Surface Relief Evolution in 316L Steel Fatigued at Depressed and Elevated Temperatures

J. Man[1]*, M. Petrenec[1], P. Klapetek[2], K. Obrtlík[1], J. Polák[1]
[1]*Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic;* [2]*Czech Metrology Institute, Brno, Czech Republic*

Abstract

Surface relief of austenitic 316L steel cycled with constant plastic strain amplitude at depressed and elevated temperature was studied using scanning electron microscopy and atomic force microscopy. The formation of PSMs and their evolution during cycling was followed. PSMs consist primarily of extrusions accompanied later by parallel intrusions. The kinetics of the early intrusion growth was assessed. At the lowest temperatures only static extrusions and intrusions were found. The findings were discussed in relation to point defect models of fatigue crack initiation

Keywords: 316L steel, fatigue, persistent slip marking (PSM), extrusion, intrusion, atomic force microscopy (AFM)

1. Introduction

The localization of the cyclic plastic strain in persistent slip bands (PSBs) followed by the formation of pronounced surface relief (nowadays called persistent slip markings (PSMs) [1]) represents a general and very important feature of cyclic straining in crystalline materials. PSMs arise in areas where PSB lamellae intersect the original flat surface. They consist of local elevations and depressions of the surface known as surface extrusions and intrusions, respectively [1, 2]. Although it is generally accepted that PSMs represent incipient fatigue crack sites, the exact mechanism of fatigue crack nucleation has not been yet clarified completely [3]. Since some models predict the temperature dependence of surface relief evolution [4, 5], for their verification it is desirable to obtain detail experimental data on PSM formation just at depressed and elevated temperatures.

The surface relief topography and its evolution have been amply studied in room temperature cycling [1, 2]. Experimental data for depressed and elevated temperatures are rare so far. Presence of extrusions and intrusions down to 4.2 K was firstly documented by Cottrell and Hull [6] and Hull [7]. Fatigue crack initiation at 298, 77 and 4.2 K was studied by Kwon et al. both for single- [8] and polycrystalline [9] copper. Basinski and Basinski using the sharp corner polishing technique revealed three characteristic morphologies of macro-PSMs in copper single crystals fatigued in the temperature interval 4.2–350 K [10]. Morphology

* Corresponding author: man@ipm.cz

and growth of extrusions in single- [11] and polycrystalline [11, 12] copper at 107, 298 and 473 K was studied by Mughrabi and his co-workers. Although atomic force microscopy (AFM) has been successfully employed for obtaining detail information on surface relief formation during room temperature cycling (for recent review see [13]), systematic study of surface relief in specimens cycled at depressed and elevated temperatures by this high-resolution technique has not been performed so far.

The aim of the present paper is to present the first results of the study of surface relief formation in individual grains of polycrystalline 316L steel cyclically strained at depressed and elevated temperatures using AFM and scanning electron microscopy (SEM). Qualitative and quantitative data on the morphology of PSM and extrusion growth are reported and discussed in relation with recent models of surface relief formation.

2. Experimental details

Austenitic 316L stainless steel was supplied by Uddeholm (Sweden) in the form of a 25 mm thick plate with the following chemical composition (in wt. %): 0.018 C, 0.42 Si, 1.68 Mn, 0.015 P, 0.001 S, 17.6 Cr, 13.8 Ni, 2.6 Mo, rest Fe. Average grain size, found using the linear intercept method, was 39 µm.

Two specimen geometries were used in the study. Cylindrical specimens with threaded ends having gauge diameter and length of 8 and 12 mm, respectively, were adopted for depressed temperatures. Cylindrical button-end specimens of 6 mm in diameter and 15 mm in gage length were used for fatigue tests at elevated temperatures. To facilitate the surface relief observation a shallow notch was produced in both specimen geometries by grinding a cylindrical surface 60 mm in diameter with the axis perpendicular to the specimen axis to a depth of 0.4 mm. After machining the specimens were annealed at 600 °C for 1h in vacuum and the notch area was polished mechanically and electrochemically. For easier orientation on the specimen surface fine circular marks 400 µm in diameter were engraved on the central part of the polished notch.

The fatigue tests at 100, 180 and 573K were carried out in a MTS computer-controlled servohydraulic testing machine in a symmetrical push-pull cycle in strain control. Plastic strain amplitude $\varepsilon_{ap} = 1\times10^{-3}$, derived from the half-width of the hysteresis loop, and the total strain rate $\dot{\varepsilon} = 1.5\times10^{-3}$ s^{-1} were kept constant during the tests at all temperatures. Details concerning the cryostat and temperature control in low-temperature tests are given elsewhere [14]. Specimens were cycled either to different stages of fatigue life (at 180 K and 573 K) or with interruptions and warming up to room temperature for surface relief study (at 100 K). Deformation history of all specimens is listed in table 1.

The detailed study of surface topography and its evolution on the specimen surface was performed using AFM (Accurex IIL, Topometrix) and SEM (JEOL

Table 1. Fatigue test conditions and PSM characteristics

Specimen No.	Temperature (K)	Cycles	PSM spacing (μm)	Extrusion height	
				Average	Maximum
UD371	180	500	4.8±1.1	32±30	140–170
UD372	180	1000	4.5±1.4	72±57	280–320
UD373	180	2000	4.6±0.9	115±64	280–320
UD374	180	4500	4.2±0.6	168±83	420–470
UD380	100	500, 1000, 2000	—	—	—
UD424	573	100	5.6±1.5	71±35	180–230

JSM-6460). AFM in contact imaging mode in air was used to obtain constant-force topographic images. A V-shaped silicon nitride cantilever with a sharpened pyramidal tip having the radius of curvature of 20 nm and the vertex angle 36° was applied.

3. Results

3.1 Surface relief at depressed temperature

Figure 1a and 1b show at low magnification typical surface relief of the specimens cycled at 180 K for 500 and 4 500 cycles. Already after 500 cycles PMSs are present in about 15% of grains. Fine parallel PSMs cover numerous grains (see Fig. 1a) With increasing number of cycles PSMs intensify considerably (see Fig. 1b). Whereas the PSM spacing was approximately constant at different stages of fatigue life (see Table 1), the fraction of grains with PSMs grow gradually and at 4500 cycles more than 75% of grains were covered by PSMs. Cyclic plastic strain is generally accommodated in grains by producing PSBs of one slip system at different stages of fatigue life. Only at 4500 cycles secondary slip system was activated in some grains in the close proximity of grain boundaries.

Details of PSM morphology produced by cycling at 180K as revealed by SEM at high magnification show Fig. 1c and 1d for different number of cycles. It is apparent from both images that individual PSMs consist of extrusions accompanied by intrusions. After 500 cycles intrusions are developed often only locally along ribbon-like extrusions (see Fig. 1c), however clear intrusions accompanying each extrusion are apparent from Fig. 1d. From the inclination of extrusions with respect to the specimen surface it can be deduced from both images that intrusions were detected by SEM at the side of extrusions where the emerging active slip plane is inclined to the surface at an obtuse angle (side denoted *A* in Fig. 11 in [4]).

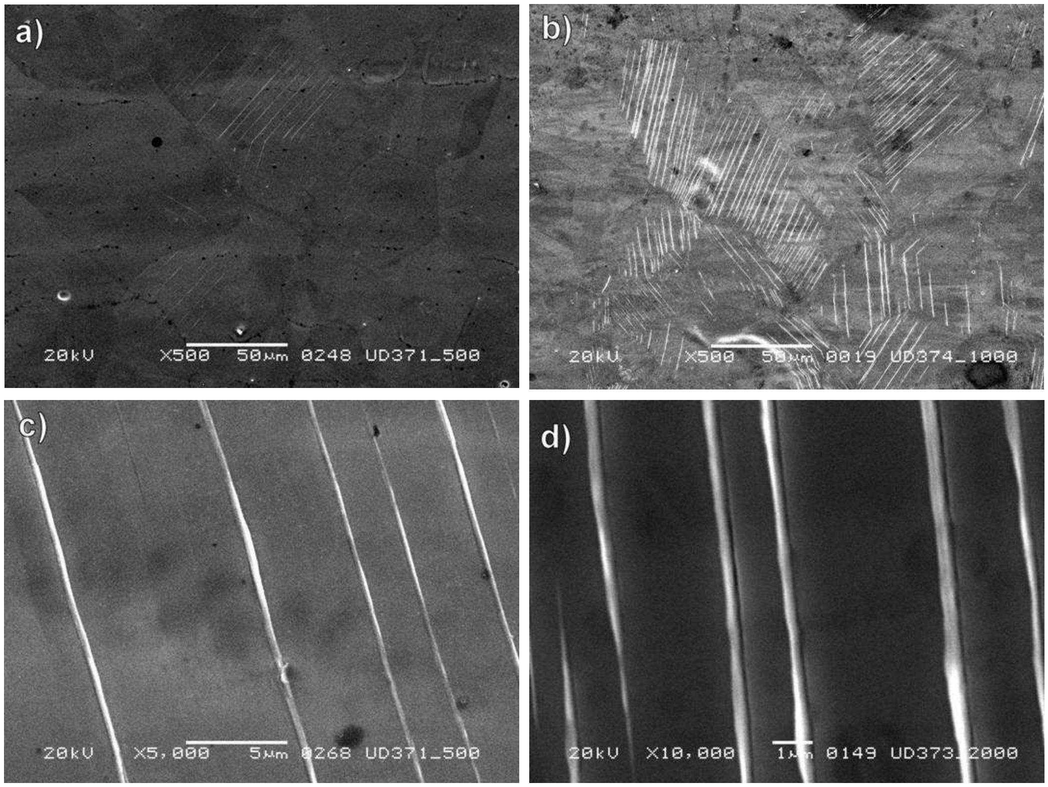

Fig. 1 SEM micrographs of surface relief in 316L steel fatigued with $\varepsilon_{ap}=1\times10^{-3}$ at 180 K to different number of cycles. (a), (c) specimen UD371, N = 500 cycles; (b) specimen UD374, N = 4 500 cycles; (d) specimen UD373, N = 2 000 cycles. Stress axis is horizontal in all SEM micrographs.

Three dimensional AFM image of the PSMs with finer details of PSM topography shows Fig. 2. The presence of intrusion accompanying the extrusion is apparent.

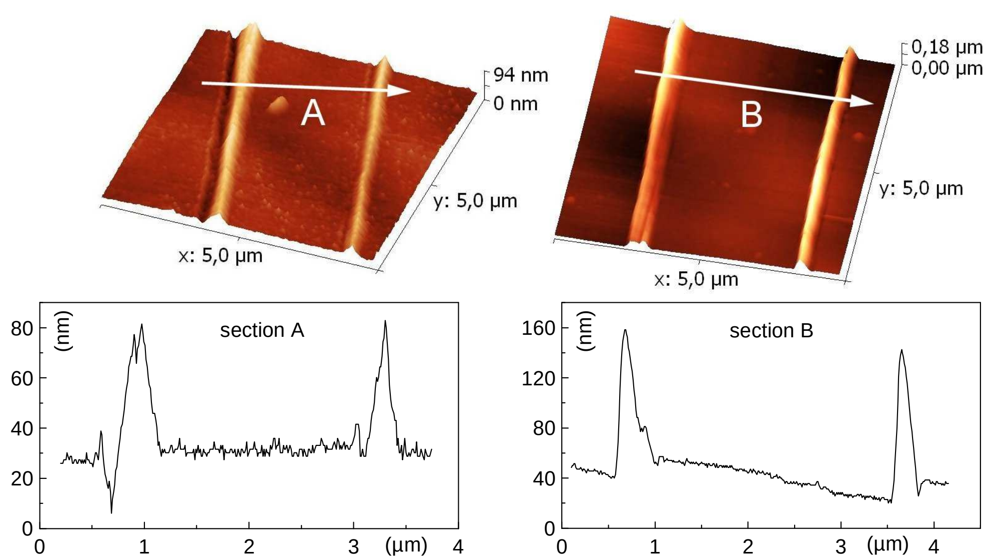

Fig. 2 Three-dimensional AFM images of details of extrusions and intrusions and corresponding profiles in 316L steel fatigued at 180 K for 500 cycles.

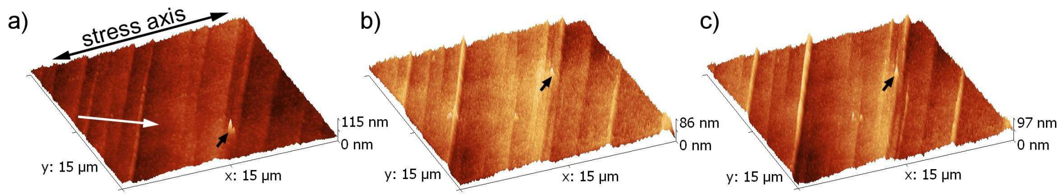

Fig. 3 Three-dimensional AFM micrographs of the surface relief within a grain of 316L steel fatigued with $\varepsilon_{ap}=1\times10^{-3}$ at 100 K for (a) 500, (b) 1 000 and (c) 2 000 cycles.

Nevertheless, the volume of the material pushed up to the extrusion is higher than that impressed in the intrusion. Also the width of the extrusion is considerably larger than that of an accompanying intrusion.

Two characteristic features of surface relief topography were revealed by AFM in 316L steel fatigued at 100 K. Fig. 3 shows AFM micrographs taken from the specimen surface at different number of cycles. The identical position on all micrographs is indicated by a small black arrow. PSMs consisting of extrusions and fine slip markings corresponding to fine slip steps during unidirectional deformation can be distinguished from this figure. The extrusions are very thin and their height at 500 cycles is typically only 10–15 nm (see Fig. 3a). With continuing cycling they grow. At 2 000 cycles their height increased to 25–35 nm (see Fig. 3c).

In addition to the above slow growth of extrusions observed in majority of grains investigated, another surface feature was revealed by AFM in several grains of 316L fatigued at 100K – see Fig. 4. Fig. 4a shows overview of a grain taken at 500 cycles and Fig. 4b and 4c represent three-dimensional details taken from the identical area indicated in Fig. 4a after 500 and 2 000 cycles, respectively. It is

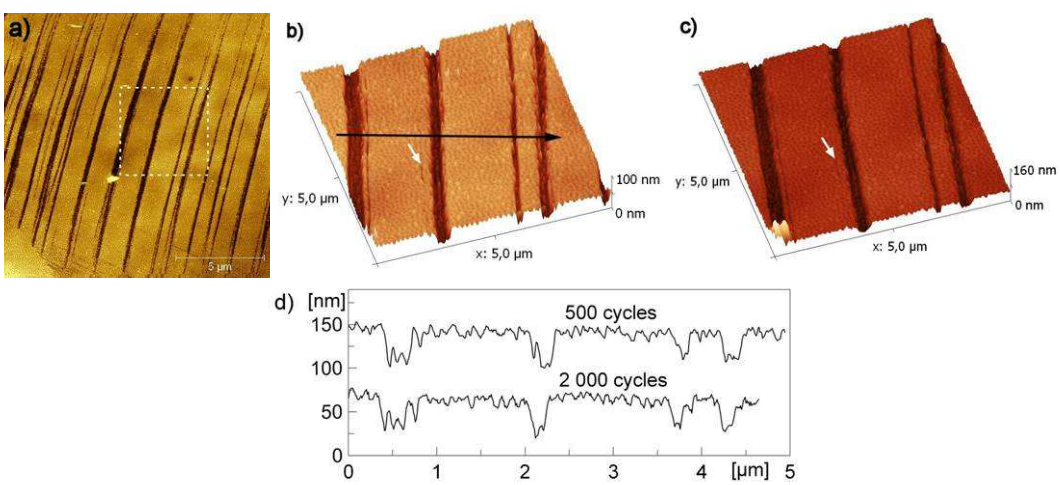

Fig. 4 Surface relief in a grain of 316L steel fatigued with $\varepsilon_{ap}=1\times10^{-3}$ at 100 K, AFM. (a) N = 500 cycles, low magnification micrograph, stress axis is horizontal. Details of the area marked in (a) by a rectangle imaged in three dimensions: (b) N = 500 cycles, (c) N = 2000 cycles, (d) Profiles of “static” intrusions in the cross-section marked in (b).

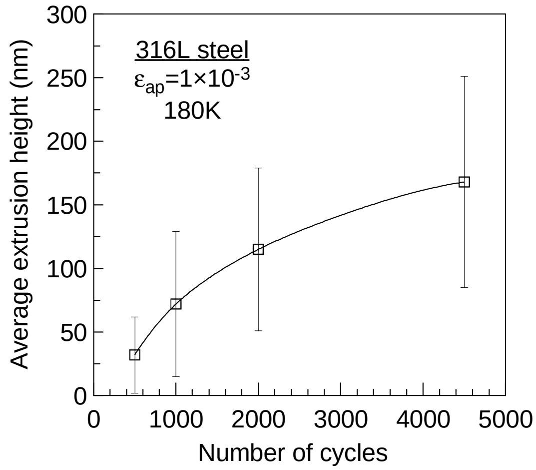

Fig. 5 Average extrusion height vs. number of cycles in 316L fatigued at 180 K.

clearly seen from these images that PSMs consist purely of intrusions which are not accompanied by extrusions. PSMs of different width are evenly distributed across a grain (see Fig. 4a). The maximum depth of intrusions was about 40 nm. Profiles of intrusions in Fig. 4d, obtained from the cross-section indicated in Fig. 4b by a black arrow, indicate no observable changes in the intrusion depth during cycling.

Quantitative data on the growth of extrusions were obtained from AFM micrograph taken on the specimen surface from the profiles of individual PSMs in selected sections. In agreement with our previous studies on surface relief and its evolution in 316L [15, 16] the sections were taken perpendicular both to the surface and to the direction of the PSM on the surface. The height of an extrusion was evaluated in three different sections. The height of each extrusion represents the average of these three values. About 10 grains and more than 100 PSMs were evaluated for specimens cycled at 180 K to different stages of fatigue life. The results of statistical treatment of experimental data obtained are listed in Table 1 (the average and the maximum extrusion height). The kinetics of extrusion growth at 180 K is presented in Fig. 5. In spite of a great scatter of experimental data systematic extrusion growth with the number of cycles is apparent. The extrusion growth rate is the highest during the initial period and then decreases with the number of cycles (see Fig. 5). The initial rate of the extrusion growth is approximately 7×10^{-11} m/cycle.

3.2 Surface relief at elevated temperature

After 100 cycles at 574 K PSMs were found only in a few grains. Characteristic features of surface relief topography at elevated temperature documented by SEM and AFM shows Fig. 6. PSMs do not cover evenly the whole surface of the grain. They were typically developed locally along the intersection of PSBs with the specimen surface (see Fig. 6a). Average PSM spacing is slightly higher in comparison with 180 K (see Table 1). Details of PSMs obtained by AFM from the

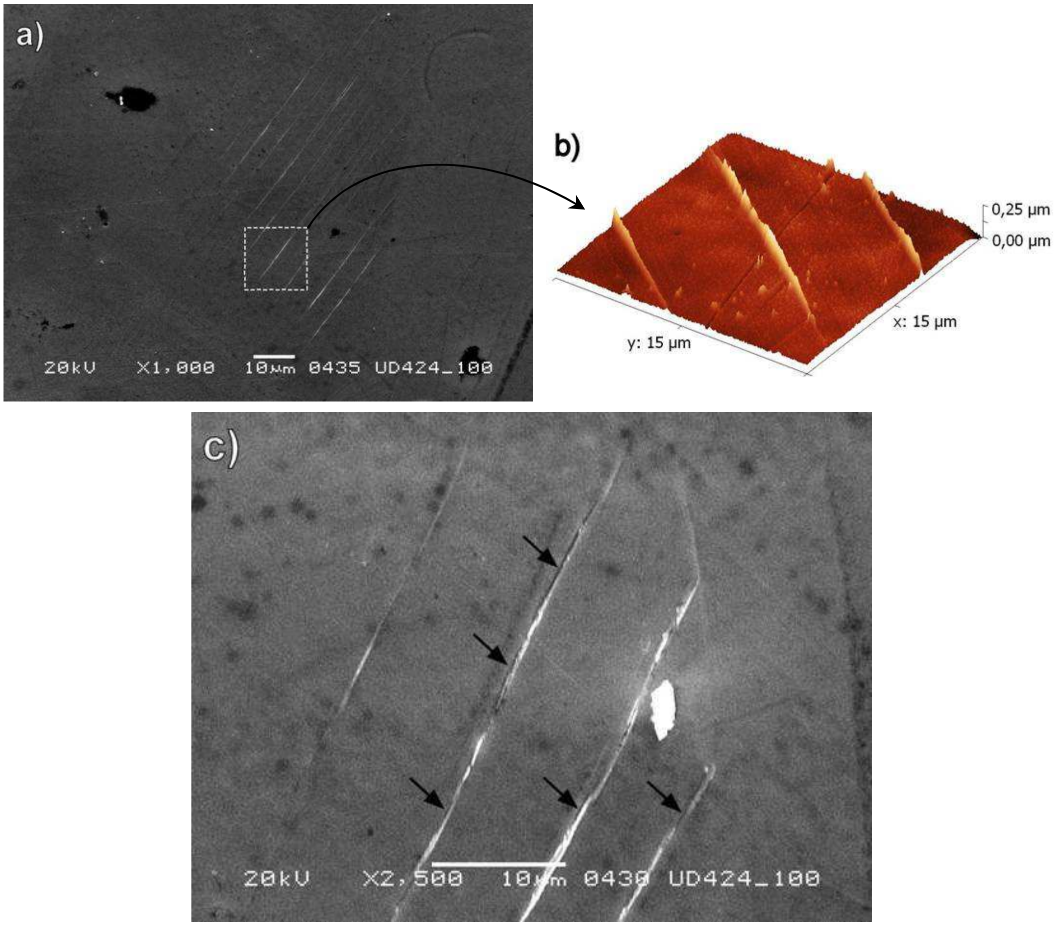

Fig. 6 Surface relief in 316L steel fatigued with $\varepsilon_{ap}=1\times10^{-3}$ at 573 K for 100 cycles. (a) Overview of a grain, SEM; (b) Detail of PSMs obtained by AFM in area denoted in (a); (c) Detail of individual PSMs, SEM. Stress axis is horizontal in (a) and (c).

area denoted in Fig. 6a shows Fig. 6b. PSMs consist of ribbon-like extrusions the height of which slightly fluctuates along their length. The average and the maximum extrusion height evaluated from several grains by AFM is listed in Table 1. Due to limited number of observations of specimens cycled at 574 K intrusions were not detected by AFM so far, nevertheless SEM micrograph (see Fig. 6c) reveals that parallel intrusions (indicated by black arrows) accompanying locally extrusions are already present.

4. Discussion

Experimental observations of the surface relief produced by plastic strain controlled low amplitude cyclic loading at depressed and elevated temperatures allow completing recent observations at room temperature. The surface relief at depressed temperatures is characterized by PSM consisting initially of extrusions. Parallel to the extrusions intrusions start to develop. The evolution of surface relief is similar at 573 K. Contrary to room temperature cycling at the lowest temperature (100 K) static extrusions were produced whose depth did not increase with further cycling.

Systematic growth of extrusion height was observed in cycling at temperature 180 K. In agreement with previous results at room temperature [16] the initial extrusion growth rate is high and decreases later. The data on extrusion height has high scatter that increases with increasing number of cycles. Several factors can contribute to this scatter as different grain size in the direction of active Burgers vector [15], different local plastic strain amplitude within PSBs [17], and in particular the inclusion of all PSMs into the evaluation of the average height, independent of the age of PSMs. The extrusions were produced also in specimens cycled at 100K but the extrusion growth practically stopped.

The finding on the shape of PSMs consisting of extrusions and developing into extrusion/intrusion pairs are compatible with the point defect models of surface relief formation and crack initiation [4,5,18]. The continuous growth of extrusions and production of intrusions at temperature 180 K indicates that some point defects are still mobile at this temperature. Since extrusion is produced at the emerging PSB and intrusion at the interface with the matrix, the mobile defects are vacancy-type defects. These defects are apparently immobile at the lowest temperature 100 K since only static extrusions are produced.

The presence of intrusions at temperature 100 K indicates the production of interstitial-type defects. Since no evolution of the depth of intrusions was observed the responsible point defects are also immobile at the 100 K and the static intrusions are produced.

5. Conclusions

The AFM and SEM study of the surface relief and its evolution in 316L steel cyclically strained with $\varepsilon_{ap} = 1\times10^{-3}$ at depressed and elevated temperatures leads to the following conclusions:

(1) General character of the surface relief of 316L steel produced by plastic strain controlled cyclic loading at depressed and elevated temperature is only slightly modified provided the temperature is not too low or too high. PSMs consisting of growing extrusions accompanied by parallel intrusions were found. Early kinetics of extrusion growth is characterized by rapid initial growth which later decreases.
(2) Cyclic straining at the lowest temperature resulted in appreciable modification of the surface relief. Static extrusions and static intrusions are present in different areas of the cycled specimens.
(3) Experimental findings are compatible with the point defects models of surface relief formation and crack initiation. Presence of static extrusions and intrusions indicate difficult mobility of respective point defects at the temperature close to liquid nitrogen temperature.

Acknowledgement

The financial support of the present work by the grants No. 106/06/1096 and No. 101/07/1500 of the Grant Agency of the Czech Republic and by the research project No. AV0Z 2041057 and grant No. 1QS200410502 of the Academy of Sciences of the Czech Republic is acknowledged.

References

[1] J. Polák, Cyclic deformation, crack initiation, and low-cycle fatigue, in Comprehensive Structural Integrity, Vol. 4, I. Milne, R.O. Ritchie and B. Karihallo (eds.), Elsevier, Amsterdam, 2003, pp. 1–39.
[2] S. Suresh, Fatigue of Materials, 2nd ed., Cambridge University Press, Cambridge, 1998.
[3] J. Man, K. Obrtlík and J. Polák, Extrusions and intrusions in fatigued metals. Part 1. State of the art and history, Phil. Mag., submitted for publication.
[4] U. Essmann, U. Gösele and H. Mughrabi, A model of extrusions and intrusions in fatigued metals. I. Point-defect production and the growth of extrusions, Phil. Mag. A 44 (1981) 405–426.
[5] J. Polák and M. Sauzay, Growth of extrusions in localized cyclic straining, Mater. Sci. Eng. A 500 (2009) 122–129.
[6] A.H. Cottrell and D. Hull, Extrusion and intrusion by cyclic slip in copper, Proc. Roy. Soc. A 242 (1957) 211–213.
[7] D. Hull, Surface structure of slip bands on copper fatigued at 293°, 90°, 20° and 4.2° K, J. Inst. Metals 86 (1957–58) 425–430.
[8] I.B. Kwon, M.E. Fine and J. Weertman, Fatigue damage in copper single crystals at room and cryogenic temperatures, Acta metall. 37 (1989) 2937–2946.
[9] I.B. Kwon, M.E. Fine and J. Weertman, Microstructural studies on the initiation and growth of small fatigue cracks at 298, 77 and 4.2 K in polycrystalline copper, Acta metall. 37 (1989) 2927–2936.
[10] Z.S. Basinski and S.J. Basinski, Copper single crystal PSB morphology between 4.2 and 350 K, Acta metall. 37 (1989) 3263–3273.
[11] H. Mughrabi, M. Bayerlein and R. Wang, Direct measurement of the rate of extrusion growth in fatigued copper mono- and polycrystals, in: Proc. of the Ninth Int. Conf. on Strength of Metals and Alloys (ICSMA 9), Vol. 2, D.G. Brandon, R. Chaim and A. Rosen (eds.), Freund Publ. Comp., London, 1991, pp. 879–886.
[12] M. Bayerlein and H. Mughrabi, The formation of either tongue- or ribbon-like extrusions in fatigued copper polycrystals, Acta metall. mater. 39 (1991) 1645–1650.
[13] J. Man, P. Klapetek, O. Man, A. Weidner, K. Obrtlík and J. Polák, Extrusions and intrusions in fatigued metals. Part 2. AFM and EBSD study of the early growth of extrusions and intrusions in 316L steel fatigued at room temperature, Phil. Mag., submitted for publication.
[14] J. Polák, J. Man, M. Petrenec, T. Kruml and K. Obrtlík, Effect of depressed temperature on the internal and effective stresses in austenitic stainless 316L steel,

in: Proc. Sixth Int. Conf. on Low Cycle Fatigue (LCF 6), P.D. Portella, T. Beck and M. Okazaki (eds.), DVM, Berlin, 2008, pp. 27–32.
[15] J. Man, K. Obrtlík, C. Blochwitz and J. Polák, Atomic force microscopy of surface relief of individual grains of fatigued 316L austenitic stainless steel, Acta Mater. 50 (2002) 3767–3780.
[16] J. Man, K. Obrtlík and J. Polák, Study of surface relief evolution in fatigued 316L austenitic stainless steel by AFM, Mater. Sci. Eng. A 351 (2003) 123–132.
[17] A. Weidner, J. Man, W. Tirschler, P. Klapetek, C. Blochwitz, J. Polák and W. Skrotzki, Half-cycle slip activity of persistent slip bands at different stages of fatigue life of polycrystalline nickel, Mater. Sci. Eng. A 492 (2008) 118–127.
[18] J. Polák, On the role of point defects in fatigue crack initiation. Mater. Sci. Eng. 92 (1987) 71–80.