

Investigation of dislocation structure of low carbon steel during static loading

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1. Introduction

Analysis of experimental and theoretical investigations shows, that there is no full and clear understanding of the character of plastic deformation process in microareas of a polycrystalline alloy. Also, there is disputable question on the beginning of plastic strain at a monotonic tension.

It should be noticed, that the investigations of heterogeneity of plastic strain carried out by the majority of authors did not take into account the fact, that deformation in near surface layer and in the volume of a material proceeded differently [1-4]. Hence, regularities of the distribution of nonuniform strain in microvolumes of a polycrystalline alloy still obviously are not enough investigated. Such investigations are very valuable because the study of micrononuniform plastic strain regularities will promote the formation of understanding of extremely complex picture of strength and plastic properties of a material as a whole.

In case of polycrystalline metallic materials and alloys with BCC lattice the periodical and gradual character of plastic deformation processes at monotonic tension can be presented as follows (Fig. 1) [5].

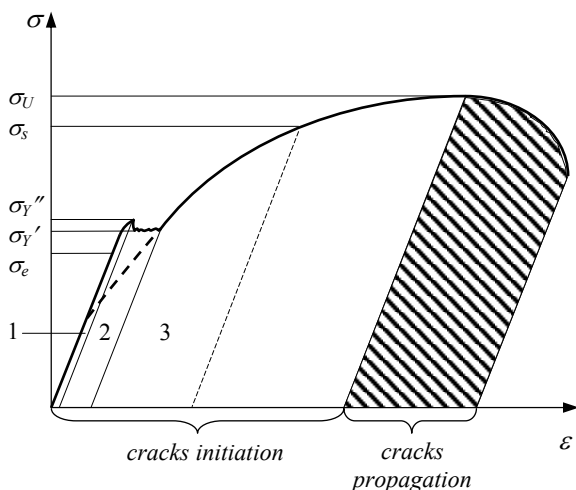


Fig. 1 Failure process stages at static loading BCC lattice possessing metals

At present in considerations of fracture processes of metallic materials it is accepted to divide all the process of strain accumulation and fracture into two basic periods: the period of cracks initiation, and the period of cracks propagation. At monotonic tension, it is possible plastic strain and the damage accumulated up to the beginning of

neck formation to classify as the period of cracks initiation, and the neck formation with the subsequent fracture as the period of cracks growth (the shaded area on Fig. 1). Further, we shall consider what basic stages of damage accumulation are peculiar to the period of crack initiation at static loading.

The first stage is the *stage of microyield*. This stage lasts from the beginning of loading until the occurrence of first lines of sliding on the yield plateau. At this stage, such characteristics as the limit of proportionality and the limit of elasticity are determined. In spite of the fact that residual macrostrains at this stage practically equals to zero, the metal microplastic strain takes place. For metallic materials with a physical yield limit, the ending of this stage is precisely fixed by the beginning of non-homogeneous strain of Lüders-Chernov.

The second stage is the *stage of yield*, characterized by nonhomogeneous strain in the form of front Lüders-Chernov passing along all working length of a sample. In the metals with yield plateau at monotonic tension, heterogeneous strain on the yield plateau spreads as plastic lava-flow, is related to fast multiplication of dislocations on a line of moving deformation front.

The third stage is the *stage of strain hardening*. At this stage in plastic metals and alloys intensive increase of dislocation density is observed, and at certain critical stress σ_s , on the surface of metal submicrocracks in the size about 1-3 μm occur. Inside the metal defective structure in the areas with critical density of dislocations also is formed. This stage terminates at the moment, when maximal load is achieved and the neck formation begins.

The deformation of metal near surface layer essentially influences the common character of plastic flow [6]. The reason of this is earlier activation of dislocation sources in near surface layer in comparison with the sources inside the metal. However attempts to find out (by the use of etching pits or electron microscopy) experimentally some gradient of dislocation density near to the surface of the deformed metals have resulted in rather inconsistent results. Some authors near the surface have found a layer with the raised dislocation density; others specify, that the dislocation density in near surface layer is identical to that in the bulk of metal, and some even affirm, that near surface layer "is impoverished" by dislocations in comparison with the volume of a crystal [7-9].

2. Experimental

The changes of dislocation structure of low carbon steel in both surface and internal metal layers at vari-

ous static loading were studied using the method of transmission electron microscopy. Tests were carried out on the samples of low carbon steel, which chemical composition is presented in Table 1.

Table 1
Chemical composition of low carbon steel, %

C	Si	Mn	Cr	P	S	Ni
0.07	0.025	0.35	0.10	0.03	0.025	0.20

The plain samples with the dimensions of deformable part $1.6 \times 12.0 \times 62.0$ mm were produced from sheets of this steel. Aiming to achieve an equilibrium structure on the cross-section of a sample and remove residual stresses arising during mechanical and subsequent processing, the samples were annealed in vacuum of $2 \cdot 10^{-4}$ mm Hg at temperature of 970°C within 2 hours. The cooling was made together with the furnace. Such temperature of heat treatment and slow cooling is chosen aiming to obtain as low as possible dislocation density in an initial condition. Microstructure investigation of the samples revealed equiaxial grains of the average size $\sim 30 \mu\text{m}$.

Directly after the heat treatment from each side of a sample by gradual electropolishing the near surface layer of about $50 \mu\text{m}$ depth was removed; and after each removal step the sample deflection was measured by optimeter. On the one hand, it provided removal of decarbonised near surface layer, if such was formed during the heat treatment, and on the other hand, gave an experimental confirmation of the absence of residual stresses in the samples in their initial state.

Mechanical properties of tested steel specimens after heat treatment are presented in Table 2.

Table 2
Mechanical properties of low carbon steel

Tensile strength R_m , MPa	Yield strength R_e , MPa	Elongation A , %	Reduction of area Z , %
360	250	35	60

Monotonic tension of samples was carried out at room temperature at strain rate $3.4 \cdot 10^{-4} \text{ s}^{-1}$. High quality of foil preparation was ensured by the use of original techniques and the devices showed in [10].

The dislocation structures were investigated in various depths from the surface of a sample (0, 5, 10, 20, 40, 60, 80, 100, 130, 160, 200, 250, 300, 400, 500 μm) at various degrees of strain on electronic microscope *IEM-7A*.

3. Results and discussion

In the initial condition the structure of annealed low carbon steel consists of the grains having small dislocation density with rare grid of suborders. At dislocation density of 10^7 cm^{-2} one dislocation falls to the area of $10 \mu\text{m}^2$, and at magnification of 40 thousand field of vision of the microscope makes $3.5 \mu\text{m}^2$. Therefore in some grains in all the field of vision of the microscope no dislocation was noticed.

After some insignificant tensile strain, i.e. at the stress about 40 MPa in some single grains in near surface layer depth up to $80 \mu\text{m}$ the patterns of plastic strain were found out (Fig. 2). The microphoto shows that in the grain

body nonuniformly distributed dislocations have appeared; the main part of them was accumulated at inclusion. The bent pieces of dislocations also are visible. Such configurations of dislocations are formed under action of the applied stress, because in annealed condition such areas of dislocations were not observed. In internal volumes of the material no change of dislocation structure in comparison with annealed samples is noticed.

In the process of increase of the degree of plastic deformation, in more and more near surface grains plastic strain is noticed, and in some grains (Fig. 3) considerably greater number of dislocations is observed. In grain A there are no dislocations, while in grain B dislocations are enough long and direct lines could be observed. There is no interaction between them.

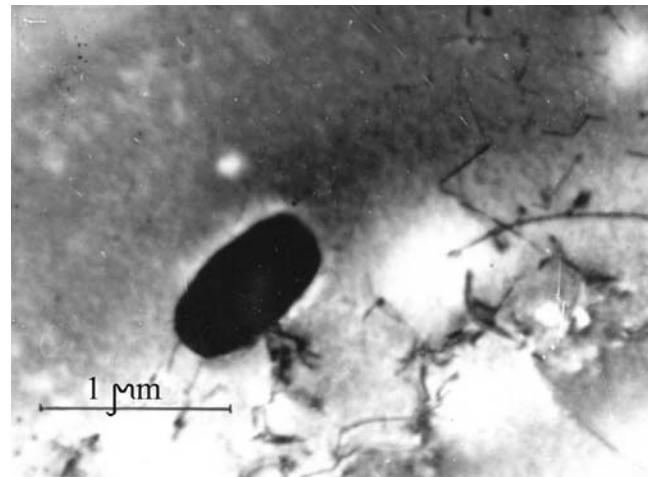


Fig. 2 Dislocation structure of low carbon steel at the stress level 40 MPa in near surface layers depth up to $80 \mu\text{m}$

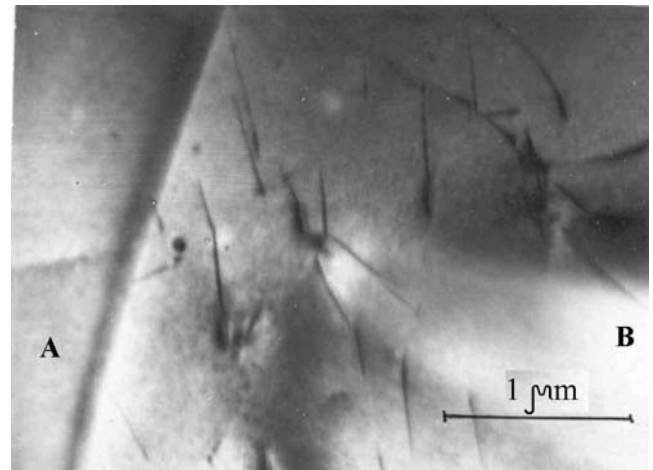


Fig. 3 Dislocation structure of low carbon steel at the stress up to elastic limit σ_e in near surface layers

At a certain critical stress equal to the elastic limit σ_e , dislocations begin to occur almost in all near surface grains. The general structure becomes more complex. Interaction of dislocations is observed. On separate dislocations some steps are visible, and the distribution of dislocations in the structure of a grain is more uniform (Fig. 4). In the photo areas with long dislocations can be seen, as cores on which other moving dislocations are arrested. At the same time these condensations of dislocations, apparently, are the places of active work of several sources. When

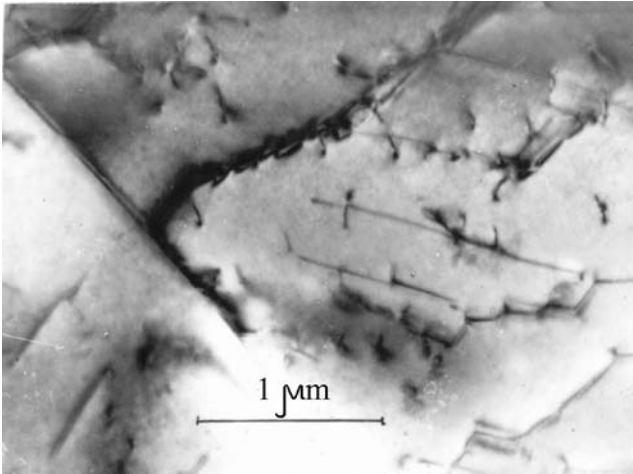


Fig. 4 Dislocation structure of low carbon steel at the critical stress equal to elastic limit σ_e in near surface layers

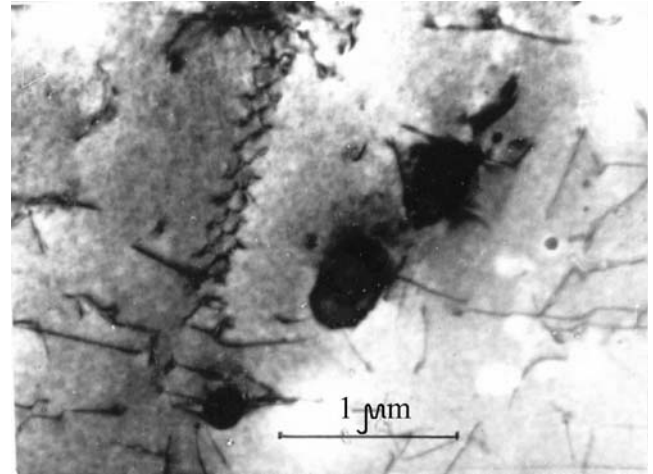


Fig. 6 Dislocation structure of low carbon steel strained to the upper yield limit in near surface layers depth up to 80 μm

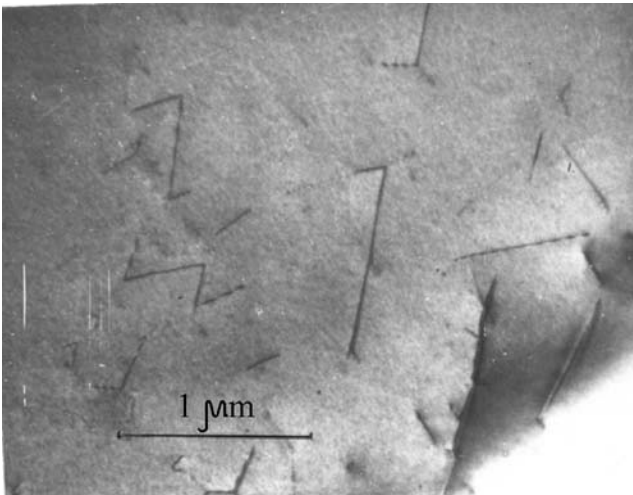


Fig. 5 Dislocation structure of low carbon steel at the critical stress equal to elastic limit σ_e in internal volume of the material

elastic limit σ_e is achieved in internal volumes of the material the formation of dislocations inside some grains (Fig. 5) is observed.

After deformation of a material up to the upper yield limit, in near surface layer depth up to 80 μm the density of dislocations grows. More and more often there are thresholds on the dislocations; these thresholds are the consequence of dislocation crossings. The bent pieces of dislocations are observed also, and dislocations are distributed in all matrixes (Fig. 6). In this microphoto hexagonal grid of dislocations which, probably, is formed during annealing and was in part kept not broken during plastic strain also is visible. At the very surface of a sample the density of dislocations also has increased. In connection with the development of the processes of their complex crossing the local textures have appeared, and the tendency to the formation of local congestions, reminding the beginning of cellular structure formation (Fig. 7) is observed. In such a way the formed dense condensations of dislocations on one hand may be active sources of dislocations, and on the other hand they may be an obstacle for other moving dislocations. Arrested in such obstacles dislocations, increase dislocation density of walls and their extent.

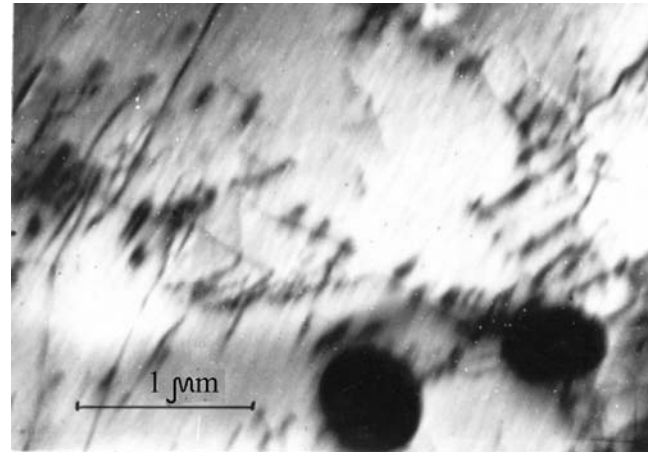


Fig. 7 Dislocation structure of low carbon steel strained to the upper yield limit at the very surface of a sample

Hence, metal deformation conducts not only to moving existing dislocations, but it results also in their multiplication. The increase of the number of dislocations may disturb their further moving due to their interaction. The barriers, interfering motion of dislocations and, consequently, their sliding, arise. This explains the hardening, accompanying cold deformation: if the metal is more deformed, then it is more difficult to deform it further.

After deformation of a material up to the upper yield limit, the increase of dislocations density is observed in internal volumes of the material as well, but here the density of dislocations is much lower than in near surface layer. In internal volumes of the material in the majority of cases are observed long irregularly-shaped lines of dislocations, on many of which thresholds are visible.

Investigating the features of dislocation structure in near surface and internal layers of a material at the stage of microyield, special attention should be paid to the changes of structure of grain borders at various strain degrees. In annealed material on grain borders the details of border structure are not resolved (Fig. 8, a). After insignificant tensile strain (20 MPa), on the background of strips of borders contours dislocations occur (Fig. 8, b), and when the stress $\sigma = 60$ MPa is applied, the amount of dislocations on borders was increased (Fig. 8, c). When the strain achieves elastic limit σ_e , in case of the inclined posi-

tion of grain border, in the object under investigation the picture of correct periodic arrangement of border contours on the image is violated (Fig. 8, d).

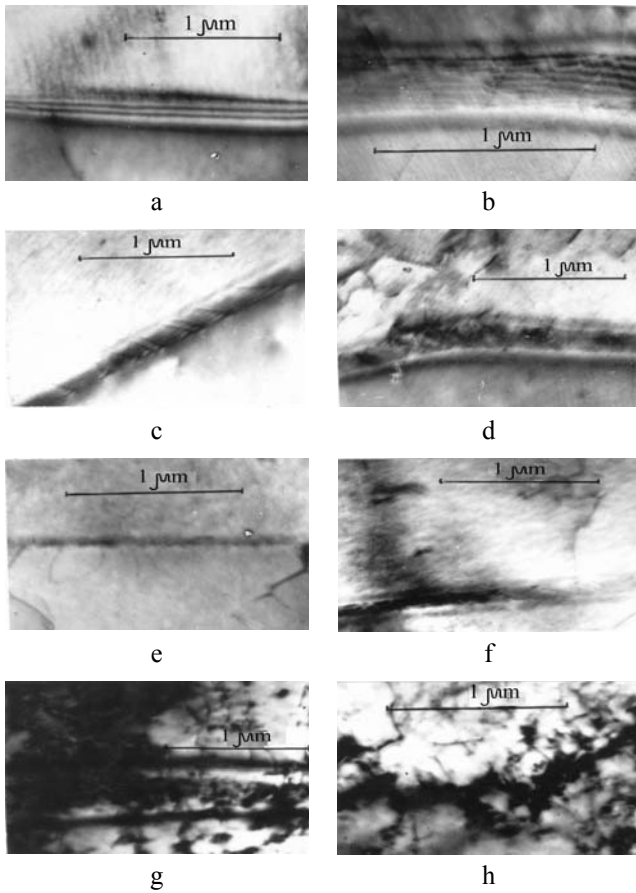


Fig. 8 Dislocation structure of low carbon steel grain borders at various strain degrees in near surface layers: a - in annealed material; b - prestrained in tension at 20 MPa material; c - prestrained at 60 MPa material; d and e - critical stress equal to the elastic limit σ_e ; f - at the upper yield limit. Dislocation structure of low carbon steel grain borders on the yield plateau inside the material (g) and in near surface layers (h)

These features of the structure and diffraction contrast of grain borders specify the presence of significant elastic-plastic strain in near boundary layers of the grains. On some boundaries bent pieces of dislocations are observed, and this testifies that they moved and went output from boundary plane to the grain. This can be seen most clearly when the border in a foil is located almost vertically (Fig. 8, e). At achievement of the upper yield limit, because of the big heap of dislocations on the boundaries, alternating dark and light strips of borders are not noticed (Fig. 8, f).

In the internal material volumes the character of the change of dislocations on grain boundaries remains the same, only dislocations on them are found out at higher stresses, and after deformation of the material up to the upper yield limit, on grain boundaries still it is possible to distinguish alternating dark and light strips of borders, though on them a plenty of dislocations is already observed.

Investigation of dislocation structures on the microyield stage in near surface and internal layers of the metal revealed, that dislocations basically are focused on

grain boundaries. Hence, the boundaries serve as obstacles for movement of dislocations though in many places along grain boundaries (Fig. 7) dislocation loops are visible proving, that borders of grains may serve and as sources of dislocations.

Analyzing experimental data [3, 8, 9], it should be noted that before the beginning of plastic deformation of a material, i.e. the movement and multiplication of dislocations inside grains, the significant amount of dislocations on grain boundaries arise. It is established, that at consecutive development of plastic strain, the density of dislocations on boundaries in all cases is higher than average density of dislocations in adjoining grains.

Thus, at the stage of microyield of low carbon steels there is an appreciable change of dislocation structure of both grain boundaries and the volume of grains. The very first changes in structure of the investigated material, occurring at loading of annealed samples and found out by electron microscopy, should be attributed to grain boundaries of near surface layer of the material: even at insignificant external loading (20 MPa) in these layers dislocations were observed. Also it is established, that at the stage of microyield the degree of deformation development process in near surface layer from a grain to a grain is very much different. In some grains the traces of plastic strain are not noticed yet, and in others the process of sliding already proceeds on two or more systems.

Layer-by-layer electron microscopy analysis has shown, that in the process of metal removal from a surface, the density of dislocations essentially decreases and the character of their arrangement changes. The raised density of dislocations is observed in near surface layer by thickness of 60-100 μm . At approach to the upper yield limit, this layer acts as a barrier interfering, an output of generated by internal sources dislocations on the metal surface. Thus, it may be assumed, that in near surface layer of metals at an initial stage of deformation the anomalous facilitated conditions of plastic flow are realized, therefore the near surface layer with increased dislocation density is formed.

At static deformation of a material on the yield plateau (yield stage), at front movement of microscopic strain the density of dislocations on grain boundaries and in near boundary areas is sharply increased both inside the material (Fig. 8, g and Fig. 9), and in near surface layer (Fig. 8, h and Fig. 10). Boundaries, perpendicular to the plane of the foil, look strongly roughed up because of a set of sticking out of them pieces of dislocations and semi-loops of various sizes that testify that, at these loading stage grain boundaries are capable to generate dislocations. In spite of the fact that on the yield plateau the density of dislocations on grain boundaries and near boundary areas both inside the material and in near surface layer has sharply increased, it is obvious, that the density of dislocations in near surface layer thickness of 100-130 μm is kept higher.

On the yield plateau in some near surface grains (Fig. 11) as well as in internal grains of metal (Fig. 12) the separate generated cells, where sites with separate small density of dislocations are surrounded by sites with the raised density of dislocations are observed. However in near surface layer more advanced cellular structure than in internal layers is observed. No essential distinction in dislocation structure of near surface layer and at the surface of

metal it is noticed. Besides, from Figs. 11 and 12 it may be seen, that process of crushing of the big size cells and formation of the smaller size cells is possible. Thus, formation of cells may proceed in two ways: crushing of larger cells or formation at once small cells.

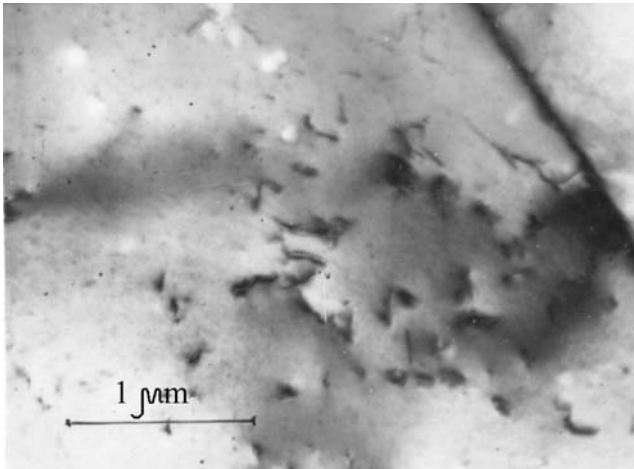


Fig. 9 Dislocation structure of low carbon steel on the yield plateau inside the material

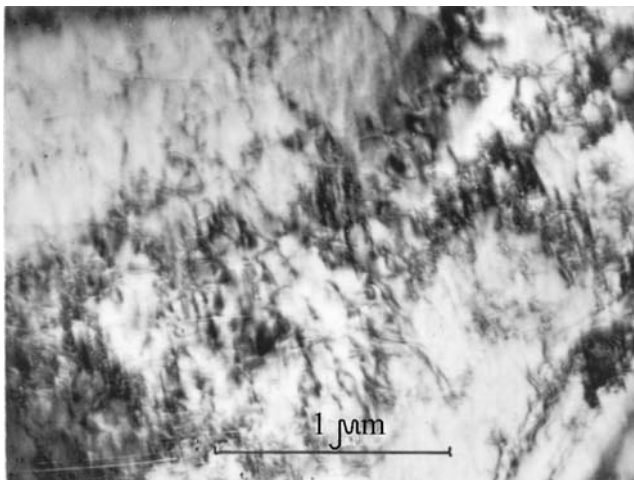


Fig. 10 Dislocation structure of low carbon steel on the yield plateau in near surface layers

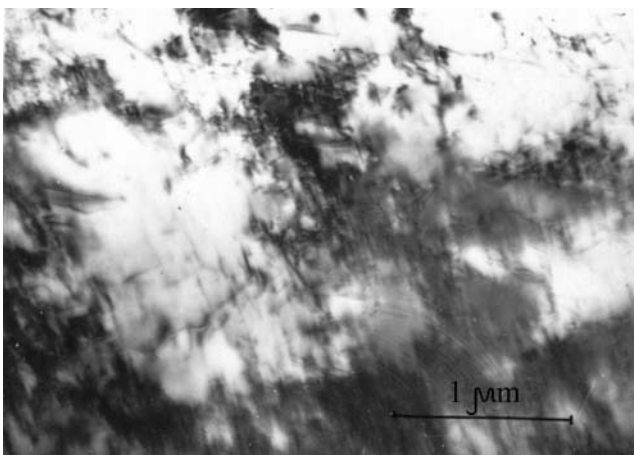


Fig. 11 Dislocation structure of low carbon steel on the yield plateau in near surface layers (the separate generated cells)

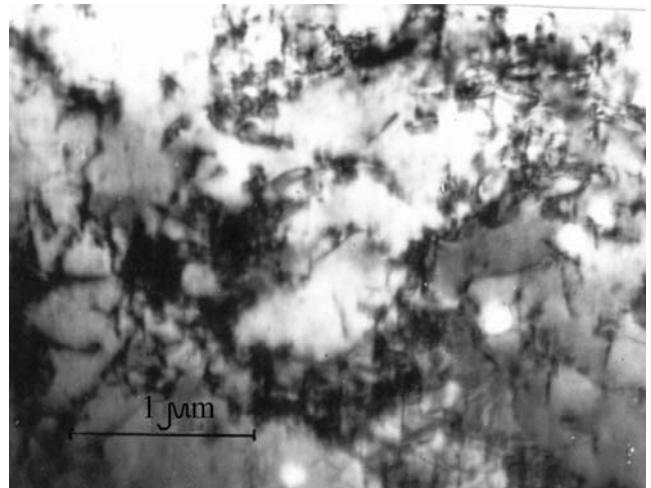


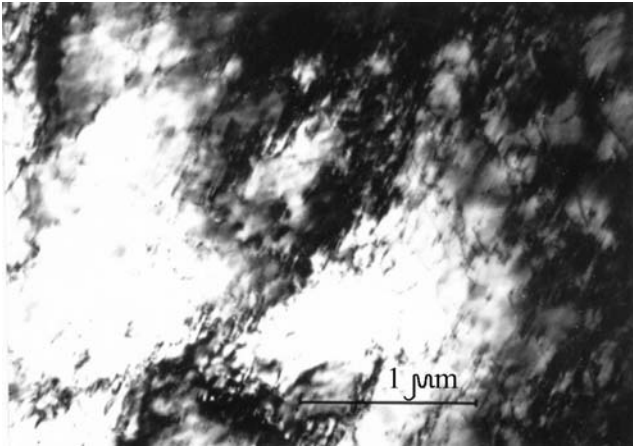
Fig. 12 Dislocation structure of low carbon steel on the yield plateau inside the material (the separate generated cells)

The change of dislocation structures in an intermediate zone (at incomplete deformation on the yield plateau), i.e. a zone which divides a site of a sample, which underwent strain Lüders-Chernov from that which did not, also was investigated. Investigating the intermediate site it was found out, that the edge of a strip of plastic deformation is related to rather sharp gradient of dislocation density. The size of the intermediate site is about two grains ($60 \mu\text{m}$). Hence, the distribution of plastic deformation in the sample on the yield plateau is going on by fast multiplication of dislocations in rather narrow zone in which the density of dislocations quickly achieves the certain size. New and new sites of the material subject to deformation are included in to the yield process, and this supports the constant level of acting stress. Also it is established, that on the yield plateau behind of strain front the average density of dislocations is practically kept constant.

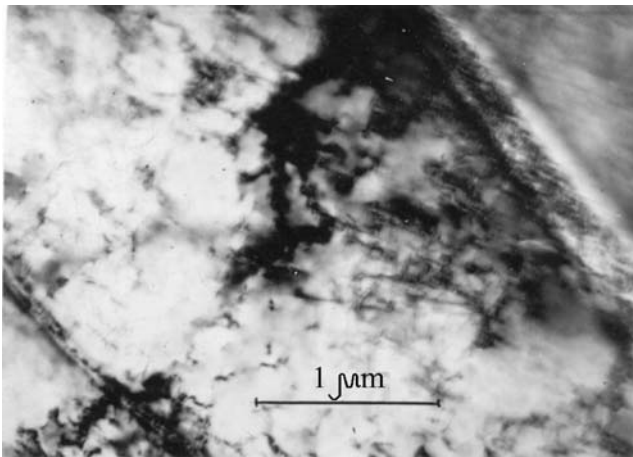
At the stage of strain hardening on all cross-section of the sample (Fig. 13) well advanced cellular dislocation structure is formed. In a microphoto of Fig. 13, a the formed cellular structure in the centre of the sample is seen, in Fig. 13, b – in near surface layer and in Fig. 13, c – low cellular structure at the surface of the sample is visible. The most effective formation of cellular structure is going on near to grain boundaries (Fig. 13, b). On the boundaries of cellular structures the dislocations are concentrated in irregular, chaotic heaps. The density of dislocations grows so rapidly, that often separate dislocations are not resolved any more. Besides dislocations inside the cells frequently settle down in such a manner that form bordering constructions and divide the cell into even smaller cells. The magnitude of cells at this stage in internal layers equals to $1.0\text{-}1.8 \mu\text{m}$, in near surface layer – $0.8\text{-}1.2 \mu\text{m}$, and at the very surface – $0.5\text{-}0.9 \mu\text{m}$.

Further increase of deformation degree up to σ_s (Fig. 1), results some further decrease of the size of cells in internal layers, and density of dislocations increase inside the cells (Fig. 14). Approaching to the surface (on depth of $160\text{-}200 \mu\text{m}$) in some grains the transformation of the cellular structure in to rectilinear congestions of dislocations (Fig. 15) is observed. Apparently, at this stage of deformation in near surface layer the number of active dislocation sources already decreases, and the plastic strain proceeds further basically due to the moving of previously formed

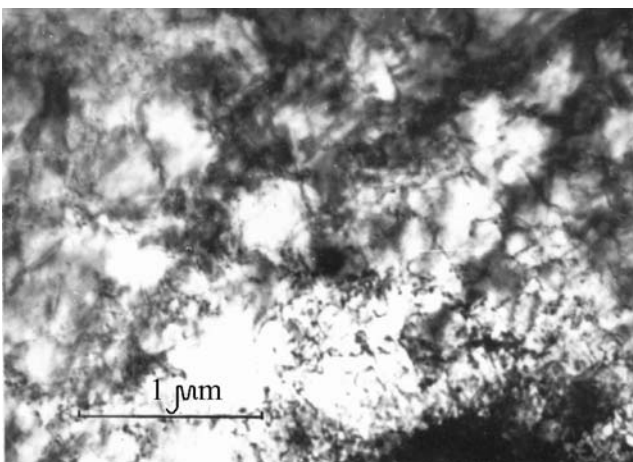
dislocations. On the depth of 20-40 μm from the surface both the cells and linear congestions of dislocations will be transformed in to band structure (Fig. 16). Bright subgrains, the density of a dislocation in which is rather insignificant, have undergone rather small strain – they have only moved in relation each to other, whereas the basic part of strain effect was concentrated in the areas dividing subgrains. These areas look like narrow strips which seem dark because of high density of dislocations in them.



a



b



c

Fig. 13 Dislocation structure of low carbon steel at the stage of strain hardening: a - in the centre of a sample; b - in near surface layers of a sample; c - at the surface of a sample

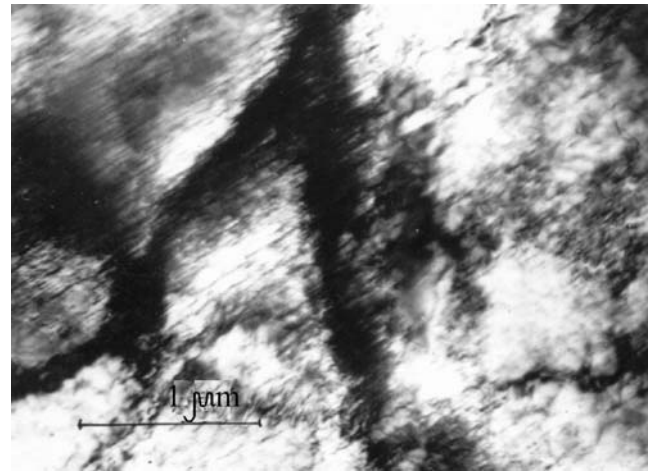


Fig. 14 Dislocation structure of low carbon steel at critical stress σ_s in internal layers

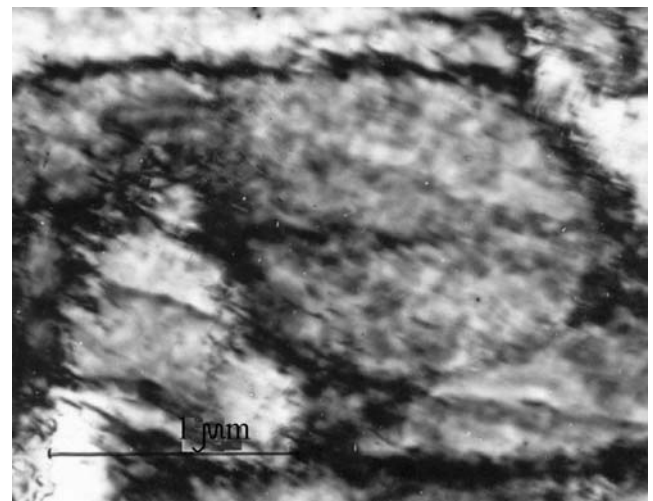


Fig. 15 Dislocation structure of low carbon steel at critical stress σ_s on the depth of 160-200 μm

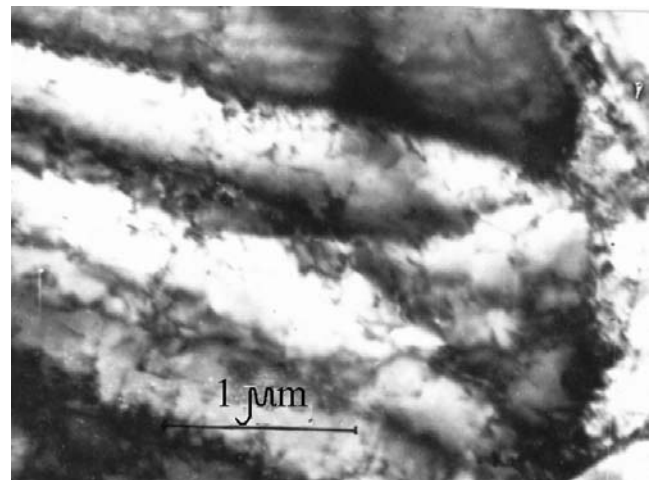


Fig. 16 Dislocation structure of low carbon steel at critical stress σ_s on the depth of 20-40 μm

At the surface and in near surface layer of a sample up to 10 μm the strongly pronounced banded structure (Fig. 17) is observed. These bands are not oriented each to other, what is seen from the change of contrast between the neighbouring strips. There is a sign-variable alternation of the contrast caused by sign-variable alternation of disorientations, which reduces elastic energy of a material.

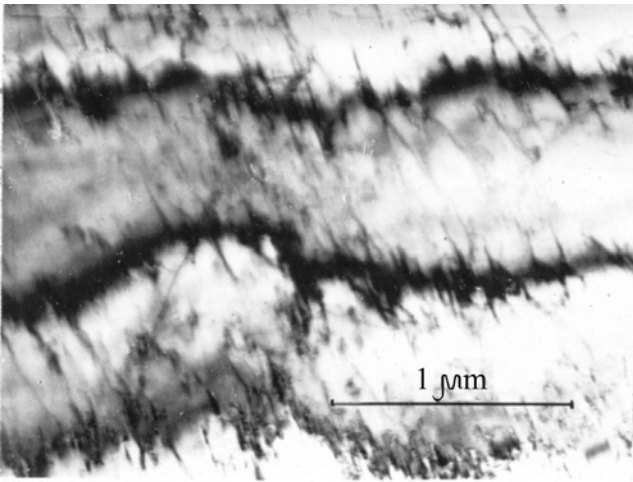


Fig. 17 Dislocation structure of low carbon steel at critical stress σ_s on the surface and in near surface layer on the depth of up to $10 \mu\text{m}$

At the stress $\sigma_s < \sigma < \sigma_U$ no essential change in dislocation structure does occur. In internal layers insignificant reduction of the cells size is noticed, and in near surface layer banded structure penetrates to the depth of $60\text{--}80 \mu\text{m}$ from the surface. At the end of strain hardening stage in some separate internal grains the newly formed linear congestions of dislocations may be noticed (Fig. 18). Transformation of these congestions in to the banded structure happens on the depth of $100\text{--}150 \mu\text{m}$ (Fig. 19). Thus, increasing the degree of deformation the difference in dislocation density in internal and in external areas decreases, however up to the fracture the density of dislocations in near surface layer remains higher.

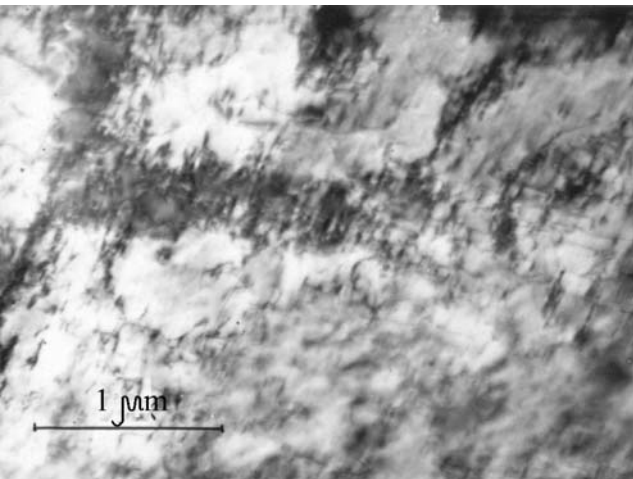


Fig. 18 Dislocation structure of low carbon steel at the stress $\sigma_s < \sigma < \sigma_U$ in internal grains

It is necessary to note, that already right at the beginning of strain hardening stage in local metal volumes the areas with critical dislocation density are formed, in these areas after certain degree of plastic strain submicrocracks about 100 nm arise. The stage of strain hardening finishes when the maximum load is achieved and the neck formation starts. The period of cracks propagation begins. Neck formation process is related to the further transformation of dislocation cellular structure with critical density of dislocations and pores initiation on walls of dislocation cells. Growth of pores and their subsequent coalescence

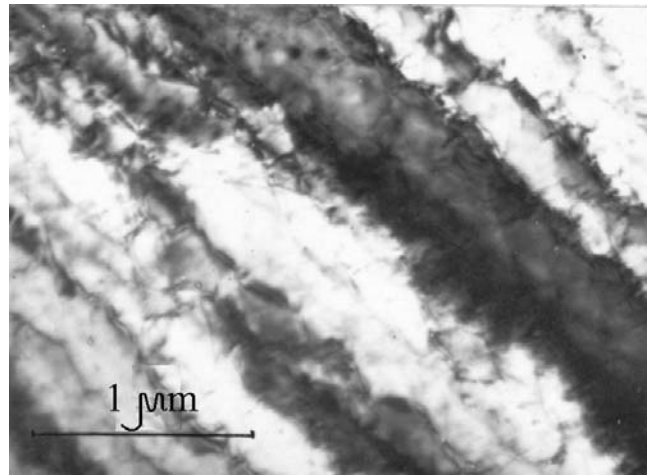


Fig. 19 Dislocation structure of low carbon steel at the stress $\sigma_s < \sigma < \sigma_U$ on the depth of $100\text{--}150 \mu\text{m}$

occurs in conditions of intensive plastic deformation. The sample finally fractures.

The analysis carried out by us using layer-by-layer electron microscopy has shown, that at all stages of deformation the character of an arrangement and density of dislocations essentially varies at moving away from the surface in to depth of the metal. At small degrees of deformation plastic flow of the metal is limited by near surface layer. At higher degrees of deformation, starting from the elastic limit σ_e , the internal layers of the metal are involved in to plastic flow also, however the increased density of dislocations in superficial area is kept, that, apparently, should render essential influence on common kinetics of macroscopic yield. Especially big difference between dislocation structures of near surface and internal layers of a material is observed in the field of the upper yield limit. Hence, distribution of dislocations in near surface layer of the deformed metal may not reflect processes of plastic flow of all metal as a whole, including its internal volumetric layers.

4. Conclusions

1. At monotonic tension of low carbon steel, the dislocations first of all are activated in the near surface layer.
2. Increase of the dislocation amount in the near surface layer is noticed on the stage of microyield, but in the internal layers it is noticed only on the yield stage. The most intensive increasing of dislocation density to the majority of near surface grains begins when the elastic limit σ_e is achieved.
3. Dislocations on the microyield stage in near surface and internal layers basically are focused on grain boundaries. Hence, the boundaries serve as obstacles for movement of dislocations.
4. The degree of strain development process in near surface layer from a grain to a grain is very much different at the stage of microyield. In some grains the traces of plastic strain are not noticed yet, and in others the process of sliding already proceeds on two or more systems.
5. The difference of dislocation density in internal and in external areas decreases in all other stages of deformation (yield, strain hardening), however up to the frac-

ture the density of dislocations in near surface layer remains higher.

6. Distribution of dislocations in near surface layer of the deformed metal may not reflect processes of plastic flow of all metal as a whole, including its internal volumetric layers.

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A. Čiuplys, J. Vilys, V. Čiuplys, V. Kvedaras

MAŽAANGLIO PLIENO DISLOKACINĖS STRUKTŪROS TYRIMAS STATINIO APKROVIMO METU

Reziumė

Tiriama, kaip keičiasi dislokacinės struktūros mažanglio plieno paviršiniame ir vidiniuose sluoksniuose statinio apkrovimo metu. Peršviečiamosios elektroninės mikroskopijos tyrimai parodė, kad visose deformacijos stadijose dislokacijų išsidėstymas ir jų tankis smarkiai ski-

riasi einant nuo metalo paviršiaus į vidinius sluoksnius. Esant mažiems deformacijos laipsniams, plastiškai deformuojasi tik paviršinis metalo sluoksnis. Didinant deformacijos laipsnį, platinė deformacija pastebima ir vidiniuose sluoksniuose. Ypač didelis paviršinio ir vidinių metalo sluoksnių dislokacinės struktūros skirtumas pastebimas takumo stadijoje.

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INVESTIGATION OF DISLOCATION STRUCTURE OF LOW CARBON STEEL DURING STATIC LOADING

Summary

Dislocation structure changes of low carbon steel in both surface and internal metal layers at static loading were investigated in this article. The analysis carried out by us using layer-by-layer electron microscopy has shown, that at all stages of deformation the character of an arrangement and density of dislocations essentially varies at moving away from the surface in to depth of the metal. At small degrees of deformation plastic flow of the metal is limited by surface layer. At higher degrees of deformation the internal layers of the metal are involved in to plastic flow. Especially big difference between dislocation structures of surface and internal layers of a material is observed in the field of the upper yield limit.

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ИССЛЕДОВАНИЕ ДИСЛОКАЦИОННОЙ СТРУКТУРЫ МАЛОУГЛЕРОДИСТОЙ СТАЛИ ПРИ СТАТИЧЕСКОМ НАГРУЖЕНИИ

Резюме

В настоящей работе приведено исследование характера распределения дислокаций малоуглеродистой стали по сечению образца при статическом нагружении. Проведенный послойный электронномикроскопический анализ показал, что на всех стадиях деформирования характер расположения и плотность дислокаций существенно меняется при удалении от поверхности в глубь металла. При малых степенях деформаций пластическое течение металла ограничивается приповерхностным слоем. При более высоких степенях деформации в пластическое течение вовлекаются и внутренние слои металла. Особенно большая разница дислокационной структуры в приповерхностных и внутренних слоях материала наблюдается в области верхнего предела текучести.

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