STRAIN LOCALIZATION DURING LOW-CYCLE FATIGUE OF IRON AND IRON-SILICON ALLOYS SINGLE CRYSTALS

V. Novák, B. Šesták and S. Kadečková

Institute of Physics, Czechoslovak Academy of Sciences, Na Slovance 2, 180 40 Prague 8, Czechoslovakia

ABSTRACT

Cyclic deformation tests were performed at 295 K and 524 K on single crystals of Fe and Fe with 0.5, 0.9 and 3wt.%Si in plastic strain ranges $\Delta\varepsilon_{pl}$ from 10^{-4} to 10^{-2}, at a strain rate of $\dot{\varepsilon}_t = 1.6\times10^{-3}\,s^{-1}$. Besides the stress-strain characteristics, slip traces and dislocation arrangements were studied. Bands of localized plastic strain reminiscent of persistent slip bands reported on f.c.c. metals were observed after deformation $\Delta\varepsilon_{pl} \geq 10^{-3}$. On Fe-0.5%Si and Fe-0.9%Si, these bands were present both at 295 and 524 K, while on Fe they were observed only at 524 K. The results of the experimental investigation are correlated with those obtained on f.c.c. single crystals. The similarities and differences in cyclic deformation behaviour of f.c.c. and b.c.c. single crystals are discussed.

KEYWORDS

Low cycle fatigue, single crystals, persistent slip bands, dislocation structure.

INTRODUCTION

Microscopic mechanism of cyclic deformation has been studied extensively on a number of f.c.c. metals in recent years. A very important feature is localization of plastic deformation in the so-called persistent slip bands (PSB) which give rise to microcrack formation in the later stages of deformation. For this reason a large attention has been paid to the conditions under which the PSB are formed. The situation is somewhat different in b.c.c. metals. The PSB were observed only on a few b.c.c. alloys (e.g. Mughrabi 1979, Pohl et al. 1980, Gonzales and Laird 1983), while for pure b.c.c. metals they have not been reported yet. It has been show already by the unidirectional strain experiments that plastic behaviour of b.c.c. metals depends strongly on temperature, strain rate and impurity content

(Šesták and Seeger 1978). Under certain conditions plastic behaviour of b.c.c. metals is similar to that of close packed metals.

The unidirectional tensile deformation of the Fe and Fe-Si single crystals, which were chosen for the present experiments, was studied in detail (Novák et al. 1976, 1984). In the present series of experiments the samples were cyclically deformed under conditions (low strain rate, intermediate temperatures) where mobilities of screw and edge dislocations are comparable and, thus, behaviour similar to f.c.c. metals may be expected. A main emphasis is laid on the observed localization of plastic deformation, and on similarities and diferences in behaviour of materials under investigation and that of f.c.c. metals in the discussion of the experimental results.

EXPERIMENTAL METHODS

Single crystals of iron and iron with 0.5, 0.9 and 3.0wt.% silicon were used for the experiments. The Fe-3%Si crystals were grown by the floating zone method, the other crystals by the strain anneal method. The diameter of the specimens was 4.0 mm and the length 40 mm, the diameter of the central gauge part of length 10 mm was diminished electrolytically to 3.5 mm. The content of carbon and nitrogen was reduced to about 1 ppm by final annealing in a closed ZrH_2 system for 100 hours at 1100 K. The total content of other impurities was less than 20 ppm in Fe, Fe-0.5%Si and Fe-0.9%Si, and about 400 ppm in Fe-3%Si. The single crystals were oriented for single slip (orientation approximatelly [$\bar{2}$49]), with the primary slip direction [111] and maximum resolved shear stress plane ($\bar{1}$01).

Symmetrical tension-compression tests were carried out in an Instron TT-DM machine at room temperature in air and at temperature of 524 K in argon atmosphere. Cyclic work-hardening curves were measured in constant plastic strain ranges $\Delta\varepsilon_{pl}$ (here defined as the width of the σ-ε hysteresis loop) between 0.0005 and 0.01. In addition, the incremental step method was used for measurement of cyclic stress-strain curves. The specimens were deformed at the total strain rate of $1.6\times10^{-3}\,s^{-1}$. To determine the internal stress σ_G and effective stress σ^*, the stress relaxation method was used. A correction to the work-hardening was made using the method proposed by Vlachynsky et al (1978). The surface of the specimens was studied by optical microscope Opton, Photomicroscope III, equipped with Nomarski interference contrast. In some cases deformation was interrupted, the specimens were repolished and the surface of the specimens was observed again after an incremental deformation. For the transmition electron microscopy (TEM) thin foils were prepared parallel to the primary slip plane ($\bar{1}$01), parallel to the secondary slip plane (101) and perpendicular to both these planes, i.e. parallel to (010). The foils were observed in a JEM 6A microscope operating at 100 kV.

EXPERIMENTAL RESULTS

Cyclic hardening behaviour. Cyclic hardening curves for Fe and Fe-0.5%Si are presented in Figs. 1a, b, where the mean peak

stress σ in tension and compression, is plotted against the cumulative plastic strain, ε_{cum}. Practically all curves have the same general shape - the region of rapid hardening is followed by saturation. The saturation have been not reached for cumulative deformation less than 6 for Fe-0.5%Si at $\Delta\varepsilon_{pl}$=0.0005 and for Fe-3%Si for $\Delta\varepsilon_{pl}$ from 0.0005 to 0.01. The initial cyclic hardening rate ($\Delta\sigma/\Delta\varepsilon_{cum}$ for $\Delta\varepsilon_{cum}$ = 0.2) is very high, between 90 and 170 MPa. These values are comparable with the maximum work-hardening rates in tension tests (Novák et al. 1976). Fig. 2. shows the work-hardening rate $\hat{\tau}$ at the peak of the hysteresis loop as a function of ε_{cum} for different plastic strain ranges $\Delta\varepsilon_{pl}$, as measured on Fe-0.5Si at 295 K. The cyclic stress strain curves which were obtained by plotting the stress at saturation, σ_s, against the strain range $\Delta\varepsilon_{pl}$, are presented in Fig. 3. Since no saturation was reached in our experiments for Fe-3%Si, the stress σ_s plotted in Fig. 3. for this alloy was defined as the stress at which the cyclic hardening rate is equal to 5 MPa.

Surface observations. The slip line structure an the surface of the single crystals cyclically deformed to saturation, $\varepsilon_{cum}\sim5$, is very diverse. The strain localization was observed in saturation on Fe-0.5%Si and Fe-0.9%Si for $\Delta\varepsilon_{pl}$ in the range from 0.001 to 0.01 at 295 and 524 K. However, on Fe specimens it was found only at 524 K for all investigated $\Delta\varepsilon_{pl}$ (from 0.0005 to 0.01). On the other hand the slip line pattern on Fe at 295 K is relatively homogeneous. Very homogeneous slip line structure was observed on Fe-0.5%Si at $\Delta\varepsilon_{pl}$= 0.0005 and on all investigated Fe-3%Si single crystals. The phenomenon of strain localization was investigated in detail on Fe-0.5%Si at room temperature. The strain is concentrated into slip bands. Due to the similarity to the persistent slip bands reported on f.c.c. metals we denote these bands as PSB. The PSB nucleate in saturation at $\varepsilon_{cum}\simeq2.5$ as indicated by arrowa in Fig. 2. The development of PSB after repolishing of the surface is ilustrated in Fig.4. Slip steps appear and disappear every half-cycle, and the pattern of the bands stays virtually unchanged for many cycles. It indicates high degree of reversibility of slip in the PSB's. The reversible strain concentrated in the PSB, determinated from interferometric measurements was $\varepsilon_{B,rev}$ = 0.008. After more than 100 cycles the extrusion growth was observed in the PSB on the top face of the samples. The extrusion growth rate was of the order of one Burgers vector per cycle. Optical microscopic study with Nomarski interference contrast shown that the narrow PSB have a pearl-like structure which changes in the broader PSB to a pattern of straight lines arranged in remarkably regular intervale - Fig. 5. The lines correspond to the traces of the so-called anomalous slip plane. No PSB's are formed in the plastic strain range $\Delta\varepsilon_{pl}$ below a rather ill-defined critical value $\sim$0.0008. Above this critical plastic strain range the volume fraction of PSB's increases linearly with the $\Delta\varepsilon_{pl}$ from zero to nearly a unity at $\Delta\varepsilon_{pl}\sim0.008$.

Dislocation arrangements. Cell structure was found in all iron specimens deformed to cummulative deformation corresponding to the end of work-hardening curves in Fig. 1a, with the exception of the iron specimen deformed at room temperature at $\Delta\varepsilon_{pl}$ equal 0.0005 when only individual long dislocations with the primary

Burgers vector were observed; their density was very low, about $10^{12}\,m^{-2}$. In all specimens of iron-silicon alloys at least in the initial stages of deformation, dislocation structure was found similar to the veining structure observed in copper (Kuhlmann-Wilsdorf and Laird 1977). The dislocations in the loop patches (Fig. 6 and 7) have predominantly the primary Burgers vector [111] and are of edge character. Their density is up to $10^{16}\,m^{-2}$. Between the veins the density varies from 10^{14} to $10^{13}\,m^{-2}$ in different specimens. The dimensions of loop patches increase with decreasing silicon content. As observed in $(\bar{1}01)$ - foils parallel to the primary slip plane, the average spacing of veins in crystals of 3%, 0.9% and 0.5%Si is 1.2, 5.8 and 8.1 μm, respectively. The secondary slip system is activated on further stage of cyclic deformation at the end of the region of the high hardening rate. The dislocations of both systems interact and dislocation networks are formed between the veins (Fig. 8) beeing the nuclei of cell walls. The veins of primary dislocations are dissolved and the cell structure is formed (Fig. 9 and 10). The regions of cell structure are observed on the surface of the specimens as the PSB's. A boundary between the matrix and the cell structure, is shown in Fig. 9. The residual deformation in the PSB as observed on the surface of the specimens copies the individual cells. The dimensions of cells correspond to the dimensions of "pearls" observed on the surface. In some cases large planar networks parallel to the anomalous slip plane are formed by the interaction of the primary and secondary slip systems. They are nearly equidistant and divide the crystal to layers in which the lattice misorientation alternates (Fig. 10). Then fine strips are observed in the PSB's on the surface (Fig. 5b). Their widths and directions correspond to the stripes of cells observed by TEM (Fig. 10).

DISCUSSION

Let us start the discussion with Fe-0.5 and Fe-0.9%Si alloys whose behaviour during cyclic deformation have many similar features to f.c.c. metals, e.g. to Cu and Ni (Mughrabi 1978, 1979, Blochwitz and Veit 1982, Basinski and Basinski 1983, Pedersen and Winter 1982, Tabata et al. 1983). The cyclic stress strain curves exhibit a plateau. It was demostrated for Cu and Ni, and it is true for our single crystals as well, that the existence of the plateau is connected with occurence of the PSB. The volume fraction occupied by PSB's increases with $\Delta\varepsilon_{pl}$ along the plateau, at the lower end of the plateau it is approximately zero, at the upper end the PSB's fill the whole specimen volume. The reversible plastic deformation ε_{B} concentrated in the PSB is independent of $\Delta\varepsilon_{pl}$ and reaches the value of ~ 0.01. The irreversible deformation in PSB's is estimated to be of the order of $0.01\,\varepsilon_{B}$ and causes the growth of extrusions on the top face of the sample. The extrusion growth rate is of the order of one Burgers vector per cycle. The initial cyclic hardening rates are comparable with the maximum work-hardening rates in the tensile test. The work-hardening rate in the peak of the hysteresis loop, ϑ, depends strongly on $\Delta\varepsilon_{pl}$. It reaches a value comparable with the value of the shear modulus G for low $\Delta\varepsilon_{pl}$ and decreases rapidly with increasing $\Delta\varepsilon_{pl}$.

All the phenomena mentioned so far have been well documented

for Cu and Ni, as well as for our Fe-0.5 and 0.9%Si crystals. However, there are also some differences in the behaviour of these materials. The dislocation structure of the PSB in Fe-0.5%Si is characterized by the cell structure consisting of both primary and secondary dislocations, in contrast to the ladder structure of PSB's in f.c.c. metals consisting predominantly of primary dislocations (Mughrabi 1978, 1979, Tabata et al. 1983, Lukáš et al. 1968). It can be explained by the fact that the plateau stress for Fe-0.5%Si is high enough to activate the secondary dislocations, as follows from the tensile experiments (Novák et al. 1976). Hence, a contribution of the secondary dislocations to the dislocation structure can be expected. The PSB's in Fe-0.5%Si are characterized by a fine structure which corresponds to the inhomogeneity of irreversible processes. The pattern observed inside the PSB's on the specimen surface is a copy of the dislocation cell structure as follows from a quantitative correlation between the periodicity of the surface relief and the dimensions of the cells. In the f.c.c. metals, on the other hand, the surface relief of the PSB's is much more homogeneous and nearly featureless. In both cases, in the f.c.c. as well as in the b.c.c. metals the veining dislocation structure, which appears at low ε_{cum} in the whole specimen volume, remains still present between the PSB's at high ε_{cum}.

The question under what circumstances the localization of plastic deformation occurs and what dislocation structure is connected with the localization are the basic problems of the cyclic deformation of b.c.c. metals. Two different types of the dislocation structure have been observed in cyclically deformed crystals: the veining structure formed predominantly by primary dislocations and the cell structure consisting both of primary and secondary dislocations. Let us denote σ_t as the stress at which the sources of the secondary dislocations are activated in unidirectional tensile tests (the transition of the Stage I to Stage II/III) and σ_f as the stress at which the cell dislocation structure develops during cyclic deformation. The Table summarizes stresses σ_t and σ_f estimated for all investigated materials from the tensile tests (Novák et al. 1976) and the present experiments, respectively.

TABLE

Material	σ_t(295 K) [MPa]	σ_f(295 K) [MPa]	σ_t(524 K) [MPa]	σ_f(524 K) [MPa]
Fe	50	~70	5	<20
Fe-0.5%Si	150	150	120	110
Fe-0.9%Si	180	180	140	-
Fe-3%Si	>400	>440	>400	>420

It is seen from the Table that the stress σ_f (the plateau stress) agrees very well with the stress σ_t for Fe-0.5%Si and Fe-0.9%Si. Thus the localization of deformation seems to be correlated with the activation of secondary dislocations. In Fe-3%Si the stress for activation of the secondary dislocations is high and was not achieved during our cyclic deformation experiments. It is in agreement with the absence of the PSP's and with the observed veining dislocation structure. It might be expected that the strain localization could be observed in saturation (i.e. for sufficiently high ε_{cum}). This opinion is supported by the results of cyclic deformation in bending (Boettner and McEvily 1965, Libovický 1984) where the PSB's were observed after high number of cycles on Fe-3%Si. The situation is more complicated for Fe. The saturation is reached at relatively low ε_{cum} in comparison to Fe-Si alloys and f.c.c. metals. Nevertheless, the typical localization of plastic deformation was not found and only distinct bands which might be considered as the PSB were visible on the specimen surface at 524 K. However, the dislocation structure inside the specimen consisted of cells homogeneously distributed in the whole volume. The cell structure was observed in all iron specimens deformed at the saturation stress higher than σ_t. The occurence of the cell structure and the absence of the PSB's is in the agreement with other investigations on pure b.c.c. metals (Mughrabi 1979, Anglada and Guiu 1981).

From our experiments it may be concluded that the cell structure develops if mean peak stress, σ, is comparable to or higher than the stress σ_t, necessary for the activation of secondary dislocations in the tensile test. For σ lower than σ_t, dislocation structure consisting only of primary dislocations is observed. The strain localization, i.e. the presence of well-defined PSB's occurs only if the saturation stress, σ_s, for cyclic deformation is near to the stress σ_t.

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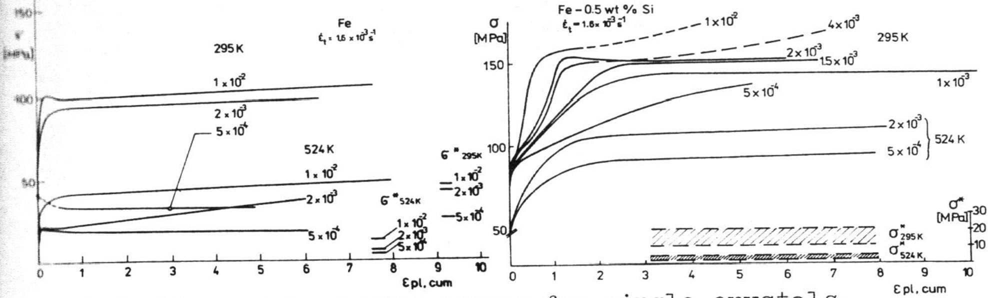

Fig. 1 Cyclic work-hardening curves for single crystals. σ^* is the effective stress. a) Fe b) Fe-0.5%Si.

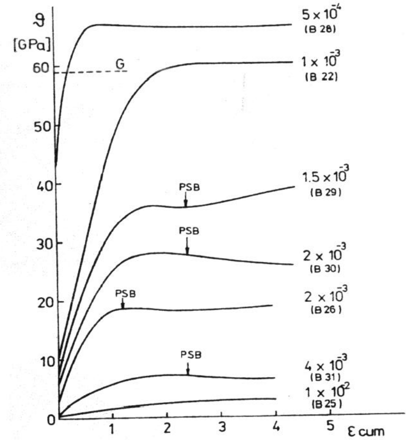

Fig. 2 Cyclic hardening curves for Fe-0.5%Si, 295 K. The arrows indicate formation of PSB's.

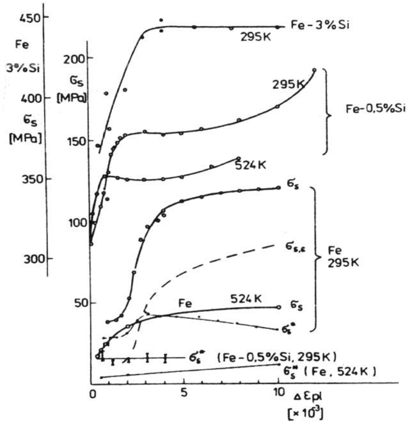

Fig. 3 Cyclic stress-strain curves for single crystals.
σ_s – the saturation stress
σ_{SG} – the internal stress
σ^* – the effective stress

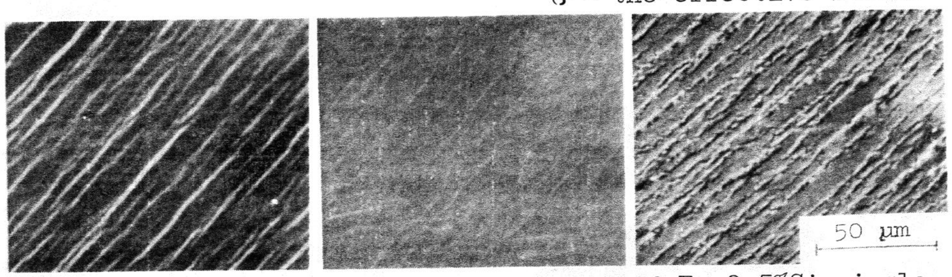

Fig. 4 Slip line pattern on the top face of Fe-0.5%Si single crystal, $\Delta\varepsilon_{pl}$ = 0.0015, 295 K. The surface was repolished after 1600 cycles. The same place on the surface. a) additional 1/2 cycle b) 1 cycle c) 675 cycles

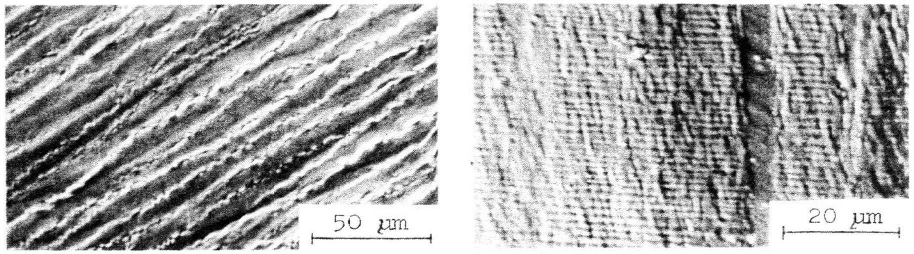

Fig. 5 Slip line structure in PSB's.
a) pearl-like structure, Fe, 524 K, $\Delta\varepsilon_{pl} \sim 0.0005$, 9000 cycl.
b) fine structure in PSB, Fe-0.9%Si, 295 K, $\Delta\varepsilon_{pl} = 0.002$. After 1300 cycles the surface was repolished, additional 283 cycles.

Dislocation arrangement (for detail see the text).

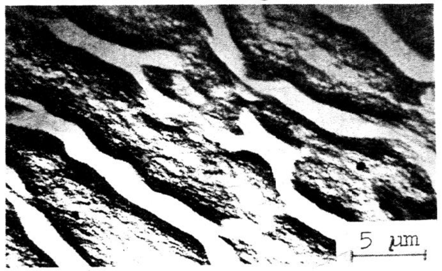

Fig. 6

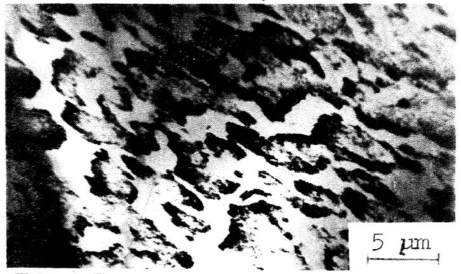

Fig. 7

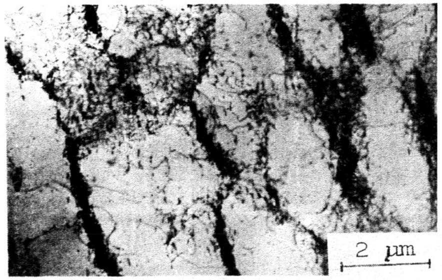

Fig. 8

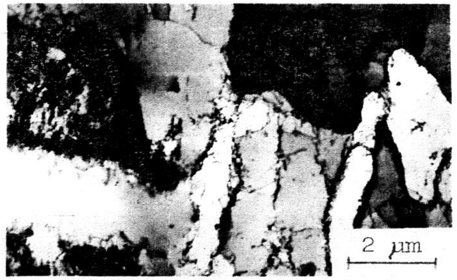

Fig. 9

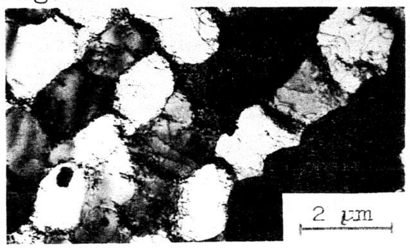

Fig. 10

Fig. 6 Fe-0.5%Si, 295 K, $\varepsilon_{cum} = 5$, $\Delta\varepsilon_{pl} = 0.0005$, foil $(\bar{1}01)$

Fig. 7 Fe-0.5%Si, 295 K, $\varepsilon_{cum} = 5$, $\Delta\varepsilon_{pl} = 0.0005$, foil (101)

Fig. 8 Fe-0.5%Si, 295 K, $\varepsilon_{cum} = 8$, $\Delta\varepsilon_{pl} = 0.01$, foil $(\bar{1}01)$

Fig. 9 Fe-0.5%Si, 524 K, $\varepsilon_{cum} = 8$, $\Delta\varepsilon_{pl} = 0.002$, foil (101)

Fig. 10 Fe-0.9%Si, 295 K, $\varepsilon_{cum} = 9$, $\Delta\varepsilon_{pl} = 0.008$, foil $(\bar{1}01)$