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Developing nanostructures and enhanced properties in heavy W alloy through SPD processing.

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by

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ABSTRACT

The ways of fulfilment of severe plastic deformation (SPD) processes by equal channel angular (ECA) pressing of hard-to-deform low-ductility materials, namely W of the commercial purity and W alloy of W-Ni-Fe system were developed. To obtain ultra-fine grained (UFG) structures in these materials the dies for high-cycle ECA pressing were fabricated.

For the first time ECA pressing was implemented and the optimal regimes of obtaining of bulk commercially pure (CP) W ingots with UFG structure were established. First experiments of ECA pressing of heavy alloy W-4.9%Ni-2.1% Fe were conducted.

TEM and X-ray investigations of the SPD-processed CP W showed that the refinement of the structure up to a mean grain size less than 1 μm takes place during ECA pressing. The grains have mainly high angle boundaries that results in enhanced mechanical properties. Hardening of the investigated material during SPD was indicated by microhardness measurements.

To attain further progress in refinement of W-alloy structure by ECA pressing and due to this increase their mechanical properties new ways were suggested.

Keywords: *severe plastic deformation, ultrafine grained materials, tungsten and tungsten alloy, equal-channel angular pressing, microstructure refinement, texture, microhardness, mechanical properties.*

1. INTRODUCTION

Strong refinement of microstructure and formation of ultrafine-grained nanostructures in metallic materials by SPD provide a potential to achieve their novel and extraordinary properties [1-3]. However, the fabrication of such homogeneous structures in bulk samples and ingots is a complex problem, which depends on different SPD processing parameters. For example, ECA pressing which is a most widely used SPD technique to produce bulk UFG structures in different metals and alloys possesses a number of different variable parameters such as processing routes, temperature, strain and strain rates, etc. which determine the character of the developed microstructure and as a result the level of mechanical properties. Therefore, it is important to analyse the relationship: processing-nanostructure-new properties in ECA pressed materials. Currently the most detailed analyses have been made for originally ductile metals (copper, nickel, aluminum) and a few alloys [2-8]. The processing features of ECA pressing which leads to the formation of UFG structures with high angle grain boundaries has been established for these materials. The characterization of the microstructure formed and properties developed demonstrated a significant increase in strength and ductility.

The significant progress in controlling the microstructure and properties of SPD ductile materials made it possible to start investigations relating to the refinement of microstructure, and improvement of mechanical properties in low ductile and hard-to-deform materials. However, the successful solution to this problem depends on a number of special requirements related to the processing of bulk low ductile materials without failure and endurance of the special die to be used for ECA pressing.

The current report presents the results of first experimental investigations demonstrated the successful refinement of microstructure in bulk CP W and W alloy ingots by ECA pressing.

2. MATERIAL AND EXPERIMENTAL PROCEDURES

A rod, 16 mm in diameter, made of CP tungsten was used for the investigations. The rod was produced from a commercially sintered ingot by drawing and further swaging in the temperature range 1500-1600° C using intermediate annealing in hydrogen at the temperature 1600° C. The interstitial impurity content complied with requirements for engineering grades of W and was determined to be: O - 0.0094, N - 0.00066, C - 0.0134 (mass %) with an error of measurement for oxygen and nitrogen of ±1.0%, and for carbon 0.5% from the measured values respectively.

The heavy W alloy was delivered for investigations as a turned rod with diameter 17.5 mm and length 300 mm, chemical content was determined to be 93%W-4.9%Ni-2.1%Fe. The interstitial impurities should be less than 100 ppm (by weight)*. The alloy is the representative of the composite alloys of W-Ni-Fe system, obtained by powder metallurgy [9]. The sample was subjected to cold deformation (swaging) with 18% rate after sintering.

ECA pressing die was installed on a 160 ton-force capacity hydraulic press. The deformation rate, as measured by the movement the cross-head and punch, was 6 mm/s. To decrease the oxidation of the tungsten during heating and for reducing the frictional coefficient, the tungsten billet was placed into a cylindrical steel shell (canned). The coarse grained cylindrical W billets were 16 mm in diameter and 60 mm in length.

Structural investigations were conducted using optical microscopy (NEOPHOT-32) and TEM (JEM-100B). Chemical etching of polished metallographic specimens using a 3% solution of hydrogen peroxide revealed the microstructure of the worked tungsten. Foils for TEM examination were prepared by thinning in aqueous solution of sodium hydroxide using a cathode voltage of 20-30 V. The mean grain size was estimated

* Data of the US Army research laboratory.

by the secant method in transverse and longitudinal sections of samples on the basis of measurement of no less than 40 grains.

X-ray diffraction analysis investigations were carried out on the DRON-4-07 apparatus. The accuracy of the measurement of diffraction angles was 0.005°. Values of coherent scattering domain size and elastic microdistortion of the crystal lattice in the processed tungsten samples were determined by the harmonic analysis method using pairs of X-ray peaks (110) and (220). The relative error of measurement of the values of coherent scattering domain size and elastic microdistortion was less than 2%. Data for texture analysis were obtained by x-ray diffractometer DRON-3M equipped with an automatic texture attachment with focusing scheme of goniometer according to Bragg-Brentano.

Microhardness was measured in transverse and longitudinal sections of the samples under the load 1.96 H on the basis of measurement of ten indentations with an allowable error of 0.5%.

3. SPD PROCESSING

ECA pressing for processing UFG structure takes advantage of repetitive pressing of a bulk billet of a round or square cross section through two identical channels usually intersecting at an angle $\varphi = 90^{\circ}$ (Fig. 1). Shear deformation in the area (region) of intersection of channels is approximately equal to a true strain of 1.15 for this angle [2,4]. Depending on the deforming material the process of ECA pressing can be accomplished either at room or elevated temperatures.

As a rule, to develop UFG structure during ECA pressing the number of passes of a billet is 8 or more. Strong refinement of microstructure occurs in the first few passes. A cellular structure with low angle grain boundaries is formed on these passages and with increasing of passages number this structure gradually transforms to UFG structure with high angle grain boundaries [2,3,8]. The change in billet orientation between passes, performed by rotating a billet around its longitudinal axis is very important. Such rotations change the orientation of the plane of deformation by simple shear and the character of material plastic flow. They determine the character of the forming microstructure and level of mechanical properties. This, in turn, will determine the nature of the microstructure formed and the level of mechanical properties. The following routes are designated: route A (no rotation), route B_a (rotate 90° clockwise and counterclockwise alternatively), route B_c (rotate 90° clockwise), route C (rotate 180°) [7,8].

Tungsten is typical of hard-to-deform, low ductile materials at room temperature that become ductile only at temperatures exceeding 1000-1200° C [10]. However, at elevated temperatures the tungsten is prone to intense oxidation and requires heating in non-oxidizing environments. As a rule, the temperature range of plastic working to give high ductility and processing without failure is not lower than 1400-1600° C. However, these temperatures are higher than temperatures observed for recrystallization and cannot be used for processing a UFG structure in tungsten. Moreover, the process of ECA pressing of tungsten requires solution of questions relating to the resistance of the die material to high temperature, wear and the high applied pressure. Thus, it is absolutely obvious that ECA pressing fulfillment of tungsten is a very complex task requiring development of special approaches for the solution of the scientific and technical problems identified above.

First of all, we should note that for processing tungsten billets without failure special dies were designed and manufactured. Since tungsten has low ductility at temperatures close to 1000° C, the angle of channel intersection in the die was increased from an angle of 90° typically used for ductile materials for this work. The determined solution was confirmed by attempts to conduct ECA pressing with an angle of channels intersection of 90°. In this case billets failed on the first or second pass (Fig. 2). Changing the angle of channel intersection from 90° to 110° a shear strain value for one pass of the ECA pressing of tungsten was reduced from e=1.15 to e=0.8 and the total accumulated strain value for eight passes was e=6.4.

While developing the ECA pressing process for tungsten at approximately 1000° C two routes with different billet rotation, namely route B_c and route C, were selected. These routes provide formation of equiaxed structures in originally ductile materials. The conducted investigations have shown that only route C can provide billet processing without failure in case of CP W (Fig. 3). This is attributed to the fact that the optimal mode of deformation for the last one is realised at processing by route C.

Considering the positive influence of a low strain rate on the uniformity of material flow [11], the process of ECA pressing was conducted at the minimum strain rate at which the equipment could be used; namely 6 mm/s. With an aim of preventing oxidation of the tungsten and improving tribological conditions at high temperature the billets to be deformed were placed into a sealed steel shell (canned).

The first stages experiments on ECA pressing of heavy alloy W-4.9%Ni-2.1%Fe didn't bring the desired results. Due to low workability of this alloy the ingots failed even after the first pass at temperature 1000° C and intersecting angle 90°. In this connection a special die with angle of channel intersection equal to 120° was fabricated to decrease the strain rate. That decreased the strain rate by a factor of two in comparison with the variant when the intersecting angle was 90° and provided true strain during one pressing cycle e=0.55. Due to this die improvement the heavy alloy samples didn't fail after one cycle of ECA pressing. However the second further pass resulted in ingot failure. Temperature increase to 1100-1150° C didn't bring the expected results of successful ECA pressing with two and more passes. Unfortunately one failed to conduct ECA pressing at these elevated temperatures at the available die. One can draw a conclusion that a subsequent work should be presented by complex investigations of factors influencing the workability of heavy alloy at temperatures higher

than 1100° – 1150° C and fabrication of a die enable pressing process within the temperature range up to 1300° C with a possibility to control a process of the deformed state of the ingots at ECA pressing.

4. MICROSTRUCTURE REFINEMENT

4.1. COMMERCIAL PURE TUNGSTEN

The optical microscopy of the initial material confirms the presence of recrystallized grains in the microstructure having a grain diameter of 60-80 μm (Fig. 4a). In the longitudinal section the grains are elongated with the long axis parallel to the axis of the ingots. TEM studies show the presence of a well-formed polygonized dislocation substructure inside of coarse grains. This substructure has low density of lattice dislocations and the size of its subgrains is about 2-4 μm (Fig. 4b).

ECA pressing of the tungsten conducted by Route C, i.e. with a rotation of a billet between subsequent passes of 180° around its longitudinal axis led to significant refinement of microstructure but its character essentially depends on the number of passes. Fig. 5 presents the microstructure of the tungsten (observed by optical microscopy) after 4 passes of ECA pressing. This structure is characterised by a great number of newly formed fine grains and subgrains, but still a certain non-uniformity exists there. In some parts of the longitudinal section an elongated grains remain, but they are strongly fragmented with curved boundaries. In the other parts of the longitudinal section one can observe areas with practically equiaxed structure. At the same time, TEM studies indicate the formation of a developed substructure (Fig. 5b). The given substructure is significantly different from the substructure in the initial tungsten. Firstly, the subgrain size after ECA pressing is within the range 0.7-1.5 μm, i.e. significantly less. Secondly, high angle misorientations occur in this structure. This is confirmed by the diffraction pattern (Fig. 5b) in which one can see the appearance of a number of spots arranged in circles and their spreading, indicative of the presence of high internal elastic stresses in the microstructure.

ECA pressing with 8 passes not only leads to some further refinement of the microstructure but also increases its uniformity. As seen from Figs. 6a and 6b representing results of optical microscopy investigations, after such processing there occurs formation of an UFG structure which is equiaxed and sufficiently homogeneous both in transverse and longitudinal sections of the billets. TEM examination confirms the formation of the UFG structure after 8 passes of ECA pressing. The mean grain size is about 1 μm, though in the structure one can see the appearance of a number of finer grains, 0.3-0.5 μm in size (Fig. 7a). It is interesting that the majority of new grains are free of dislocations and the character of the diffraction pattern confirms the majority of grains have high angle grain misorientations. At the same time the examination of the longitudinal section of the billet indicates that grains are a bit elongated (Fig. 7b).

The X-ray studies indicate a significant broadening of diffraction peaks after ECA pressing which points to the refinement of the initial microstructure and the increase in elastic microdistortions of the crystal lattice. Moreover, ECA pressing leads to the formation of texture maxima corresponding to ideal {111}<121>, {001}<110> type orientations. The formed texture is close in its appearance to the texture of the CP tungsten subjected to cold rolling [12].

4.2. HEAVY TUNGSTEN ALLOY

As it was mentioned above, the tested alloy was obtained by powder metallurgy. Usually burden, which consists of powder mixture in required proportion, is pressed after thorough stirring in the hydraulic press under the applied pressure 1-2 tons/cm^2. Then they are sintered in the hydrogen medium at temperature 1500±20° C. During sintering more fusible Ni-Fe powder mixture creates a liquid phase in which tungsten is partially dissolved. Ni and Fe has a good mutual solubility. After solidification a Ni based solid solution interlayer is formed around tungsten particles, that provides high alloy density and plasticity. Processes of recrystallization and coalescence of certain W grains resulted in growth of tungsten grains during process of sintering [13].

According to the phase diagram of state [14] figurative dot of the testing alloy is situated in the area of two-phase α (W) + γ-phases, where α is a tungsten based solid solution, γ is a solid solution of Fe and W in Ni. Investigations of W alloy by optical microscopy in the transverse section showed that the structure consists of equiaxed spherical particles of W powder (α phase) with sizes within the range 10 - 100 μm ($d_{average}$ = 32.5 μm), surrounded with γ-phase interlayers of solid solution from 1-2 to 30-35 μm in width. At that γ-phase occupies not more than 20% of metallographic section square (Fig. 8a). In the longitudinal section of the rod microstructure is the same as described above.

ECA pressing of heavy W alloy in the die with channel intersecting angle of 120° led to small microstructure refinement. At that, features of dislocation activities as curved strain band are observed in some tungsten grains (Fig. 8b). TEM investigations showed that in case of insignificant dislocation density and substructure absence in recrystallized grains of initial W rod, as it was in CP W, dislocation density noticeably increases after one pass of ECA pressing. Fine structure of γ solid solution interlayers surrounding W grains (α-phase) after one pass ECA pressing is presented on Fig. 9. Very fine grains with sizes from 10 to 50 nm are observed in the structure at high magnification. Microdiffraction pattern also indicates a great γ-phase refinement: a multitude of small reflections arranged in the circles.

5. MECHANICAL PROPERTIES

Microhardness investigations of Vickers type showed that testing materials are appreciably strengthened after ECA pressing. After 8 passes values of microhardness have achieved 6100 MPa as compared to 4500 MPa in the initial state.

Recent investigations indicate that microstructure refinement by SPD in such low ductile materials as intermetallics enable to increase significantly not only strength but also plasticity [1,2]. However the increase of plastic properties requires greater refinement of microstructure up to grain size 0.5 μm. Such approach could be rather perspective both for W and W alloys. The increase of strength and plastic properties is very actual.

Microhardness of coarse grained W alloy was about 5640 MPa but after one pass of ECA pressing it amounted to 6445 MPa. It should be mentioned that microhardness of the testing alloy in the initial state 550-600 MPa higher than typical values of microhardness in CP W. After one pass of ECA pressing alloy microhardness growth double the amount than in CP W.

6. DISCUSSIONS AND CONCLUDING REMARKS

The obtained results indicate that ECA pressing can be used as an advanced method for processing UFG structure in tungsten with a grain size less than 1 μm. Such a structure has been obtained from ECA pressing by route C at a temperature of about 1000° C. However, ECA pressing of such low ductile, hard-to-deform material has a number of specific features as compared to ECA pressing of ductile materials [1,5]. One feature is primarily concerned with modifications to the die that provides decreasing intensity of shear strain due to increment of angle of channel intersection resulting in increased workability of the tungsten. Another feature is processing by route C, though, as shown in work [8], the most homogeneous microstructure can be formed in aluminium by using Route B_c. However, Route B_c didn't provide the necessary workability (technological plasticity) and in the case of CP W ECA pressing for 8 passes was successfully accomplished only by using Route C.

As known from previous work on ECA pressing of copper, aluminum and nickel [1,2,8], the process of UFG structure formation is connected with the formation of cell dislocation substructures which transform subsequently to granular type microstructures having high angle grain misorientations. The similar structural evolution can be observed in the tungsten during ECA pressing. After two-four passes mainly dislocation substructure is formed and after eight passes one can observe UFG structure with mostly high angle grain boundaries.

After ECA pressing the mean grain size of copper, aluminum and nickel is usually 0.2-0.3 μm, but in tungsten the mean grain size is about 1 μm. As known [15], a final grain size during SPD is determined by the balance between the accumulation of defects and their recovery. In this connection, the larger grain size in the tungsten after ECA pressing can be explained by the decreasing intensity of shear strain and relatively elevated homologous temperature of pressing ($T=0.39\ T_m$) in comparison with $T=0.22\ T_m$ for Cu [2].

Therefore further reducing of grain size can be achieved by decreasing of the deformation temperature or increasing the intensity of the ECA pressing. At the same time, as it was mentioned above, temperature decreasing or increasing the strain intensity of CP W results in samples failure due to a lack of their ductility. This problem can be solved by using of back pressure during ECA pressing. It was shown recently [1], that back pressure of 1-2 GPa facilitate low ductile materials to save their shape during ECA pressing. There is a ground to expect that such kind of technical modification will allow to decrease successfully the temperature of ECA pressing of CP tungsten up to 800-900° C and increase the intensity of shear straining at the expense of the die with angle of channels intersection 90° with out ingot failure. At this, forming of UFG structure with mean grain size 0.3-0.5 μm is possible.

In spite of the enhanced strength the W alloy also can be subjected to ECA pressing in the die with angle of channel intersection 120°, at temperature 1150° C and strain velocity 6 mm/s. However, the ingot was failed after 1-2 passes. Moreover, mainly γ-phase (Ni-based solid solution) was strongly deformed and α-phase (W-based solid solution) was not essentially refined.

In this connection, taking into consideration the revealed problems of the W-alloy ECA pressing it is supposed to fulfil the following tasks: 1) to fabricate a new die out of heat-resistant and high-strength material using back pressure; 2) temperature of ingots heating before ECA pressing will be increased up to 1300° C. Probably, it will be useful to increase angle of channel intersecting that will let to increase the quantity of passes during ECA pressing keeping the initial billet shape.

Thus, it is established within the frames of the present project that severe plastic deformation by ECA pressing of hard-to-deform materials, such as W and W-alloys can be successfully fulfilled. The ECA pressing results in strength enhancement and strong refinement of microstructure with a grain size up to 1 μm.

The ways of obtaining of UFG structures in W with a grain size 0.3 - 0.5 μm are determined that will ensure the increase both strength and ductility. Possibilities of processing of UFG structures in bulk W-alloy billets are scrutinized. The subject of further investigation must be presented by these tasks.

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APPENDIXE
(8 PAGES)

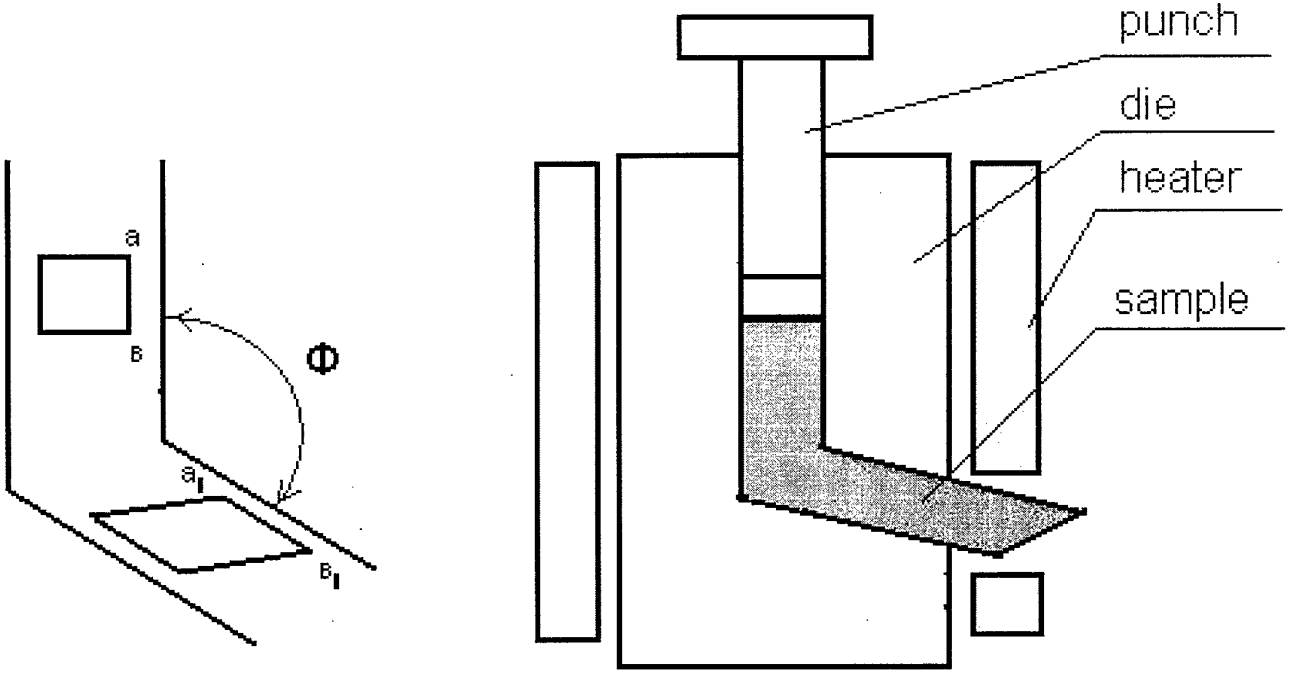

Figure 1. The principle features of an Equal Channel Angular pressing die.

Figure 2. Billets of commercially pure tungsten after ECA pressing in matrix with angle of channels intersection equal to 90°.

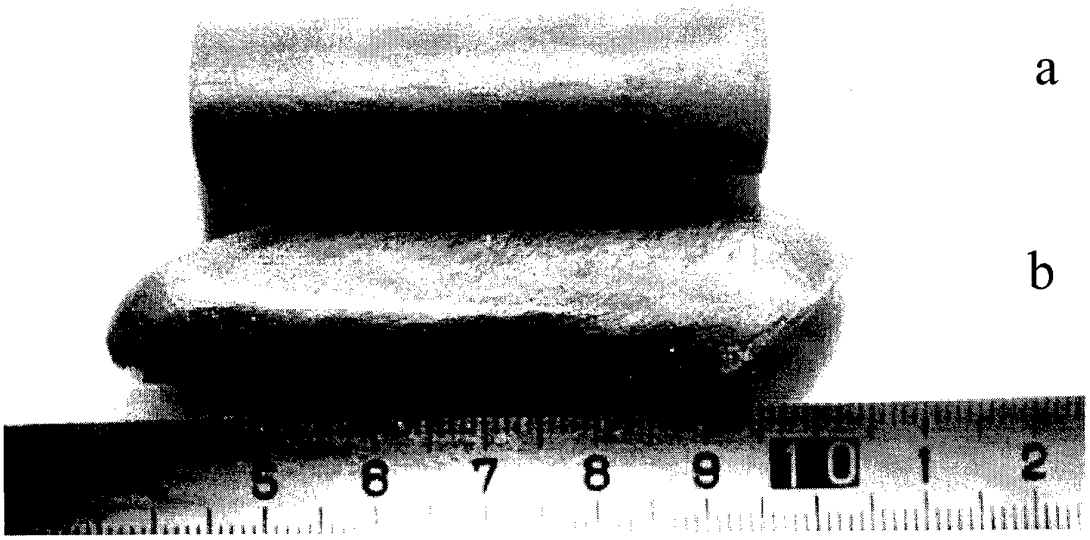

Figure 3. Billets out of CP tungsten: (a) before, (b) after ECA pressing by Route C, 8 passes, using the modification (see the text for details) of SPD processing.

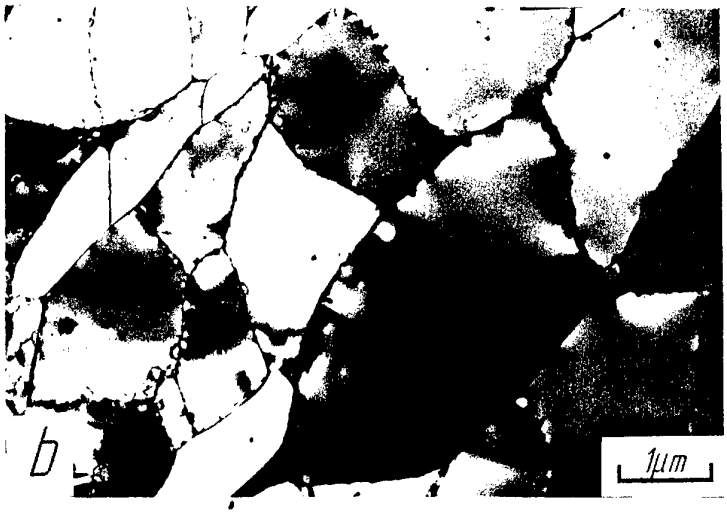

Figure 4. Microstructure of initial tungsten: (a) coarse recrystallized grains observed by optical microscopy; (b) dislocation substructure of these grains observed by TEM.

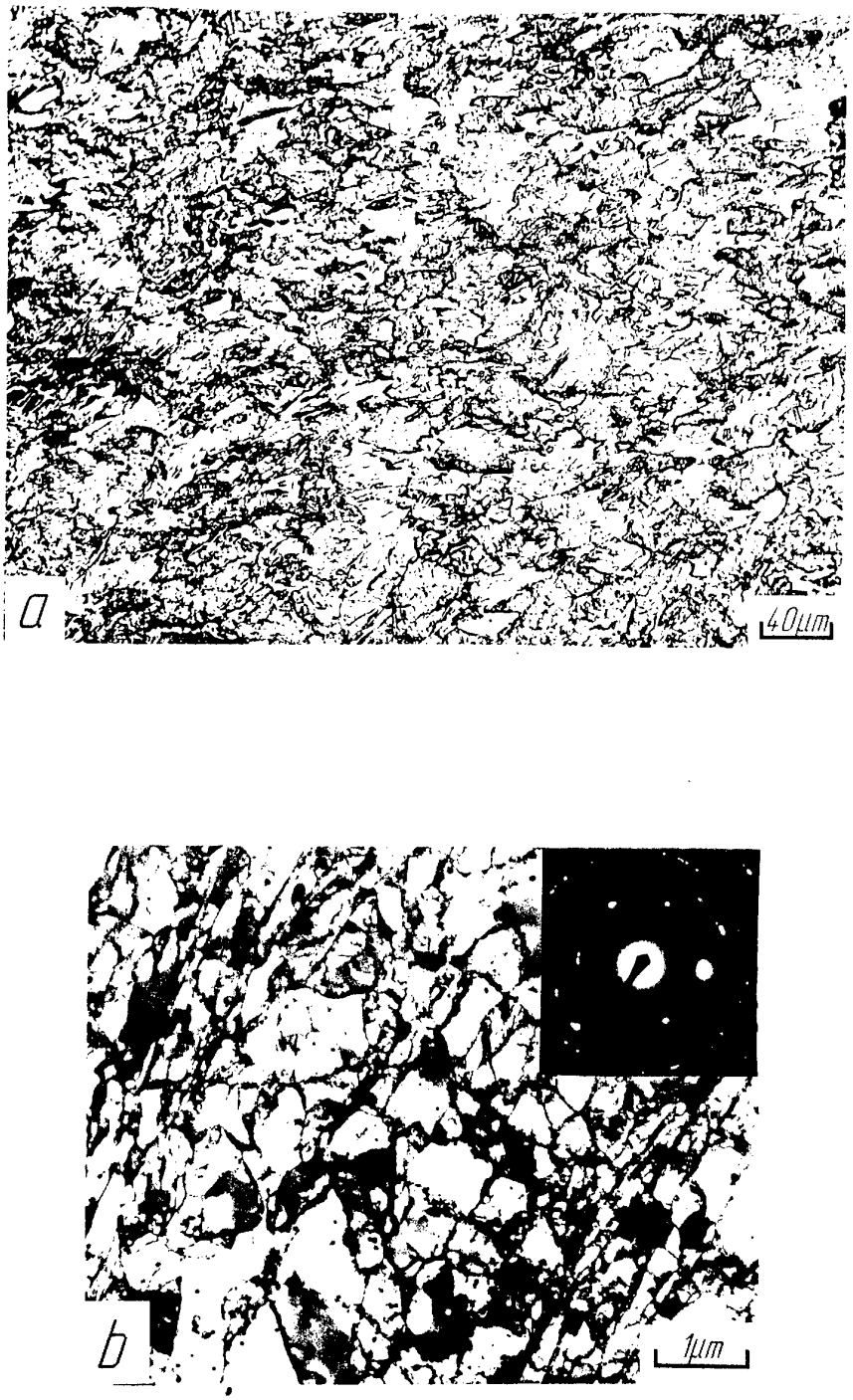

Figure 5. Microstructure of tungsten after ECA pressing, 4 passes; (a) optical microscopy, (b) TEM structure and microdiffraction pattern.

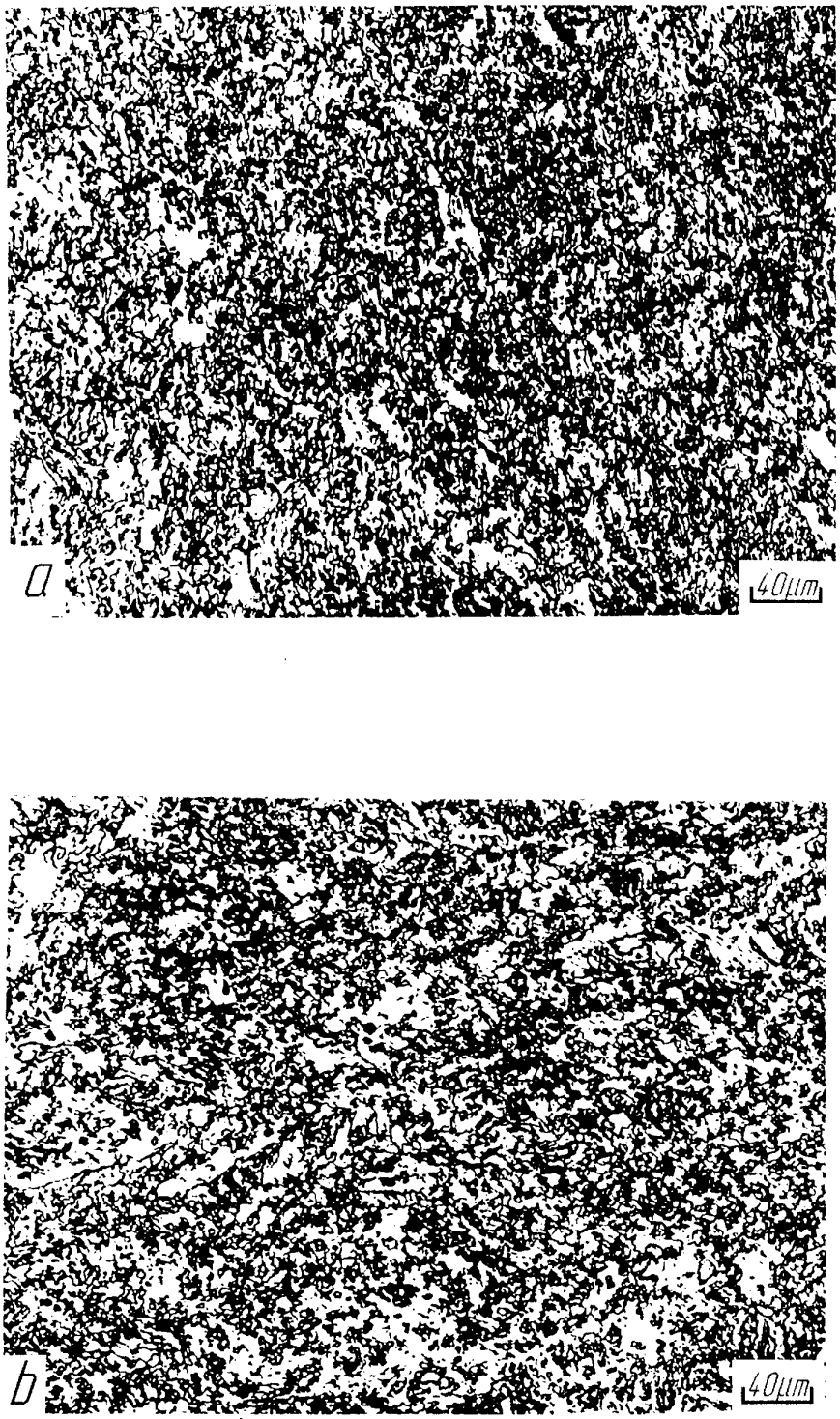

Figure 6. Microstructure of tungsten after ECA pressing, 8 passes (a) cross section of a billet, (b) longitudinal section of a billet.

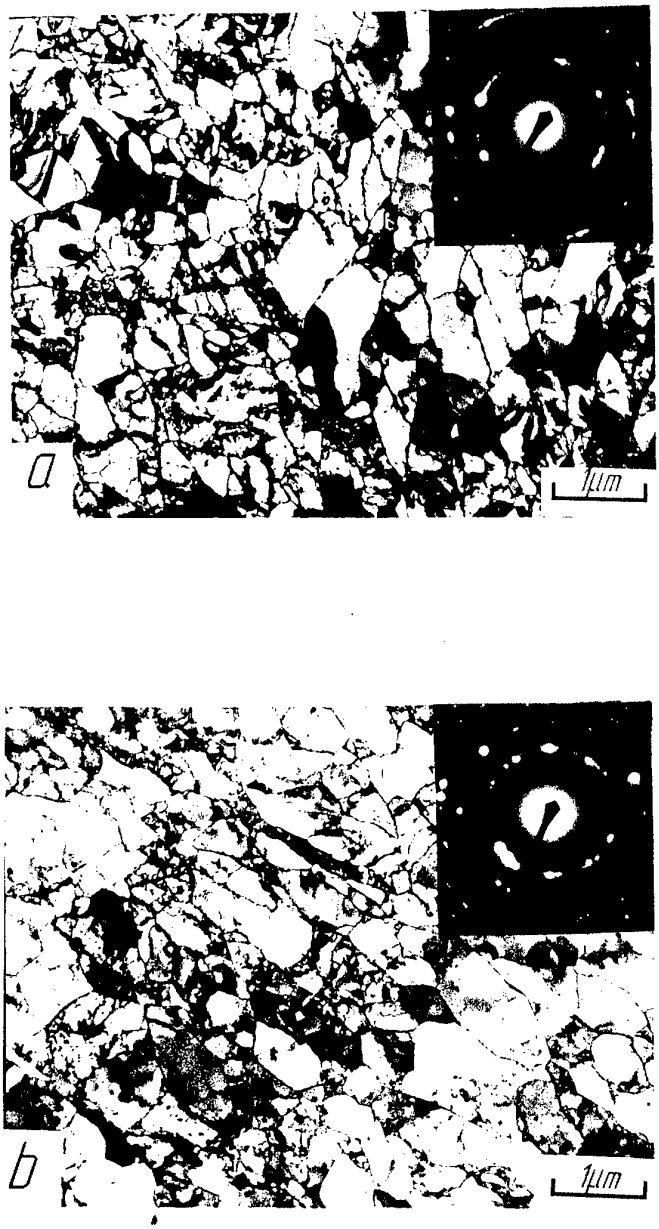

Figure 7. TEM structure of tungsten and microdiffraction pattern after ECA pressing, 8 passes (a) cross section and (b) longitudinal section.

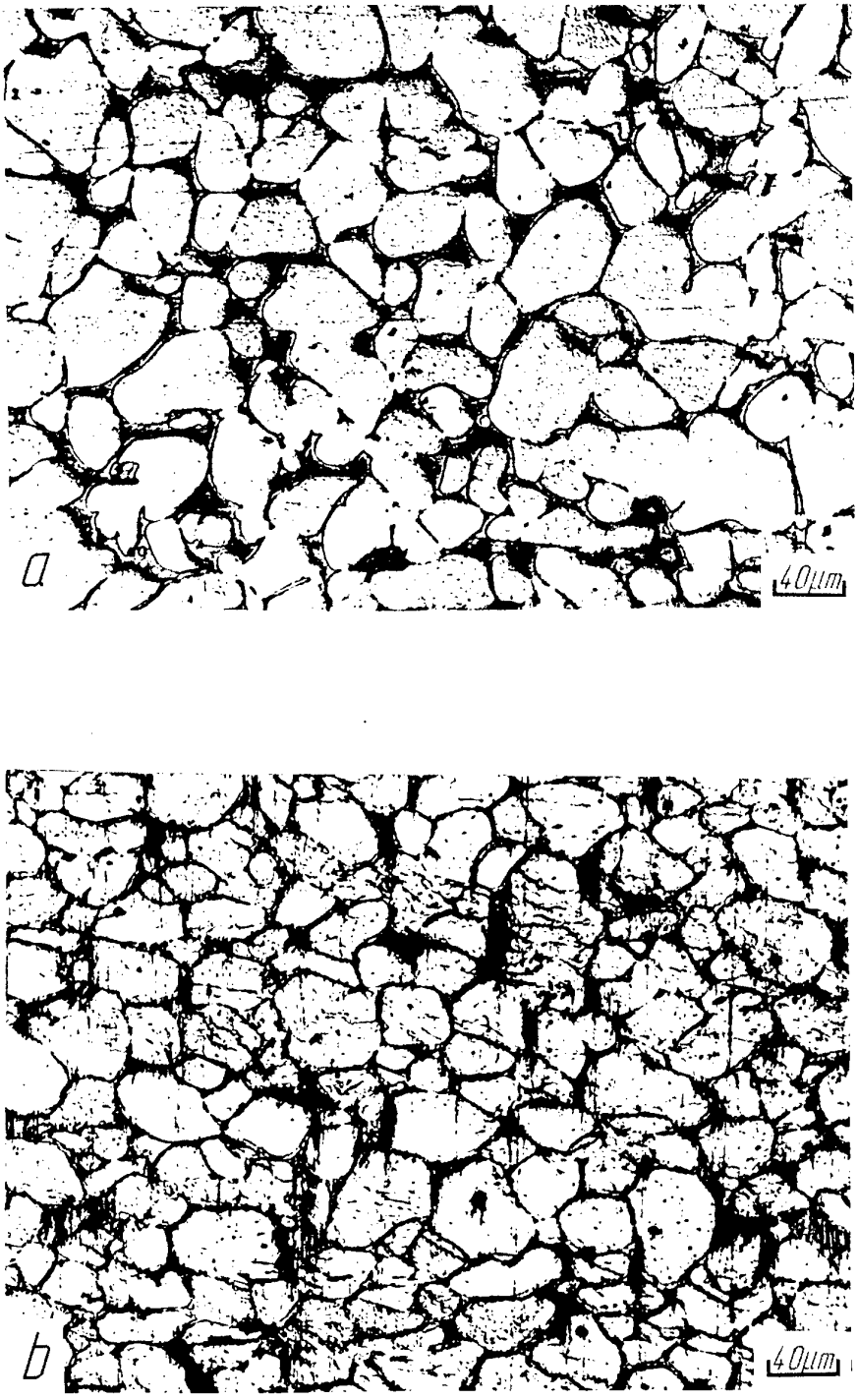

Figure 8. Two-phase (α+γ) microstructure of alloy W-4.9%Ni-2.1%Fe in longitudinal section: a - the intial rod, b - the ingot after one pass of ECA pressing. Optical microscopy.

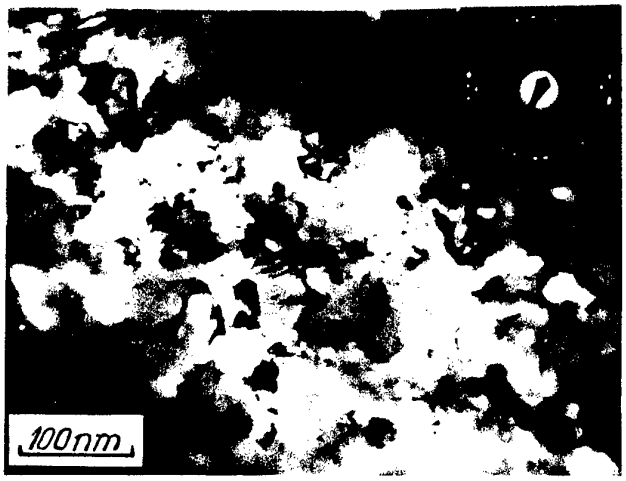

Figure 9. TEM structure of γ - solid solution after one pass ECA pressing. Bright-field image.

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13. For the reporting period the regimes of severe plastic deformation were revealed and successful equal channel angular pressing of hard-to-deform CP W was performed resulting in the refinement of the microstructure and increase of microhardness. Approaches to refine microstructure in hard-to-deform W alloy were developed.		
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