Thermal stability and high-temperature deformation of tungsten nanocomposite

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Abstract This paper presents the result of research of thermomechanical treatment impact on polycrystalline tungsten structure and properties. Samples were exposed to deformation. Two deformation technics were applied: uniaxial rolling and cross direction rolling. After deformation all samples acquired the form of foil. After deformation the relation between the structure of the foils and the annealing time was investigated.

The investigation of high-temperature deformation was conducted using the samples which underwent preliminary heat treatment, and which didn’t. The result of heat treatment and tensile strength investigation of pre-machined polycrystalline tungsten samples at elevated temperature is presented in this study. It is shown that annealing at 2670 K after uniaxial rolling could result in significant increase of high temperature creep resistance of tungsten.

The factors that determine the effect of reducing high-temperature deformation of modified tungsten have been identified. As a result of mechanical treatment with subsequent annealing a unique nanoscale structure is formed. The tungsten nanocomposite has been obtained for the first time. During investigation of tungsten nanocomposite thermal stability its structural stability has been confirmed.

1. Introduction

One of the most important task of material science is invention of structural materials resistant to elevated temperature and mechanical treatment into corrosive media. Structural materials of high-temperature and high-loaded systems must have such properties as high tensile strength, high ductility and low creep at elevated temperatures. Tungsten based materials are used as a high temperature material in many fields of science and technology due to the high rates of long-term strength and creep resistance. However, increasing temperature and operation time lead to deterioration of tungsten properties, so it is needed to enhance creep strength at temperatures above 1770 K.

To improve high-temperature strength of tungsten an idea of creation tungsten nanocomposite is suggested. Grid of nanoscale potassium filled pores which forms in its structure should contribute to a slowdown of recrystallization processes. Insoluble nanoscale potassium pore will prevent the formation and growth of recrystallized nuclei.
2. Experimental

The influence of thermo mechanical treatment on tungsten based materials structure and properties was studied using microfoils 100 µm thickness. The foil-shape samples with two types of structure: equal axis grains (after cross-direction rolling deformation) and oblong grains (after one-direction rolling deformation) are used in this research. Thermal stability investigation was conducted in vacuum chamber under pressure $2.6 \times 10^{-4}$ Pa. The annealing was carried out by direct drawing a current trough the samples in three stage: the 1-st - 30 min long up to 1670 K with heating rate 300 deg/min and after up to 2670 K with heating rate 50 deg/min; the 2-nd - 90 min long at temperature 2670 K; the 3-rd - 180 min at the same temperature.

To determinate time dependence of high temperature annealing on material structure the samples were marked by diamond paramecium after the 1-st 30 minutes annealing.

The high temperature deformation was studied under the constant load at temperature 2470 K. The investigation of high-temperature deformation was conducted using the samples which underwent preliminary heat treatment, and which didn’t.

The experimental result are supplement with X-ray and micro structural analysis by using the JEOL 6460 microscope.

3. Result and Discussion

Different mechanical treatment and annealing at 2670 K of polycrystalline tungsten samples resulted in formation of various recrystallized structure with same chemical composition. After high temperature annealing all modified tungsten foils have a structure with grid of nano-sized pores (from 50 to 150 nm) on grain boundaries. Pores present in specimens with oblong grains and with equal axis grains alike and almost all pores are located on borders of grains.

Samples from the cross-rolled foil acquire equally grain size structure after high-temperature annealing. But samples with uniaxiality rolling have elongated grains structure, the crystallographic orientation of all the grains changed in the same direction [100] (Figure 1). Dislocation density within their grains body decreased, the X-ray spectrum lines half-width is evidence of this phenomena (Figure 2). Long-term annealing does not lead to significant changes in the material structure: clusters of small pores at big grains boundaries are maintained after that. Observed effect has been stable up to 2670 K.

![Figure 1](image1.png)  ![Figure 2](image2.png)

**Figure 1.** Amount of crystallites with end on direction  
**Figure 2.** The X-ray spectrum lines half-width

The structure difference is described by various mechanical treatment. In initial material unsolvable potassium is located in stuff pores with effective diameter about 0.1-1.0 microns. One direction rolling forced pores get the elliptic form extended in a direction of rolling. In course of annealing at
elevated temperatures this form becomes unstable and breaks up to the pores of the smaller dimension built in rows in a direction of rolling. Pores row hinder grain growth in perpendicular to the rolling direction in process of recrystallization under annealing. It leads to the formation of structures with oblong grains elongated towards the direction of rolling (Figure 1). In such a structure grains slippage considerably decrease.

Specimens with oblong grains structure after heat treatment at 2670 K under constant load high-temperature deformation has shown significant reduction (3 times) of deformation rate, and on the contrary, deformation rate under constant load of samples with equal axis grains structure is accelerated. It is caused by different materials structure which formed after heat and mechanical treatment (Figure 3). The cross-rolled sample from non-heat treated tungsten has recrystallized during creep testing at 2470 K and had a structure with a smaller average grain size than the sample from the pre-annealed material. So its deformation rate was significantly higher than non-heat treated one (Figure 4).

![Figure 3. High-temperature deformation of tungsten with equal axed grains](image)

![Figure 4. High-temperature deformation of tungsten with oblong grains](image)

Comparing pre-annealing samples with equal axis grains structure and oblong grains structure shows that first one have greater creep rate than materials with oblong grain. The sample with equal axial has considerably large speed especially at the second stage of deformation, as shown in Figure 5. It means that after annealing samples with oblong grains have greater creep resistivity, than without it.

The factor determining a high temperature deformation reduction is a special structure which is formed under uniaxial rolling with subsequent high temperature annealing. This structure is characterized by elongated grains with grid of nano-sized pores at their boundaries which are developed increasing creep resistance [1-3].

The results of investigations of thermal stability and the factors, which determine the effect of reducing high temperature deformation of modified tungsten, are presented. It is shown that thermomechanical treatment (one direction rolling with subsequent high-temperature annealing) results to formation of tungsten nano-composite unique structure which have an effect of significant reduction of high-temperature deformation [4-6]. Such materials as tungsten nanocomposite, which is thermally stable up to 2670 K, obtained for the first time.
4. Conclusion
Research of influence mechanical and heat treatment on structure and properties of polycrystalline tungsten are conducted. It is shown that heat treatment at temperature 2670 K of one chemical compound tungsten after different type of rolling deformation leads to formation of various recrystallized microstructure. One-direction rolling with subsequent annealing initiate textured structure (oblong grains are elongated far the [100] direction), cross-direction rolling with subsequent heat treatment leads to form the structure with huge equal axis grains. However both type of samples have the structure with grid of nanoscale pores (from 50 to 150 nanometers) on grains boundaries.

The high-temperature deformation tests at 2670 K under constant load show significant reduction of oblong grain sample creep (up to 3 times), and in the contrary the raise of deformation speed in equal grain sample after annealing.

The factor defining reduction of high-temperature deformation is the unique structure with oblong grains and grids of potassium nanoscales pores on their boundaries, which form as a result of a one-direction rolling and high-temperature annealing after that.

The tungsten nanocomposite has been obtained for the first time. During investigation of its thermal stability it is shown that tungsten nanocomposite thermally stable up to temperatures 2670 K.

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References
[1] Alekseev S, Taubin M and Yaskolko A 2009 20th Workshop ISTC in Korea (Seul)
[2] Alekseev S, Taubin M and Yaskolko A 2013 Proc. of 2nd Biotechnology World Congress (Dubai)
[3] Alekseev S, Taubin M, Pavlov A and Yaskolko A 2013 BIT’s 4th World Gene Convention p 301.
[4] Wright P K 1978 Metallurgical Transactions 9 955
[5] Yamazaki S, Ogura S and Fukazawa Y 1978 High Temperature – High Pressure 10 329
[6] Briant C L 1993 Metallurgical Transactions 24 1073