inconel 738LC cyclically strained in strain control at room temperature. Extrusion and intrusion topography and kinetics are documented. The dependence of extrusion height on the number of cycles is obtained. Two regimes of extrusion growth are identified. Average intrusion growth rate is assessed.

1. Introduction
The extrusion and intrusion formation within persistent slip markings (PSMs) of crystalline materials represents the first surface indication of fatigue damage in a material specimen or in a component [1]. Since fatigue cracks nucleate within the PSMs, experimental data on the topography of extrusions and intrusions and the kinetics of their evolution can reveal the details of fatigue damage mechanisms.

Surface relief within PSMs and its connection with crack initiation was studied extensively in a number of materials (see review [2]). Though fatigue cracks originate from intrusions, much more direct quantitative information has been obtained on extrusions. Quantitative data on intrusions have been acquired particularly using sectioning and plastic replica techniques. The intrusion growth rate is reported higher than that of extrusions [3].

Quantitative data on the surface relief within PSMs in nickel-base superalloys, which are designed for critical parts of gas turbines, are sporadic [4,5]. An extrusion height is reported [4,5] and limited data on extrusion growth rate can be inferred from [4]. Therefore, the present paper deals with the quantitative observation of surface relief evolution with focus to extrusions and intrusions kinetics in Inconel 738LC (In 738LC) cyclically strained under total strain control at room temperature using two advanced techniques, namely confocal scanning laser microscopy (CSLM) and atomic force microscopy (AFM).

2. Experimental
Inconel 738LC polycrystals were supplied by PSB Velká Bítěš, a.s. as conventionally cast rods in fully heat treated condition, i.e. after 1120 °C / 4 h / air cooling (AC) + 845 °C / 24 h / AC. Chemical
composition and the details of microstructure are shown elsewhere [6]. The average grain size, found using the linear intercept method, was 3.6 mm.

Cylindrical button-end specimens had gauge length and diameter of 15 mm and 6 mm, respectively. They were fatigued in a symmetrical push-pull cycle in strain control at room temperature. The strain was measured and controlled using a sensitive extensometer with a 12 mm base. The strain rate of $2 \times 10^{-3} \text{s}^{-1}$ and total strain amplitude of $\varepsilon_a = 0.55\%$ were kept constant.

The surface relief was studied in a CSLM (Olympus LEXT OLS3000) and in an AFM (Acurex III, Topometrix). The maximum lateral resolution of the CSLM is 120 nm and the resolution in the direction of the beam is 5 nm (for the detailed description of the technique, see [6,7]). The CSLM software can provide the surface relief profiles. The AFM in contact imaging mode in the air was used to obtain constant-force topographic images. Further details has been reported elsewhere [3,6].

3. Results

To study the surface relief evolution the CSLM inspection of the specimen surface was performed at the following numbers of cycles: $N = 30, 60, 100, 150, 200, 300$. $N = 300$ corresponds approximately to 50% of the fatigue life $N_f$. The AFM was used to observe both the specimen surface and the plastic replica at $N = 300$ cycles.

An example of AFM micrograph of the metallic surface is shown in figure 1. Three parallel PSMs consisting of extrusions and intrusions can be seen. The extrusion height varies considerably along a PSM. There are even PSM segments where extrusions were not distinguished. These locations are indicated in with small black arrows in figure 1. Surface profiles in the section perpendicular to the specimen surface (denoted AB in Figure 1) are shown in figure 2. In the direction from the left to the right moderate and deep intrusions marked with the arrows and followed by extrusions are shown in figure 2a (CSLM technique). The typical surface relief obtained in the section AB with the AFM is shown in figure 2b. Shallow intrusions and clear extrusions can be seen in individual PSMs.

Figure 3 shows the growth kinetics of a hilly extrusion. The specimen was removed from the testing machine at preselected number of cycles and the extrusion height was measured using the CSLM. The extrusion height vs. number of cycles can be approximated with the linear dependence and the extrusion growth rate of 0.29 nm/cycle is obtained. The results presented in figure 3 confirm

\begin{figure}[h]
\centering
\includegraphics[width=0.45\textwidth]{figure1.png}
\caption{Three-dimensional AFM micrograph of surface relief. $N = 300, \varepsilon_a = 0.55\%$.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.45\textwidth]{figure2.png}
\caption{Surface relief profiles in the section perpendicular to the specimen surface (denoted AB in Figure 1). (a) CSLM, (b) AFM.}
\end{figure}
quantitatively that slip bands in the In 738 LC superalloy are persistent.

Three-dimensional AFM micrograph of surface relief using plastic replica is shown in figure 4 in non-inverted format. Several PSMs with intrusions of various depths are apparent. The deepest intrusion can be found in the PSM located in the center of the image. Its maximum depth is 0.6 μm. The intrusion morphology and occurrence are in agreement with those found using the CSLM technique (compare figure 5).

Figure 5 shows inverted CSLM micrographs (i.e. intrusions appear as thin projections protruding from PSMs) in three-dimensional representation taken in the identical part of a metallic specimen at different numbers of cycles. At the number of cycles $N = 23 \% N_f$, intrusions are not present continuously within the PSMs but there are locations where intrusions were not distinguished (see figure 5a). The intrusion morphology varies along a PSM. Thin almost ribbon-like or completely

![Figure 3](image1.png)

**Figure 3.** Extrusion height vs. number of cycles in cycling with constant total strain amplitude $\varepsilon_a = 0.55 \%$.

![Figure 4](image2.png)

**Figure 4.** Surface relief obtained by AFM using plastic replica. $N = 300$ cycles (0.5 $N_f$).

![Figure 5](image3.png)

**Figure 5.** CSLM micrograph of surface relief in metallic specimen showing intrusion evolution (inverted image). (a) $N = 140$ cycles, (b) $N = 300$ cycles (0.5 $N_f$).
isolated peak-like intrusions can be observed. Figure 5 shows that the amount of intrusions increases with increasing number of cycles – see the location marked with black arrows in figure 5b. The average intrusion depth was measured in selected sections. It increases from 0.30 μm at N = 140 cycles to 0.61 μm at N = 300 cycles. These data allow assessing intrusion growth rate to 1.9 nm/cycle.

4. Discussion
Detailed quantitative study of the PSM topography and its evolution using the high resolution CSLM and AFM techniques revealed characteristic features of the surface relief of nickel-based In 738LC superalloy. The topography of PSMs comprises extrusions and intrusions analogous to those in simple FCC and BCC metals and alloys [1]. The dependence of extrusion height vs. number of cycles can be divided into two regimes. In the incipient regime up to about 5 % Nf the assessed extrusion growth rate is approximately one order of magnitude higher than the constant extrusion growth rate in the interval between 5 to 50 % Nf. The present data are in good agreement with the results obtained previously [3,8]. In the second regime, the extrusion growth rate was reported 0.2 to 0.4 nm/cycle for polycrystalline copper [8] and 0.35 nm/cycle for 316L steel [3]. Extrusion growth rate is reported 1.52 nm/cycle [3], which is higher than that of extrusions in agreement with the present results. However, the intrusion growth rate can be influenced by probable crack initiation at the tip of an intrusion.

Present results show that both the extrusion height and the intrusion depth vary significantly along PSMs after 140 or 300 cycles elapsed – see figures 1, 4 and 5. Therefore, local activity of a PSB leading to the surface relief formation within the corresponding PSM changes during cycling.

5. Conclusions
The detailed study of the evolution and topography of extrusions and intrusions within PSMs of cyclically strained Inconel 738LC using CSLM and AFM leads to the following conclusions:
(i) Slip markings consisting of extrusions and intrusions that appear early in the fatigue life are persistent.
(ii) Two regimes of extrusion growth are identified. Short initial regime is followed by constant growth rate period.
(iii) PSB activity leading to extrusion and intrusion formation varies locally during cycling.
(iv) CSLM proves to be a suitable technique for the direct observation of intrusion topography and crack initiation.

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