BULK NANOSTRUCTURED REFRACTORY METALS WITH ENHANCED MECHANICAL PROPERTIES PRODUCED BY EQUAL CHANNEL ANGULAR PRESSING

Interim Technical Report

by

R.Z. Valiev and I.V. Alexandrov

(10 September 2002 - 11 December 2002)

United States Army

EUROPEN RESEARCH OFFICE OF THE U.S. ARMY

London England

CONTRACT NUMBER: N62558-02M-6024

R&D 9362-AN-01

Name of Contractor

Institute of Physics of Advanced Materials
Ufa State Aviation Technical University
Russia

Approved for Public Release; Distribution unlimited

1ST **INTERIM REPORT**

A. The Cover Page

(1) Bulk nanostructured refractory metals with enhanced mechanical properties produced by equal channel angular pressing

(2) Professor R.Z. Valiev

(3) Professor V.S. Zhernakov

(4) N62558-02M-6024

(5) Interim Report

(6) 10 September 2002 - 11 December 2002

(7) The Research reported in this document has been made possible through the support and sponsorship of the U.S. Army. This report is intended only for the internal management use of the Contractor and U.S. Government.

REPORT DOCUMENT PAGE			*Form Approved OMB No 0704*
1. AGENCY USE ONLY.	**2. REPORT DATE** December 11, 2002	**3: REPORT TYPE AND DATES** Interim Report (10.09.2002 – 11.12.2002)	
4. TITLE AND SUBTITLE Bulk nanostructured refractory metals with enhanced mechanical properties produced by equal channel angular pressing			**5. FUNDING NUMBERS** N62558-02M-6024
6. AUTHORS: Prof. R.Z. Valiev, Prof. I.V. Alexandrov			
7. PERFOMING ORGANIZATION NAMES AND ADDRESS Institute of Physics of Advanced Materials, Ufa State Aviation Technical University. 12 K. Marx St., Ufa 450000, Russia			**8. PERFORMING ORGANIZATION REPORT NUMBER**
9. SPONSORING / MONITORING AGENCY NAME AND ADDRESS U.S. Government and European Research Office of the U.S. Army. USARDSG-UK, FISCAL-OFFICE, EDISON HOUSE, 223 OLD MARYLEBONE ROAD, LONDON NW1 5TH, UNITED KINGDOM			**10. SPONSORING / MONITORING AGENCY REPORT NUMBER**
11. SUPPLEMENTARY NOTES In co-operation with Dr. R. Dowding, U.S. Army Research Laboratory Aberdeen Proving Ground, Maryland 21005-5066			
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release, distribution unlimited			**12b. DISTRIBUTION CODE**

13. ABSTRACT The aim of the present project is to obtain a set of bulk billets 100 mm long with UFG structure made of refractory metals such as tungsten and tantalum. The previous positive experience of refining the tungsten's structure using equal channel angular pressing (ECAP) was taken into account.

For the production of bulk billets with UFG structure having a more long-lengthed size than those obtained in our earlier works it has been necessary to introduce modifications into the die-set being used and to carry out updating of the whole die-set for ECAP.

That is why the main objective of the first stage was to find a new approach to the modernization of pressing process, to design and manufacture a new die set and to test its work, using relatively long billets made of hard-to-deform refractory metals.

14. SUBJECT TERMS Refractory materials, equal channel angular pressing, Ultrafine-grained microstructure			**15. NUMBER OF PAGES**
			16. PRICE CODE
17. SECURITY CLASSIFACATION OF REPORT Unclassified	**18. SECURITY CLASSIFICATION OF THIS PAGE** Unclassified	**19. SECURITY CLASSIFICATION OF ABSTRACT** Unclassified	**20. LIMITATION OF ABSTRACT** UL

Abstract.

The aim of the present project is to obtain a set of bulk billets 100 mm long with UFG structure made of refractory metals such as tungsten and tantalum. The previous positive experience of refining the tungsten's structure using equal channel angular pressing (ECAP) was taken into account.

For the production of bulk billets with UFG structure having a more long-lengthed size than those obtained in our earlier works it has been necessary to introduce modifications into the die-set being used and to carry out updating of the whole die-set for ECAP.

That is why the main objective of the first stage was to find a new approach to the modernization of pressing process, to design and manufacture a new die set and to test its work, using relatively long billets made of hard-to-deform refractory metals.

Keywords: *severe plastic deformation, ultrafine-grained structure, W and Ta, equal-channel angular pressing, refractory materials.*

Introduction.

Within the frames of our previous projects EARO # 68171-99-M and # 68171-01-M-5641 we demonstrated for the first time a possibility to refine the microstructure in such hard-to-deform and low-ductility materials as CP tungsten and its alloys by ECAP. Using these results it has become possible to find the decision of this new task of producing of longer-sized billets from W and Ta with uniform ultrafined structure by a design and fabrication of a new specialized die-set, choice of the optimal route and the temperature and rate conditions of ECAP.

The main purpose of the first stage of the project was the development of processing approaches for obtaining relatively long billets made of CP tungsten.

1. Development and application of the processing approach.

To implement of the project task, aimed at processing by equal-channel angular pressing (ECAP) of relatively long bulk nanostructured billets of pure W 10 mm in diameter and 100 mm in length with the grain size less than 0.5 μm having enhanced mechanical properties, we developed a complex processing approach, which includes as follows:

- Processing of rods ∅16 mm × 60 mm having ultrafine-grained (UFG) structure by ECAP.
- Processing of rods ∅10 mm × 100 mm by means of direct pressing (extrusion) of ECAP billets.
- Investigations of structure and mechanical properties of the billets in the initial state, after ECA pressing and its subsequent direct pressing.

For the first stage we applied the experience obtained during our previous project fulfillment.

A necessity to combine ECA pressing with a subsequent extrusion is connected first of all with technical task to produce billets having the relation L/d=8-10, that is essentially higher than a typical billet with L/d=5-6.

The enhanced length of the billet leads to additional problems connected with increasing the endurance of the die-set.

Meanwhile, additional extrusion of the ECAP billets, according to our recent investigations of Ti materials, contributes to additional enhancing of mechanical properties. These were the reasons to determine the processing approach.

To realize ECAP processes and extrusion during the next second quarter we will use press-forging equipment – hydropress DB2426 up to160 tons by force with the velocity of traverse moving 6 mm/s and the crank press LKP up to 400 tons by force with a velocity of traverse moving up to 300 mm/s. We will also use two types of experimental die-sets for ECA pressing. The first one will be used, applying parts out of a heat-resistant alloy with an exploitation

temperature up to 900 °C and the angle of channels intersection 135° (Fig. 1a). The second one is a special die-set with an angle of pressing channels intersection 120°, which is made out of high-strength tool alloy with the exploitation temperature up to 500 °C (Fig. 1b). To realize the extrusion process we will use the die-set with removable forming matrixes, which provide varying of the extrusion coefficient. To analyze microstructure by transmission electron microscopy (TEM) we use the microscope JEM-100B. For mechanical testing we will apply a special device for small samples' tensile testing with a computer control.

Investigation of the ECAP of Ta aimed at processing of UFG structure, will be realized after receiving from The Customer of Ta rods. The special ECAP die-set was manufactured in frames of the other project. It allows using backpressure during ECAP.

2. Designing, manufacturing and testing of the die-set.

For the realization of the given approach the following works were done.

1. The working documentation of the die-set for ECAP W in isothermic conditions was developed and its main parts were produced. (Fig. 2a)

One of the features of the die-set is that the shape-forming part is manufactured with two parallel vertical channels shifted relatively to each other. These channels are connected by the third channel at an angle of 120° to the vertical axis (Fig. 1b). When using this type of a shape-forming parts two acts of shear straining are performed during one cycle of pressing. This corresponds to the most optimal route of ECAP for producing of UFG W.

To determine ECAP parameters the computer finite elements simulation using ANSIS 5.5-program was performed as well. The results of the simulation will be presented in the next report.

2. The testing of the first die-set for ECAP was approbated (scheme – Fig. 1a).

This type of die-set was made for the previous project and at present its technical testing is carried out.

The first experiments showed that the main shortcoming of this die set is a low resistance to crumpling of shape-forming elements when heated up to 1000-1200 °C. Its usage appears to be more effective at high temperatures of the billets consisting more than 1200°C, because the resistance to the deformation of W at these temperatures is 2-2,5 times less in comparison with the temperature of 1000 °C. It is planned to perform this process in the next quarter.

3. The die set for extrusion of ECAP billets is designed and produced for extra microstructure refining and the required dimensions obtaining.

Extrusion is proposed to be for the billets heated at 1000 °C. Extrusion rate parameters will be defined during the ongoing investigations.

The planned extrusion ratio will constitute 2, with the final billet's dimensions Ø11x120 mm.

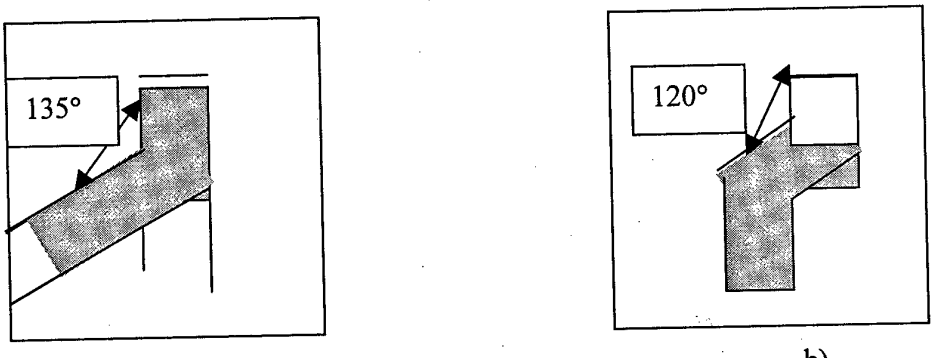
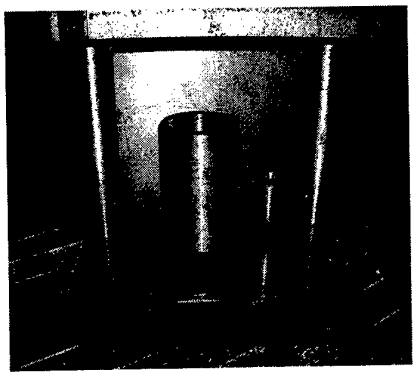
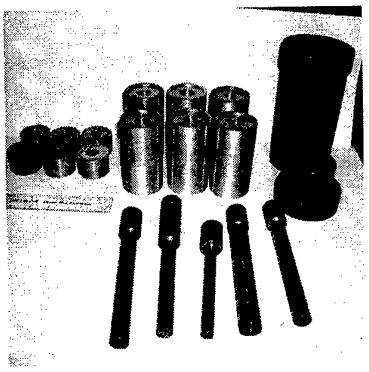


Fig.1. Principal schemes of the dies with an angle of channels intersection 135° (a) and with parallel pressing channels, intersecting at the angle 120° (b).

Thus the first quarter work was aimed at developing the processing approach, which includes ECAP of W billets and their further direct pressing (extrusion). The modified die-sets were designed and their main parts were fabricated. However, still unsolved remains the problem of receiving of the initial materials. This problem is being solved now from our side as well as from the side of our partner.



a)



b)

Fig. 2. Main parts of the special die-set with parallel pressing channels, intersecting at the angle 120° (a), ferrule, punches, insertion parts and matrixes for extrusion (b)

3. Tasks for the second quarter.

In frames of the second quarter we are planning to conduct the following work:

- To accomplish fabrication and to approbate the experimental die-set with parallel pressing channels;
- To conduct investigations on extrusion of W billets, aimed at selecting the processing regimes;
- To conduct microstructure characterization of the W and Ta initial materials, after ECA pressing and extrusion.

References

1. T.G. Langdon, M. Furukawa, M. Nemoto, Z. Horita, Using Equal-Channel Angular Pressing for Refining Grain Size, JOM, Vol. 52, № 4, 2000, pp. 125-134.
2. K.T. Hartwig, S.N. Mathaudhu, H.J. Maier and I. Karaman, Hardness and Microstructure Changes in Severely Deformed and Recrystallized Tantalum. Ultrafine Grained Materials II, TMS, 2002, pp. 151-160.