Rev. Adv. Mater. Sci. 4 (2003) 155-162

NANOCRYSTALLINE STRUCTURE FORMATION UNDER SEVERE PLASTIC DEFORMATION AND ITS INFLUENCE ON MECHANICAL PROPERTIES

S. Firstov, M. Brodnikovsky, M. Danylenko and Yu. Podrezov

Institute for Problems of Material Sciences of National Academy of Sciences of Ukraine,
3 Krzhizhanovsky Str, Kiev, 03142, Ukraine

Received: May 8, 2003

Abstract: The effect of equal canal angel pressure deformation on the stress-strain curves of high deformed materials was studied. The influence of the deformation degrees on dislocation and cell structure evolution was analyzed. The dominant features of nanocrystalline structure formation under severe plastic deformation were discussed. The structural sensitivity of the mechanical properties of high deformed Fe-Armco was studied.

* *Presented at NATO Advanced Research Workshop on Nanostructural Materials and Coatings for Biomedical and Sensor Applications, Kiev, August 4-8, 2002*

1. INTRODUCTION

The last decade has seen an important development that drastically changed the status of nanostructured materials as a branch of material science. Instead of being exotic and purely descriptive object they are increasingly resorting to very useful and carefully investigated type of structure. This achievement owns much to introduction of nanostructured materials as the main elements in super-tiny electronic plates. Undoubtedly, so-called functional materials and their unique electromagnetic properties have priority in such investigation. The wave of publications about mechanical behavior for this class of materials immediately followed as well.

According to physical theory of strength the dispersion of crystalline structure promotes essential rise of strengthening and improves low temperature plasticity. Moreover, microcrystalline materials can demonstrate superplasticity state at high temperature. The super high mechanical properties could be expected when extrapolating this tendency to nanocrystalline structures; however, as the amount of experimental data increases, it becomes obvious that only a few data confirm such assumption.

As a rule these are thin layers of films and coatings obtained by evaporation and spraying as well as near-surface deformed layers obtained by power milling or high speed friction treatment that demonstrate high strengthening of nanocrystalline structure. In this case the main mechanical tests are nano-, micro-, and Vickers- indentation. All these microtesting methods give limited information about plastic deformation and fracture mechanisms in these nanocrystalline materials under loading.

Severe plastic deformation is a more preferable method, which gives the possibility to create nanocrystalline structure in macroscopic volume. Traditional deformation methods (elongation, compression, ruling, draft, etc.) give such a possibility on very thin samples (thin foil or wire). In the last case the comparison of the mechanical properties of materials with different deformation degrees is hindered by the scale effect. The most careful investigation of Fe-Armco strain hardening at high degree deformation was carried out by Langford [1] on wire samples. The study of this object by electron microscopy showed nano dispersion cells structure which gives good prospects for strength and fracture toughness of high deformed materials.

2. EXPERIMENTAL PROCEDURE

Creation of new high deformation methods is promising both for development of a strain hardening theory and for deformed materials structure engineering. The method of equal canal angel (ECA) pressure was

Corresponding author: Yu. N. Podrezov, e-mail: podrezov@materials.kiev.ua

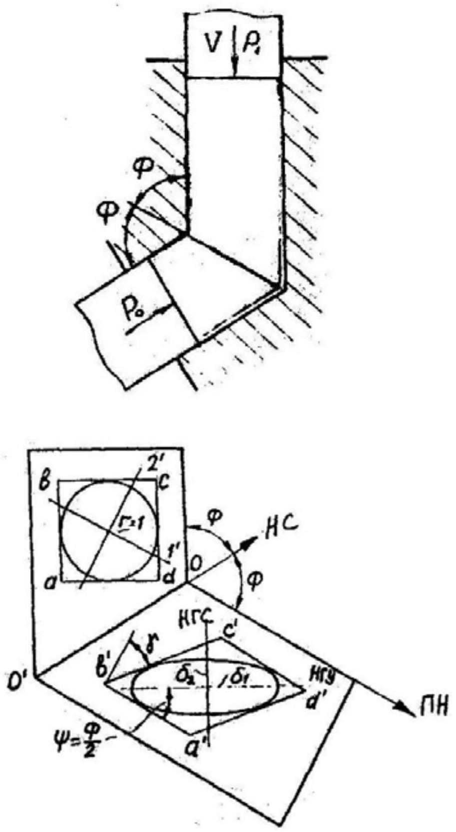

Fig. 1. ECAP scheme (a), ECAP process schematic showing billet shear (b), [2].

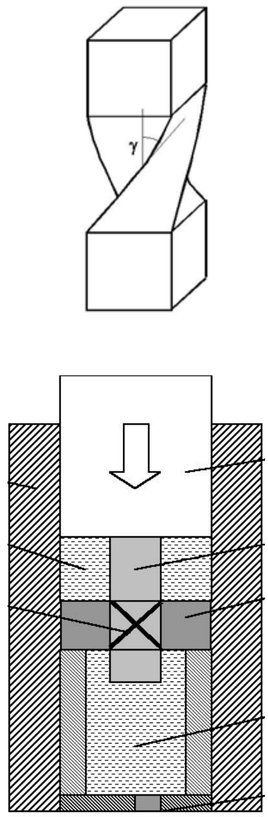

Fig. 2. a) Deformation by twist extrusion (TE). The form of the channel [3]; b) An installation scheme for hydro-mechanical extrusion.

created by Dr. Segal [2]. Simple shift uniform deformation of high intensity can be achieved on a macro sample 15x15x150 mm without changing its sizes (Fig.1). The other method based on deformation by a twist extrusion (TE) scheme was proposed by Dr. Beygelsimer [3] (Fig.2). The use of shift uniform deformation without change of sizes makes it possible to carry out repeated procedures in different (or the same) deformation directions and as the result to control the structure formation process at high deformation degrees.

AL-Mg-Sc alloys deformed by ECA - pressure method demonstrate the record value of superplastic deformation 2400% when speed of deformation 10^{-3} sec^{-1} is usual for superplasticity, and moreover keep the superplastic ability up to high speed deformation 10^{-1}-1 sec^{-1} [4]. After ECA - pressure deformation titanium has maximum strengthening (yield point is 800-900 MPa) that improves its application in medical branch [5]. Extremely high fracture toughness in high deformed Fe-Armco after (ECA) pressure was obtained in our previous works [6]. These results and some other experimental data [7,8] demonstrate high ability and good perspective of severe plastic deformation methods.

The achievement of high strength in deformed materials with nanostructure is considerably more complicated task compared with predictions of the microscopic strength theory. According to the models of this theory the strength of materials is connected with structural size dimension according to Hall-Petch equation: $\sigma_T = \sigma_0 + \varepsilon_y d^{-1/2}$ or Holt's variation for cell materials $\sigma_T = \sigma_0 + \varepsilon_y d^{-1}$. The extrapolation to nano scale of grain or cell size dimension theoretically predicts the very high strength of nano-crystalline materials. But experimental results demonstrate essentially worse situation. A. W. Tompson reviewed experimental data from variety of investigations [9] carried out on high deformed Fe - Armco wires. The relation between flow stress increment σ - σ_0 and substructure size is shown in Fig.3. In this graph we added our data obtained on Fe - Armco deformed by (ECA) pressure.

An important conclusion about interconnection between structural evolution and strengthening of high deformed materials follows from these results. Firstly, it is the change of strengthening mechanism sensitivity from grain size parameter (the Hall-Petch equation) to cell size parameter (the Holt's equation) at critical grain size d < 0,4 mm. Secondly, it is the theo-

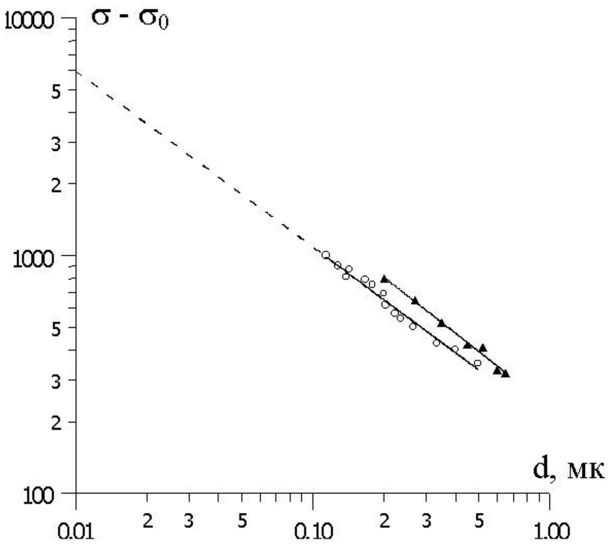

Fig. 3. Effect of cell size on yield point of high deformed Armco-Fe after ECA pressure (1) [6] and wires drawing (2) [1].

retical possibility to obtain super high strengthening in nanocrystalline Fe-Armco. It follows from experimental data (see the dashed line) that extrapolation of experimental data to nanostructure sizes (10 nm) gives the value of yield point for such material approximately 6000 MPa. But the third conclusion restricts such possibility. Difficulty is amplified due to the fact that cell or subgrain size in Armco-Fe obtained by different methods of severe plastic deformation (rolling, drawing, EC-pressure), can not be less than 100 nm. As the result, the yield point for such material is only 1000 MPa.

3. RESULTS AND DISCUSSION

Substructural Evolution

In order to explain the restriction of structural dispersion in deformed materials, careful investigations of deformed substructure by TEM were carried out. More important experimental work was published by Langford and Cohen, who investigated high deformed Fe-Armco wires. Practically the same results were obtained in our work [6] where severe plastic deformation in Fe- Armco was produced by rolling and ECA - pressure.

It was established on deformed Armco-Fe that the sells begin to form during deformation process at a strain of about 0.2 and tend to adopt the elongated ribbon shape with further reduction of dimensions. There are important departures from this behavior. Firstly, it is because of polygonization (formation of cell walls) which increases the number of sells. Secondly, it is because of dynamic recovery (via cell wall migration) which decreases the number of cells. Both of these phenomena prevent the wide – thickness ratio of the cellular cross section. As it is known [10] the cells in deformed iron start with equiaxed shapes and then become ribbon-like in cross section under deformation.

One can write the following expression for *f*, the fraction of the initial number of cells in a cross section at any given deformation power:

$$f = \frac{N}{N_1} \exp\left[-(e - e_i)\right], \qquad (1)$$

where N_i is the initial number of cells per unit cross-sectional area as formed at some early strain e_i and *N* is the number of cells per unit cross-sectional area at some subsequent strain *e*. Measurements of *N* have been made during the deformation and corresponding values of *f* vs *e* are plotted in Fig.4. As it can be deduced from Fig. 4, *f* is near unity both for wire drawing and for rolling deformation under low and middle deformation degrees. This result is in a good agreement with Tailor-Pallany law: both bulk material and structural elements (grains, cell) have the same shape under deformation. Since ECAE is a process capable of producing plastic deformation without causing substantial change in geometric shape of billet, cell size of ECAE deformed iron at the middle deformation degrees is substantially higher.

Under high deformation power the principal change in *f* vs *e* relation takes place. There is the large decrease in *f* throughout the subsequent elongation, signifying that substantial dynamic recovery is operative during the cellular refinement. This means that many

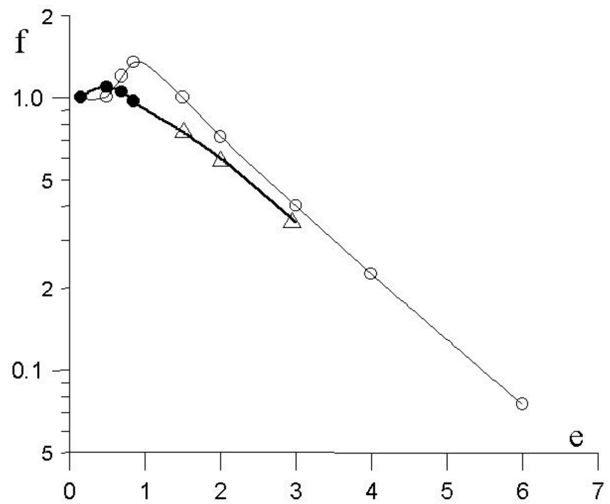

Fig. 4. The dependence of *f*-parameter vs. deformation degree: • – rolling, Δ – ECAP, o – wire-drawing [1].

cells are being lost from the structure simultaneously with cross-sectional reduction of the remaining cells. It is clear that cell wall migration constitutes very important aspect of the structural changes and the critical deformation degree e_c at which the change in f vs e relation takes place is a very important parameter.

Cell size evolution under deformation is accompanied by increasing of cellular misorientation [11]. A series of histograms were combined showing the distribution of cellular misorientations as a function of deformation. It was noted that distribution of misorientations Φ widens under plastic deformation and its average value rises with the strain increase. Our experimental results are in a good agreement with Φ vs e relation.

According to Tompson's [9] model of structural evolution, static and dynamic recovery are the main reasons limiting the minimal size of structural elements under deformation in high deformed materials. From one hand, many cells are being lost from the structure during this process, from another hand, the recovery process promotes boundary perfection and increases misorientation of cells. But this model cannot explain the existence of critical deformation degree e_c, which characterizes the beginning of significant activation of this process.

The disclination model of severe plastic deformation is proposed by Rybin [13]. This model is based on analysis of micromechanical stress arising around a dislocation during plastic deformation. According to this model plastic deformation in crystals is developed by moving of dislocation at a low and middle deformation. But after transformation of substructure from dislocation forest to cells at $e_i \sim 0{,}2$ the change of deformation mechanism takes place and severe deformation is evoked by generation of disclination modes without diminution in subgrain size.

Theoretical calculation within the framework of the disclination model gives the critical cell size ~ 0,2 μm for BCC metals. As far as the critical cell size depends on some physical constants (Burgers vector, elastic modulus, fault energy), for some FCC metals its value is essentially lower. It is in a good agreement with last experimental data [4,14] obtained on Ni and Al deformed by ECA pressure which show that the structural element average size was 30-50 nm. The disclination theory predicts monotonic rise of misorientation with the increase of deformation power and does not imply any special features of mechanical behavior of high deformed materials after the critical deformation e_c.

V.I Trefilov *et al.* [15] demonstrated such features of mechanical behavior in high deformed refractory BCC metals under testing at brittle-ductile transition temperature. The sharp rise of plasticity and decreasing of brittle-ductile transition temperature was observed. These effects were explained in a framework of Reads theory of critical cell misorientation [16]. According to this theory the stress τ, which is necessary to overcome the cell boundary by dislocation slipping depends on the angle of misorientation Φ_p and may be determined from an equation

$$\tau = \frac{\Phi_p G}{2\pi}, \tag{2}$$

where G is the shear modulus.

For $\Phi_p = 3^{\circ}$ the cell boundary is a strong barrier for slipping. Such boundaries are formed in substructure of deformed BCC-metal after critical deformation degrees [11,12]. Emergency of these defects changes mechanical behavior of deformed materials. It was proposed in [15] to correlate the change of mechanical properties of high deformed materials with the change of the effective cell size d_{eff} of structure formation which are bounded by high angle structural defects. Then $d_c < d_{eff} < d_g$ and this structural parameter decreases with the increasing of the strain degree.

Structural sensitivity of mechanical properties

We observed the change of mechanical behavior near the critical deformation e_c in high deformed Fe-Armco after ECA pressure [6]. The growth of the fracture toughness for specimens with cracks introduced into the plane perpendicular to the plane of deformation was shown. At the same time the decrease of the fracture characteristic for specimens with cracks introduced into the plane parallel to the plane of deformation was observed (Fig. 5). The increase of the

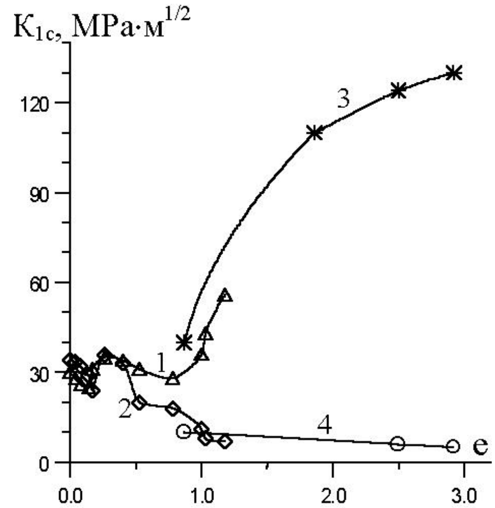

Fig. 5. Fracture toughness of strained iron. 1, 2 – rolling, 3,4 – ECAP (1,3 – crack introduced into the plane perpendicular to the plane of deformation; 2,4 – crack introduced into the plane parallel to the plane of deformation).

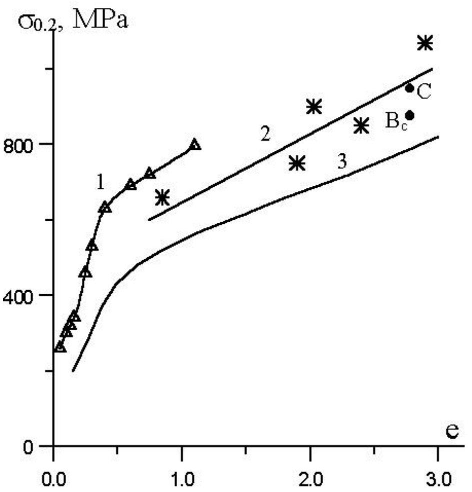

Fig. 6. Effect of strain on yield point of rolled (1), ECAP (2) materials and drawn wires [1] (3).

deformation degree ($e > e_c$) promotes the change of the failure mechanism as follows: quasi-cleavage → quasi-cleavage with delamination. On the contrary, transition from the parabolic stage of strain hardening to the linear one is not related with the change of the deformation mechanism. Therefore the processes occurring near the critical deformation e_c are very important and must be investigated more carefully.

Both the recovery model and the disclination theory give very pessimistic predictions for usefulness of the methods of intensive plastic deformation for further decreasing of subgrain size up to several nanometers. However, a lot of experimental works [17-20] demonstrate nanostructure in deformed near surface layers obtained by power milling or high speed friction treatment with grain size 1-10 nm. Dispersion of substructure and strengthening of near surface layers take place after dynamic treatment by ultrasonic or the multiple time damping process [17]. Such layers have the very high level of microhardness.

It was shown by Yurkova and Belotsky [20] that high speed friction of Armco-Fe which occurs simultaneously with surface blasting by ammonium gas results in formation of nanoctructural layers (subgrain size 3-5 nm) with 200 μm in depth and H_μ = 13000 MPa (the yield point is approximately 4000 MPa) (Fig.6). X-ray analysis of the layer shown high content of nitrogen in solution and in phase. The same strong dispersion of structure (d=5nm) was observed by Lapunov and Dzubenko [21] after milling of clean fine-fraction iron powder (containing 0,2 mas % of oxygen) during 60 h in vacuum. In this experiment the nanostructured state was kept after consolidation of powder by the cold pressing method. But sintering treatment at 1100 °C promotes recrystallization of nanostructure. After finishing treatment this material had grain size 30 μm and 10% particles of Fe_2O_3. Probably, added oxygen has penetrated inside Fe powder through the free fine powder surface in spite of vacuum protection.

It is necessary to take into account two main reasons to explain such high dispersion of substructure. Firstly, the metallic surface layers obtain much more deformation under repeating intensive dynamic effects than after any static deformation methods. Secondly, near-surface deformation occurs in the presence of atmospheric air or special gas which penetrates into the material. Interaction between dislocation and added atoms slows down the recovery process and as the result diminishes subgrain size. This mechanism has an autocatalytic nature: structural dispersion accelerates diffusion of addition element in the depth of metal and rises its consternation facilitating structural dispersion.

We must emphasis the important role of deformation temperature in the process of structure evolution. Since dynamic deformation is accompanied by sample heating, there exists optimal temperature condition when a maximum of structural dispersion appears. It follows from [20] that in friction-deformed Armco-Fe the dispersion effect sharply increases when the test temperature is more than 300 ^{0}C. This is the temperature of dynamic deformation ageing when dislocation velocity and velocity of diffusion for additional elements are approximately equal.

The noted features of structure formation in deformed materials predetermine regularities of designing their mechanical properties. Analysis of strengthening curves for a deformed material is usually based on structural changes occurring in the material at its continuous loading. Thus, an assumption can be made that each next structural condition is obtained by an evolution of previous one, and the change of deforming stress is a consequence of the evolution. Structure sensitive models of strain hardening are described in details by many authors [16, 22-24].

Practically the same approaches are used to analyze structural sensitivity of mechanical properties of pre-strained materials. The known postulate of mechanics that deforming stress being achieved in a material at repeated deformation corresponds exactly to deforming stress achieved in it at unloading moment at initial deformation is widely used. This approach that was used by Longford [1] for analysis of hardening curve of high-deformed iron wire (Fig.6).

This postulate does hold well in range of low and middle deformation degrees for materials which have not susceptibility to a recovery process. For example, it is verified for Al-Ti-Cr intermetallic specimens in our experiments [25] under uniaxial compression tests, Fig.7a. But this postulate does not hold for materials which have inclination to recovery, in par-

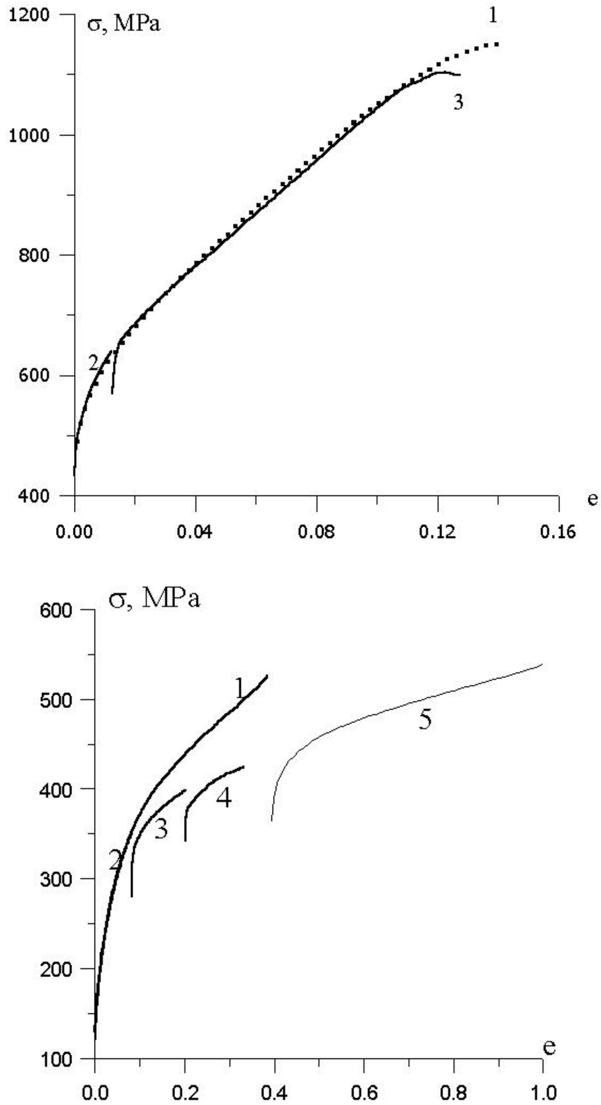

Fig. 7. a) Stress-strain curves of Al-Ti-Cr alloys (1,2 – continuous deformation, 3 – repeating deformation); b)Stress-strain curves of clean Al (1,2 – continuous deformation, 3-5 – repeating deformation).

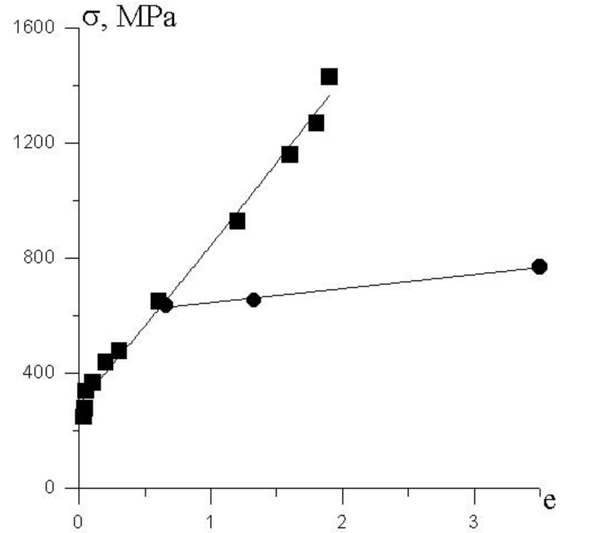

Fig. 8. Stress – Strain curves for Ti: (■) – tension, (●) rolling.

ticular such results were obtained on aluminum (Fig.7b). The strain hardening curve obtained by single loading compression test (Fig. 7b, curve 1) was compared with curves obtained after repeated deformation (the second deformation – curve 2, the third deformation – curve 3, and the forth deformation – curve 4). Experimental data show that pre-strained materials have both lower yield point and lower strain hardening coefficient than it follows from the mechanic postulate.

Unfortunately, due to the fact that conditions of uniaxial deformation are limited by friction, size effects under compression, and by a neck formation process under tension it is impossible to verify the postulate at high deformation powers (more than *e*=0,4). Therefore, in order to obtain the strain hardening curve of high-deformed aluminum in unified loading condition we have made repeated grinding of lateral faces of a specimen deformed by single compression. As the result the specimen was put into rectangle shape and tested by repeated compression. This strain hardening curve (curve 5) lays noticeably lower than that for single compression (curve 1) but its start position coincides with the end of last curves obtained by repeated compression (curve 4). Since a summary hardening curve of predeformed materials could be obtained by mating results of consecutive loading cycles, but because of a recovery process its strain hardening coefficient may be essentially lower than that obtained under continuous loading.

We obtained similar effects on titanium specimens [26]. Strain hardening curves obtained by uniaxial tension (subject to neck formation) and data of hardening of the material after high deformed rolling were compared (Fig. 8). In this case the hardening is stronger essentially for the specimens tested in continuous loading than that for the specimens tested in reloading of pre-deformed material. In particular, failure stress 1600 MPa is achieved under uniaxial tension of pure titanium. In high deformed rolled titanium the stress never exceeds 600-700 MPa. Undoubtedly a difference of both loading conditions and specimen texture should be taken into account in this case. However, only these factors in any case do not explain essential difference in titanium properties in conditions of continuous loading and pre-deformation states.

Larikov [27] investigated the dependence of hardness HV on deformation degree for deformed single

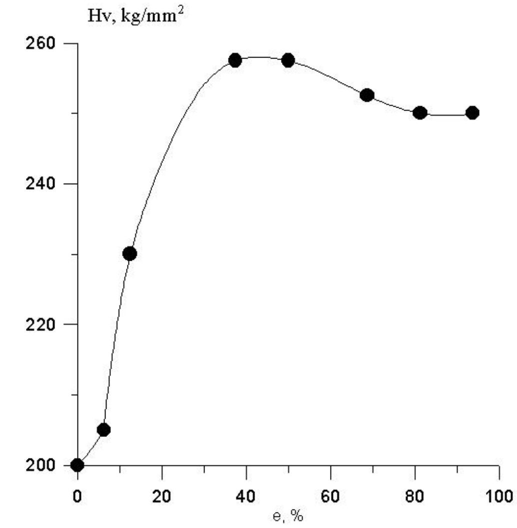

Fig. 9. Dependence of hardness HV on deformation degrees of molybdenum single crystals.

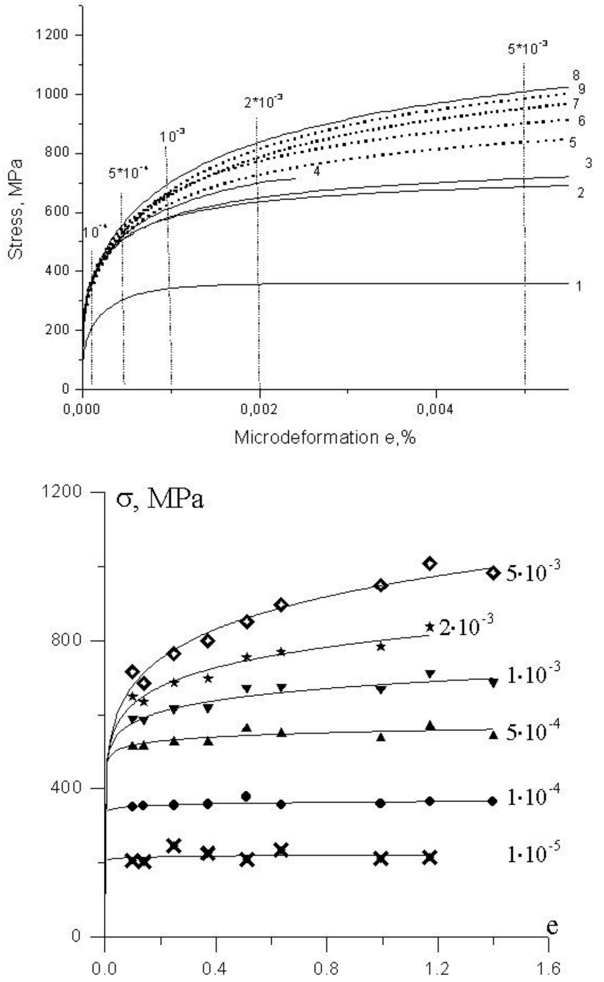

Fig. 10. a) Microplastisity curves of molybdenum deformed by rolling up to different deformation degrees 1 initial, 2 – 9%, 3 – 13%, 4 – 23%,5 – 39%, 6 – 53%, 7 – 63% 8 – 68%, 9 – 73%; b) Stress prestrain curves for different plasticity levels.

crystals of molybdenum. Diminution of hardness at high deformation degrees (Fig.9) accompanied by smearing of diffraction patents was observed. He explained the recovery process by the increasing of mobility of screw dislocation components and as the result by activation of cross slip in a plastic deformation mechanism. The processes of structural relaxation proceeding by recovery mechanism both during plastic deformation and during unloading seems to be the most important factors to promote loss of strengthening in high deformed materials.

We analyzed micro-plasticity curves of pre-deformed rolled molybdenum specimens (the range of deformation degrees 9-75%). It was found (Fig. 10a) that at the initial stage of repeating deformation micro-plasticity stress-strain diagrams are insensitive to pre-deformation degrees (and therefore, to structural state of deformed materials).

In the case of micro-deformation (at 10^{-5}, 10^{-4} and $5{\cdot}10^{-4}$ deformation degrees) (Fig. 10b) deformation stress is practically the same for all degrees of predeformation of investigated materials. Differences become noticeable at 2Ч10^{3} (the yield point). And only at comparatively high repeating deformation degree 5Ч10^{-3} the stable increase of stress is observed with the rise of deformation. It is obvious that hardening curves obtained on micro-deformation levels can not be explained within the scope of the traditional structural model. At the initial stage of repeated deformation the recovered cells structure interacts with moving dislocations by special way.

These data are important for creation of an adequate model of mechanical behavior of high-deformed materials, and may be useful in explaining of the anomalous dependence of the fracture toughness on deformation power of materials tested in ductile-brittle transition temperature region (Fig. 5). In this case the laws of interaction of dislocations being emitted from a crack tip with primary structure under conditions when the number of the dislocations is not too large should be accounted for.

4. CONCLUSIONS

The possibility of obtaining super high strengthening in nanocrystalline Fe- Armco follows from extrapolation of experimental data to nanostructure sizes. For grain size 10 nm such extrapolation gives the value of material yield point approximately 6000 MPa. But cell or subgrain size in Armco-Fe obtained by different methods of severe plastic deformation (rolling, drawing, ECA-pressure), can not be less than 100 nm. As the result, the yield point for such material is only 1000 MPa. Static and dynamic recovery are the main reasons limiting the minimal size of structural elements under deformation in high deformed materials.

The critical deformation degree e_c characterizes the beginning of considerable activation of the recovery process. The change of mechanical behavior near the critical deformation e_c was observed in high deformed Fe-Armco after ECA pressure. The growth of the fracture toughness for specimens with cracks introduced into the plane perpendicular to the plane of deformation was shown, and the decrease of the fracture characteristic for specimens with cracks introduced into the plane parallel to the plane of deformation was observed. The increase of the deformation degree ($e > e_c$) promotes the change of the failure mechanism as follows: quasi-cleavage $\rightarrow$ quasi-cleavage with delamination.

The postulate of mechanics that deformation stress being achieved in a material at repeated deformation corresponds exactly to deformation stress achieved in it at unloading moment at initial deformation does hold well for materials which have not susceptibility to a recovery process. But this postulate does not hold for materials which have inclination to recovery. Structural relaxation proceeding by the recovery mechanism both during plastic deformation and during unloading causes loss of strengthening in high deformed materials.

At the initial stage of repeated deformation the recovered cell structure interacts with moving dislocations in a special way. In the micro-deformation level hardening stress is practically independent on previous deformation degrees in wide interval of deformation. The usual increase of strengthening with rise of deformation is observed only from the yield point.

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