CHANGES IN DISLOCATION SUBSTRUCTURE OF S235JR STEEL DURING FATIGUE LOADING

Dagmar JANDOVÁ[a], Ľubomír GAJDOŠ[b], Martin ŠPERL[b], Jaroslav KAISER[c]

[a] *VZÚ Plzeň s.r.o., Pilsen, Czech Republic, EU*
[b] *ÚTAM AV ČR Praha, Prague, Czech Republic, EU*
[c] *ZČU v Plzni, Pilsen, Czech Republic, EU*

Abstract

Changes in dislocation substructure were studied in normalized and annealed S235JR steel in relation to the number of symmetrical reversed stress cycles with the amplitude σ_a = 242 MPa and corresponding mean life N_f = 17,950 cycles. The microstructure of the steel consisted of ferrite with a small amount of pearlite. The substructure was observed in as received condition and after application of various relative numbers of cycles n/N_f, these being 0.25, 0.50 and 0.75. An irregular dislocation net occurred in the virgin specimen, however, during cyclic loading the dislocations started to accumulate gradually in slip bands and to form a cell substructure in grains of favourable crystallographic orientations. Total dislocation density, dislocation density in slip bands and inter-band distance were measured using transmission electron microscopy (TEM). The total dislocation density was found to slightly decrease and the density in slip bands to increase with increasing number of cycles. The results concerning the dislocation density were compared with changes in the microplastic limit (MPL) which were determined by the measurement of the inductance of the “specimen - coil” system. They consisted in a rapid initial decrease at the first stage of the fatigue process and in a gradual increase during the major part of the life. These changes can be interpreted on the basis of changes in dislocation density as was verified independently by X-ray diffraction and nanoindentation tests. On the basis of the measurement of dislocation density by TEM it appears that changes in MPL can be connected with the dislocation density in slip bands rather than with the total dislocation density.

Keywords: Carbon steel, cyclic loading, transmission electron microscopy, dislocation, inductance

1. INTRODUCTION

The dislocation substructures in alloys after cycling loading have been comprehensively investigated in bcc and fcc metals and alloys [1]. The density and distribution of dislocations differ from those, which are observed after the static loading. Stress cycling of a specimen results in clustering of dislocations into bands and formation of dislocations cells. The planar arrangement of dislocations is typical for alloys with low stacking fault energy (SFE) while dislocation cells occur in alloys with relatively high SFE [2]. Cross slip of screw dislocations is the controlling process for dislocation cell formation. Therefore, in ferritic steels dislocation cells, rather than dislocation bands, can be expected to be formed.

On the basis of changes in dislocation substructure a fatigue failure can be predicted [3]. Density of dislocations as well as their arrangement influence mechanical and physical properties of steel. It is believed that the boundary between damaging and non-damaging stresses is the so-called micro-plastic limit (MPL) defined by the stress co-ordinate of the point of deviation from linearity on the increasing branch of the hysteresis loop L - σ (inductance - stress). It corresponds to the state when local stresses around pile ups of dislocations at obstacles, predominantly grain boundaries, start to obstruct magnetic domains in rotation to the direction of tensile macrostress [4]. During fatigue loading the MPL does not keep a constant value but it varies.

Another characteristic point on the hysteresis loop L - σ is that of zero-stress inductance L_0 which characterizes the basic magnetic state of a ferromagnetic material upon zero applied stress. The zero-stress inductance L_0 varies with the number of stress cycles much as the MPL.

2. EXPERIMENTAL PROCEDURES

Plain carbon steel S235JR (max 0.17 wt. % C, max 1.4 wt. % Mn, max 0.012 wt. % N) was chosen as the material of the study. Rod specimens of 10 mm in diameter were normalized and vacuum annealed at 640°C for 3 hours, furnace cooled. The yield stress and the ultimate tensile strength of the specimens were determined as an average of three specimens to be R_e = 276 MPa and R_m = 426 MPa. These specimens were used for indirect measurement of magnetic permeability μ - by measuring changes in inductance. The measurements were carried out using a special apparatus for very low impedances. It enabled the output to be sampled and stored by means of the measuring bridge Peekel - Autolog 18 bit, software Autosoft NT.

Investigations of steel structure-phase state were carried out using light optical microscopy (LOM), while substructure was observed using transmission electron microscopy (TEM) of thin foils. The foils were prepared from the steel in as received conditions and after selected numbers of fatigue cycles. Cross slices of the thickness 1 mm were cut from the gauge region of fatigued specimens. Then they were grinded to a thickness of 0.1 mm and discs of 3 mm in diameter were punched. The grey coloured discs in the scheme in **Fig. 1**. represent positions of discs, which were used for final jet electropolishing in 6% solution of perchloric acid in methanol at temperatures ranging between -60°C and -50°C. The areas of about 5,000 μm² around the central hole were transparent for electrons accelerated by a voltage of 120 kV. The observed substructure represented the material conditions in a depth from 1.5 mm to 2 mm under the specimen surface. TEM micrographs taken in two best foils of each specimen were used for quantitative evaluation of dislocation substructure.

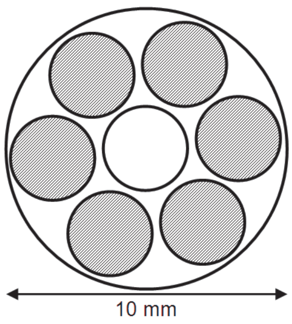

Fig. 1 Cross section of a fatigue specimen at the gauge region. Scheme of discs used for preparation of thin foils

3. RESULTS

3.1 Measurement of inductance

A typical curve of inductance vs. stress is shown in **Fig. 2**. It can be seen here that the ideal mean curve of the inductance vs. stress hysteresis loop is an increasing function of stress. This is due to a concurrence of the direction of tensile strain and magnetic flux lines. The zero stress inductance L_0 and the MPL are indicated on the hysteresis loop *L - σ.* During cyclic loading they change their positions in relation to the co-ordinate system. By plotting these changes against the number of stress cycles n diagrams L_0 - *n* and *MPL* - *n* can be obtained.

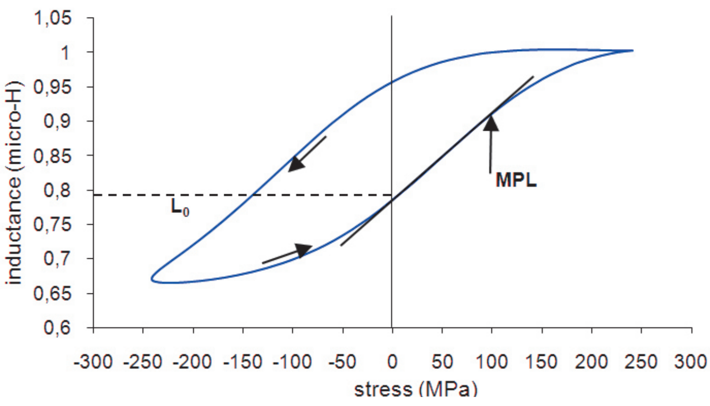

Fig. 2 Inductance (L) vs. stress (σ)

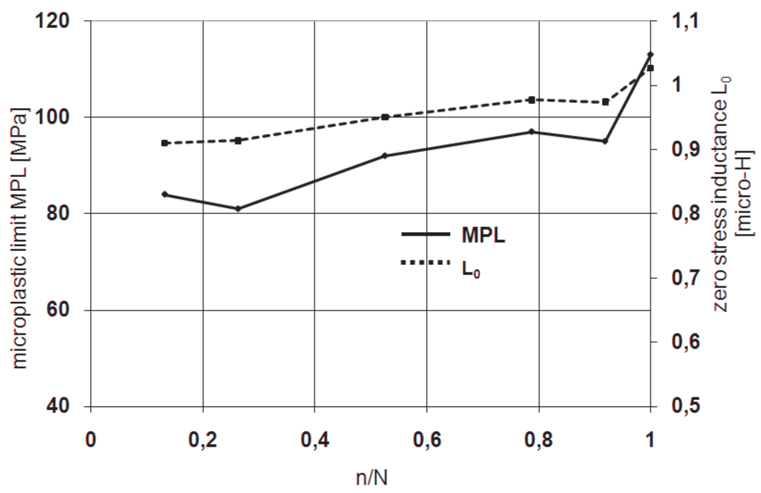

Fig. 3 Variation of MPL and L_0 with the relative number of cycles n/N

Fig. 3 represents the variation of L_0 and *MPL* with the relative number of cycles *n/N* for push-pull loading of the specimens with amplitude σ_a = 242 MPa and frequency 1 Hz.

Both curves exhibit a similar trend: where one curve is decreasing the other one is also decreasing and vice versa. Because L_0 is a measure of magnetic permeability at zero stress it can be supposed that, roughly speaking, the *MPL* varies in the same way as magnetic permeability does. On the basis of the work of Kikuchi et al. [5] it was concluded [6] that permeability, and thus inductance and microplastic limit, are influenced by dislocation density.

3.2 Microstructural study

Microstructure of the steel investigated consisted of ferrite and about 6 volume % of sferoidized pearlite (**Fig. 4**). The mean size of ferritic grains was 15 µm. Some cementite particles were sparsely scattered within ferrite grains. Dislocation substructures of four specimens labelled J1, J2, J3 and J4 were evaluated which represented as received conditions, 0.25, 0.50 and 0.75 of the mean life under cyclic loading respectively. At least ten TEM micrographs of each specimen taken at a magnification of 50,000 were used for measurement of dislocation density. In addition, a projected separation distances between dislocation bands with high dislocation density (inter-band distances) were measured at a magnification of 12,000.

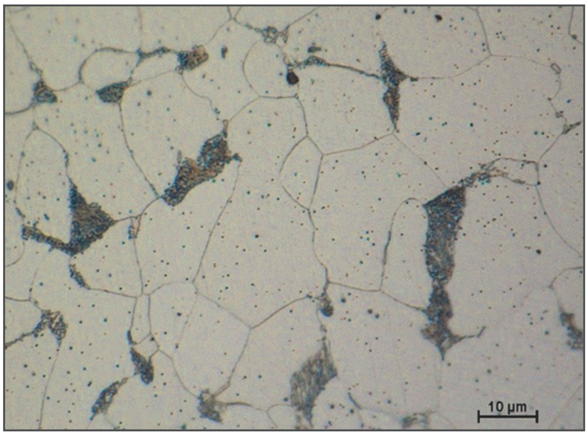

Fig. 4 Microstructure of J1 specimen. LOM micrograph

The dislocation density was determined using the Keh-Weismann´s intersection analysis [7]. A rectangular grid was put on TEM micrograph and intersections with dislocations were counted. Then dislocation density ρ was determined using the formula

$$\rho = \left(\frac{n_1}{L_1} + \frac{n_2}{L_2}\right) \cdot \frac{x}{t} \qquad (1)$$

where n_1, n_2 are numbers of intersections with dislocations made by two systems of parallel lines with a total length L_1 and L_2 respectively, x is a factor of visibility and t thickness of the foil. The foil thickness was calculated from a number of dark fringes n in a bright field image of a high angle grain boundary taken under two beam conditions using an expression

$$t = n \cdot \xi_g \qquad (2)$$

The following two-beam conditions with the strong reflexions 110, 200 or 211 were used for imaging of dislocation. The corresponding extinction distances given in **Table 1** were substituted for ξ_g in formula (2).

It was supposed on the base of literature data [8], that slip dislocations in ferrite are screw dislocations with Burgers vector $\vec{g} = 1/2[111]$. Dislocations are out of contrast if $(\vec{g} \cdot \vec{b}) = 0$. Factors of visibility in two beam conditions are given in **Table 1**. Results of quantitative evaluation are shown in **Table 2**.

Table 1 Factor of visibility of dislocations and extinction distance in ferrite using acceleration voltage 120 kV

Reflection vector	Factor of invisibility x	Extinction distance ξ_g [nm]
110	2	28.89
200	1	42.27
211	1.3	53.82

Table 2 Results of quantitative evaluation of dislocation substructure - mean values with standard deviation

Specimen	Relative number of cycles n/N_f	Total dislocation density $[10^{14} m^{-2}]$	Dislocation density in dislocation bands $[10^{14} m^{-2}]$	Inter-band distance [μm]
J1	0	0.4 ± 0.2	-	-
J2	0.25	2.1 ± 0.7	3.9 ± 1.3	0.9 ± 0.1
J3	0.50	1.7 ± 0.4	4.3 ± 1.5	1.1 ± 0.1
J4	0.75	1.5 ± 0.2	7.0 ± 1.3	1.0 ± 0.2

A chaotic dislocation net with low dislocation density was observed in the ferritic grains of J1 specimen, which represented as received conditions (**Fig. 5a**). The mean dislocation density was $0.4 \cdot 10^{14}\ m^{-2}$. During the cyclic loading new dislocations were generated and were accumulated in dislocation bands separated by dislocation-poor regions. With increasing number of cycles dislocation density in the bands increases while the total density remains almost the same; it slightly decreased from $2.1 \cdot 10^{14}$ to $1.5 \cdot 10^{14}\ m^{-2}$. (**Table 2**). Taking into account the standard deviations differences in total dislocation density are not important. Development of dislocation substructure after the cyclic loading is shown in **Figs. 5b**, **c** and **d**. The dislocation bands become waved as a result of stress cycling, linked up with increasing number of cycles and formed dislocation cells. The cellular structure was developed in some grains at early stages of the cyclic loading. Formation of dislocation cells within two adjacent ferrite grains was observed in J2 specimen (**Fig. 6**). Well developed cells occurred near the grain boundaries while dislocation bundles were in the grain centre.

Dislocation bands as well as dislocation cells were observed in J3 and J4 specimens. In the dislocation bands dislocation density significantly increased and dislocation cells were better developed. Simultaneously in areas between dislocation bands dislocation density decreased and the mean inter-band distance remained constant. In comparison with J2 specimen the cellular substructures were observed more often, however the substructure with dislocation bands prevailed.

CONCLUSIONS

A comparison of the results of microstructure analyses with the results of electromagnetic measurement revealed some correlations between dislocation substructure and parameters of the hysteresis loop of inductance vs. applied stress.

During the first quarter of the fatigue life heterogeneous dislocation substructure formed that consisted of dislocation bands with high dislocation density separated by regions with low dislocation density. Moreover, in some ferrite grains cellular structure was developed. The features of dislocation substructure remained the same up to about three quarters of the fatigue life. The increase in dislocation density in dislocation bands (and also on cells boundaries) was the only parameter, which changed significantly during cyclic loading.

Measurement of inductance of specimens in certain stages of the cyclic loading showed the same trend of variation of both the microplastic limit MPL and the zero stress inductance L_0 during application of stress cycles. They slightly decreased in the early stages of the fatigue life. The dislocation bands act as obstacles for dislocation motion similar to grain boundaries. They probably effect the rotation of magnetic domains. An increase in dislocation density in strips causes stabilization of these obstacles and consequently a gradual increase in MPL and L_0. The assumption that permeability increases with dislocation density in a certain range, supported by [5], was verified not only by transmission electron microscopy, as used in this study, but also by X-ray diffraction analysis and nanohardness tests [9].

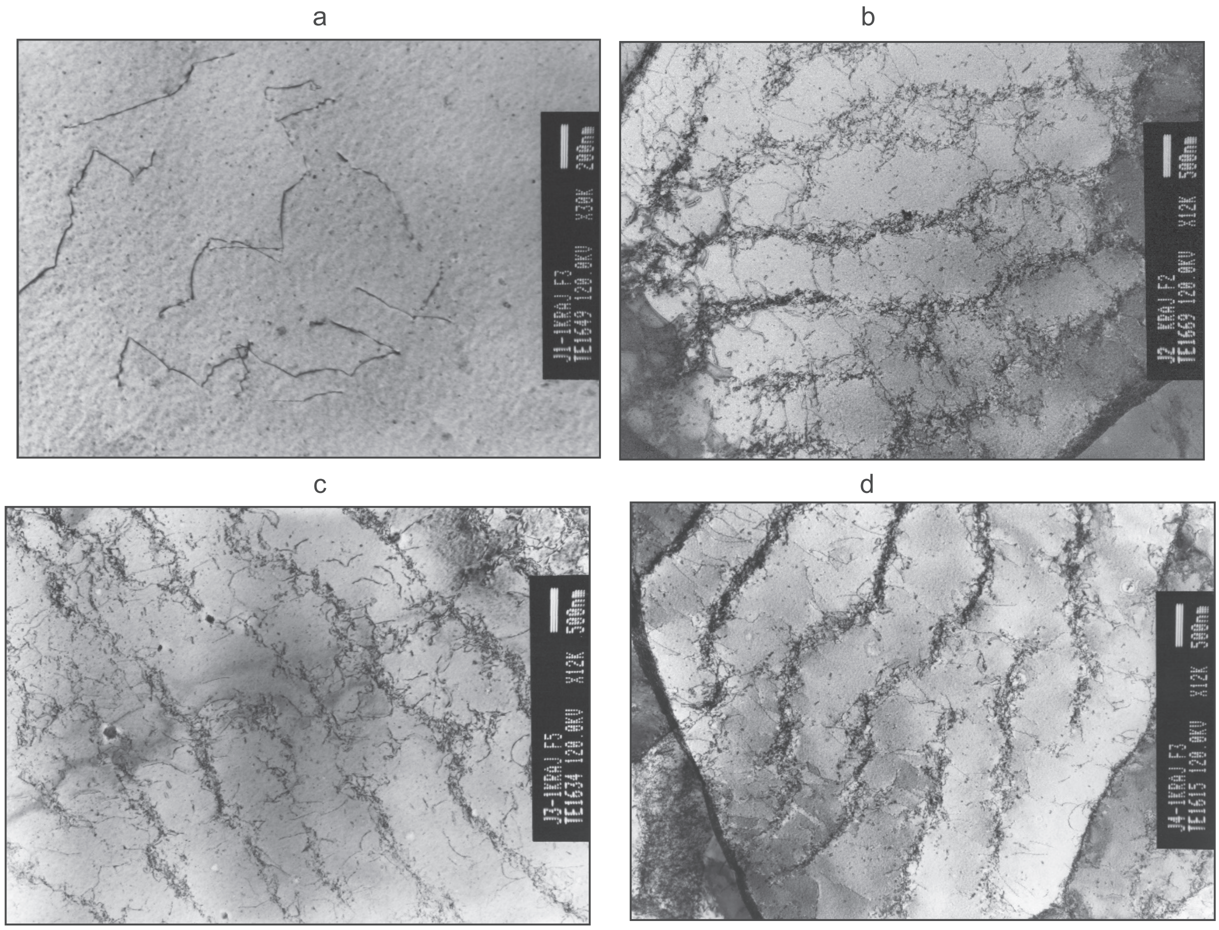

Fig. 5 Dislocation substructure: a) J1, b) J2, c) J3 and d) J4 specimen. TEM micrographs

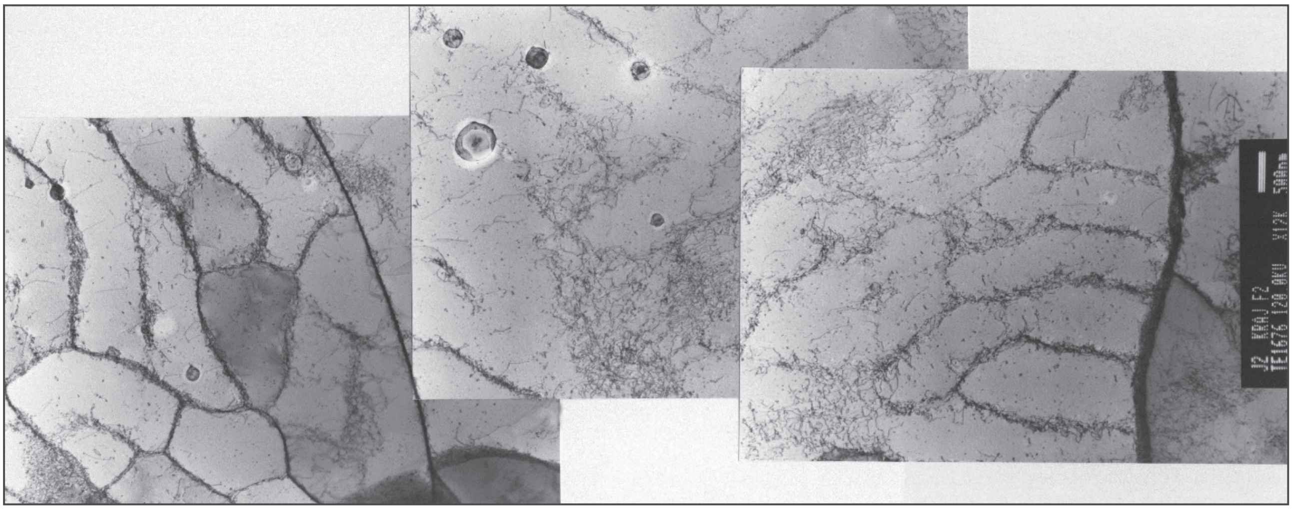

Fig. 6 Dislocation substructure of J2 specimen. TEM micrographs

ACKNOWLEDGEMENTS

We are very grateful for financial support of our research from the Grant Agency of the Czech Republic (project no. 105/10/2052) and the Technology Agency of the Czech Republic (projects no. TE01020118 and TE02000162).

REFERENCES

[1] MUGHRABI, H., ACKERMANN, F., HERZ, K. Persistant Slipbands in Fatigued Face-Centred and Body-centred Cubic Metals. In *Fatigue mechanisms: Proceedings of ASTM-NBS-NSF Symposium*: May 1978, Kansas City. J. T. Fong Ed. ASTM-STP 675, American Society for Testing materials, 1979, 69-105.

[2] KOVÁCS, I., ZSOLDOS, L. *Dislocations and Plastic Deformation*. Pergamon Press, 1973.

[3] BRINCKMANN, S. *On the Role of Dislocations in Fatigue Crack Initiation*, Rijksuniversiteit Groningen, Groningen, 2005. ISBN 90-367-2237-3.

[4] GAJDOŠ Ľ., ŠPERL M., KAISER J., MENTL V. Microplastic Limit of Steel Determined by the Measurement of Changes in Electrical Impedance. In Conf. Proc. *Experimental Stress Analysis 2011*. T. Návrat, V. Fuis, L. Houfek, M. Vlk Eds. Brno: University of Technology, 2011

[5] H. KIKUCHI, Y. HENMI, T. LIU, K.ARA, Y. KAMADA, S. KOBAYASHI AND S.TAKAHASHI. The Relation Between AC Permeability and Dislocation Density and Grain Size in Pure Iron. *Int. J. Applied Electromagnetics and Mechanics*, 25, 2007, 341-346.

[6] GAJDOŠ Ľ, ŠPERL M., KAISER J., MENTL V. Utilizing Electromagnetic Properties to Determine the Micro-Plastic Limit of Low-Carbon Steel CSN 411375. *Int. J. Applied Electromagnetics and Mechanics* (in print).

[7] KEH, A. S., WEISSMANN, S. *Electron Microscopy and Strength of Crystals.* New York: Wiley, 1963, p. 231.

[8] HIRT, J. P., LOTHE, J. *Theory of dislocations*. New York: Wiley, 1982.

[9] GAJDOŠ Ľ, ŠPERL M., FRANKL J, KAISER J., MENTL V., KYNCL J., LUKEŠ J., KUŽEL R. Verification of the Trend of MPL Variation in Fatigue by Modern Methods. In: Proc.of the Int. Conf. *Local Mechanical Properties 2013,* Kutná Hora, Czech Republic. P. Haušíld, A. Materna and M. Vilémová Eds. Praha: Publishing House CVUT. ISBN 978-80-01-0534-4.