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## MICROSTRUCTURE ANALYSIS OF TUNGSTEN HEAVY ALLOYS OBTAINED BY SOLID AND LIQUID PHASE SINTERING

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**Abstract:** Heavy alloys based tungsten with composition 90W-7Ni-3Fe were homogenized 2 hours and pressed through bilateral compaction in a rigid die, followed by sintering in solid state at 1275 °C, 1h, in hydrogen atmosphere. The dimensions of the tungsten particles were of approximately 3,6 μm and their apparent density of 4 g/cm<sup>3</sup>. After the sintering in solid state the heavy alloy was sintered with liquid phase at 1500 °C for 30-60 min. The microstructure of the heavy alloy based tungsten, sintered in 2 steps, shows that the spherical particles of W are more fine at the sintering in solid state than that at sintering with liquid phase, has a tensile of 15 % and a tensile strength of 900 MPa.

**Key words:** Heavy alloys, composites, liquid phase sintering.

### 1. INTRODUCTION

The microstructure of the heavy alloy based tungsten consists from W spherical particles crystallized in system bcc enclosed into a matrix formed from solid solution crystallized in fcc system [1]. Usually, Ni and Fe are used to form the ductile matrix of the solid solution alloy W-Ni-Fe. The heavy alloys based tungsten are used like penetrators with kinetic energy, counterweights, protection screen against radiations, electrical contacts of high power etc., due to their high density, a very good mechanical strength and a good worn out strength [2]. Sintering with liquid phase of the homogenized W, Ni and Fe powders, at a temperature of about 1500 °C constitute the conventional manufacturing technology of the heavy alloys based tungsten with high density .

The microstructural factors, like the dimension of the tungsten particles, fraction of tungsten from the matrix and the contiguity W/W, influence the mechanical properties and the fracture behavior of the heavy alloys based tungsten. The fracture critical modes of the heavy alloys based tungsten are characterized through inter-particle cleavage into the W grains, inter-particle fracture at W/matrix limits

and fracture of the matrix phases [4]. The interface W/W is known like being the weakest interface, generally, at the interfaces W/W [3]. An important microstructural parameter into the heavy alloys based tungsten is the contiguity W/W, which is defined like a fraction of the interfacial area of the W/W from the total interfacial area [4]. There was established that the adiabatic deformation at shearing may be intensified through a fine microstructure of the heavy alloys based tungsten [5].

Many researchers studied new methods to obtain fine microstructures for the heavy alloys based tungsten: through alloying with refractory elements, through cold deformation followed by recrystallization], through sintering in solid state and through mechanical alloying. As well, the microstructure's finishing ways of the heavy alloys based tungsten didn't give satisfactory results regarding the rising of the penetration capacity because of an incomplete density or of brittleness.

The aim of this paper is to study the finishing microstructure of the heavy alloys based tungsten obtained through a sintering process in 2 steps, its effects on the microstructure evolution and on its mechanical properties. Sintering in solid state was first made at 1275 °C

for 1 h to reach the complete density without a significant growing of the grains into the heavy alloys based tungsten. It was then followed by a sintering with liquid phase, to control the microstructural factors, as the W particles dimension, the contiguity W/W and the fraction of matrix volume, which avoid an abrupt rising of the W grains during the sintering process.

## 2. EXPERIMENTAL PART

The powders used to make the mixtures were: tungsten powder, elaborated to S.C. SINTEROM S.A. from Cluj-Napoca, with an average dimension of particles of 3,6  $\mu\text{m}$ ; nickel carbonyl powder, sort INCO 123, with the average dimension of particles 5,5  $\mu\text{m}$  and Höganäs iron powder, sort NC 100.24, with the particles dimension < 63  $\mu\text{m}$ .

The homogenization of the powders mixture was done using an homogenizer with horizontal axes, in inclined glasses recipients, fixed in a metallic frame. To ensure a good



**Fig.1.** The sem image of the microstructure the heavy alloys based W, 90W-7Ni-3Fe sintered at 1275 °C.

dispersion of the constituents there were homogenized first the nickel and iron particles for 1 h and then was added the tungsten powder, continuing the homogenization for another hour. From this mixture there were made samples with the section of 5,8 mm x 5,8 mm and the length between signs of 25 mm for the tensile strength tests, and samples with 10 mm x 10 mm x 50 mm for the impact tests. The compaction pressure was 200 MPa.

The green compacts were sintered at 1275 °C in hydrogen atmosphere. The heavy alloy based tungsten sintered in solid state was sintered with liquid phase at temperatures from 1450 °C at 1525 °C with sintering length varying from 30 min to 60 min. The samples sintered in 2 steps were annealed at 1150 °C, for 1h, in nitrogen

atmosphere, followed by cooling in water to prevent the brittleness with hydrogen and segregation of impurities during the cooling period. The densities of the sintered samples were determined using the water immersion method. The dimension of W particles, the volume fraction of the matrix and the configurations W/W of the heavy alloys sintered in 2 steps was characterized through SEM.

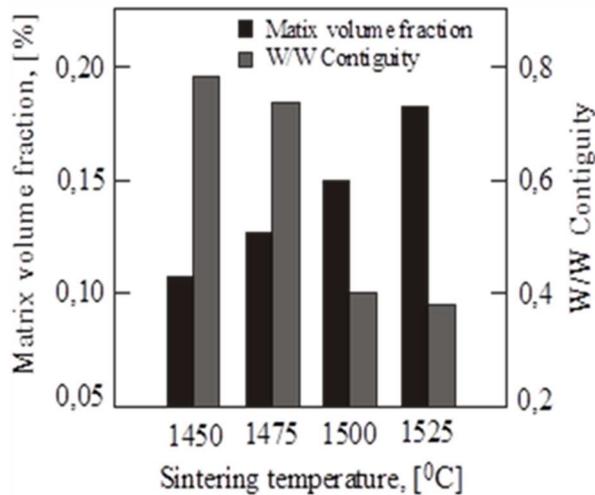
## 3. RESULT AND DISCUSSIONS

The microstructure of the heavy alloy based tungsten sintered in solid state show that the W grains were intercalated one another and the matrix phase is isolated like W continuous phase as it can be seen in figure 1. The heavy alloy based tungsten sintered in solid state has a high relative density of about 99 %. The average dimension of W particles was about 3  $\mu\text{m}$ , determined through microstructural analysis of the heavy alloy sintered in solid state.



**Fig.2.** The fractography of the sample for tensile strength test for the heavy alloy based W sintered in solid state at 1275 °C, 1h.

The volume fraction of the matrix phase were 10 %, and the contiguous fraction of the limits area W/W, as called contiguity W/W, of the heavy alloy sintered in solid state was about 0,75. Comparing the fraction of the matrix phase of 16 % and the contiguity W/W of 0,30 for the conventional sintering, with that one with liquid phase of the heavy alloy, it can be seen that for the sintering in solid state, the fraction of the matrix phase diminished and the contiguity W/W grow up. To determine the mechanical properties of the heavy alloy based tungsten sintered in solid state there were done tensile tests. The mechanical properties were sensibly dependent by microstructural factors, as the dimension of the tungsten grains, the volume fraction of the matrix and the contiguity W/W.



**Fig.3.** The variation of the fraction into the matrix volum, and of the contiguity W/W, of the heavy alloys based W sintered in 2 steps depending on the sintering temperature, from 1450 °C at 1525°C.

There was communicated, there exists four ways of fracture for the heavy alloys based tungsten: cleavage of the W grains, fracture of matrix phase, inter-particle fracture at the interfaces W/W and inter-particle fracture at interfaces W/matrix. The fractography of the heavy alloys sintered at 1275 °C presents a fragile fracture characteristic, especially at W/W limits, as it can be seen in figure 3.

The brittle fracture character of the heavy alloy sintered in 2 steps is due to, mainly, the high contiguity W/W and to the small volume fraction of matrix. The volume fraction of the matrix phase was reduced because the solubility of W into the matrix diminish; the rising of the W/W contiguity is due to diminishing of the volume of matrix fraction and to the growing up of the angle between tungsten particles and matrix phase in the same time with the diminishing of the sintering temperature.

During the tensile solicitation, the material deterioration accumulates like micro cracks at W/W limits. It may be considered that the low ductility is associated with a high contiguity W/W, because the micro cracks joint easily one each other without blunting the fissures extremities. Doing a comparison with the heavy alloys based tungsten sintered in conventional mode with liquid phase, the heavy alloy based tungsten sintered in solid state is not adequate for practical application because its small tensile. The tensile variation is tied by the volume fraction of matrix and by the contiguity

from the heavy alloys. The deformation at tensile adapts into the heavy alloy at deformation of the interconnected phase in a continuous mode. When the W/W contiguity is big, as it is for the samples sintered in solid state, the micro cracks formed at the brittle limits of W/W joint easily one each other without a deformation of the ductile phase - matrix. While the contiguity W/W diminished, the deformation adapts through the phase deformation from the ductile matrix before the micro cracks interconnection from the limits W/W from the heavy alloys based tungsten.

When the sintering was done below 1450 °C, W particles sintered in solid state weren't yet interconnected one each other and the matrix phase was isolated in a similar mode to the sintering in solid state of the heavy alloy based tungsten. When the temperature was less than 1475 °C, the W particles spheroids and the matrix phase penetrated into the W/W limits, similarly to the case of the heavy alloy conventional sintered with liquid phase. Figure 3 presents the variation of the volume fraction of the matrix and of the contiguity W/W with the sintering temperature. In the same time, the volume fraction of matrix rises, and the contiguity W/W diminishes with the sintering temperature. The rising is due to the solubility growing of the W into the matrix phase and to the growing of the interface angle W/matrix.

The length of the sintering with liquid phase raised from 30 min at 60 min at 1500 °C to study the effect of the sintering length on the microstructure evolution of the heavy alloy based tungsten sintered in 2 steps. The average of the particles dimension were determined on SEM microphotographs. The W particles from the heavy alloy sintered in 2 steps became more coarse with the rising of the length of sintering with liquid phase. The mechanical properties of the heavy alloy based tungsten sintered in 2 steps were evaluated through tensile tests and then compared with those of the heavy alloy conventionally sintered with liquid phase. It is wish able that the heavy alloy based tungsten sintered in 2 steps has the tensile and tensile strength similarly with the heavy alloy based tungsten conventional elaborated, maintaining simultaneously a fine microstructure.

The sintering with liquid phase was done at 1500 °C for 60 min in hydrogen atmosphere, followed by a cooling which speed was 10 °C/min. For the heavy alloy sintered in 2 steps the tensile strength was 900 MPa and the tensile 15 %, as it can be seen in figure 4. The tensile of the heavy alloy sintered in solid state grow up from 0,5 % at 15 % through sintering in 2 steps due to the rising of the matrix's volume fraction and to the diminishing of the W/W contiguity. The microstructure's control of the heavy alloy sintered in 2 steps was efficient because there was maintained a fine microstructure, simultaneously with a high tensile strength and a good ductility.

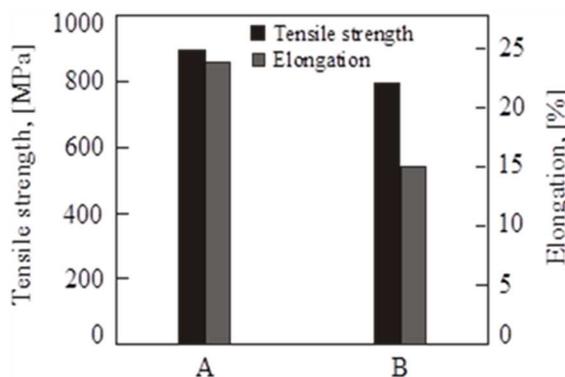
#### 4. CONCLUSIONS

- The control of the microstructure of the heavy alloy based tungsten sintered in 2 steps was efficient because it permitted the maintaining of a fine microstructure. When the sintering is done in solid state at 1275 °C, 1h, the sintered heavy alloy made from fine tungsten powders, has a high density of approx. 90 %, a good tensile strength of approx. 1100 MPa, due to the structure with fine particles, while the tensile is small, under 1 %, because of the small volume fraction of phase-matrix and of the high contiguity W/W.

- The heavy alloy based tungsten sintered in solid state was then sintered with liquid phase at 1500 °C, for times varying between 30 and 90 min. It has finer W particles, of about 6-15µm, compared to the heavy alloy conventional sintered with liquid phase.

- The high tensile strength of 900 MPa and the good tensile of 15 % were obtained through

a sintering process in 2 steps, maintaining effectively a fine microstructure into the heavy alloy based tungsten.



**Fig.4.** The variation of the tensile and the tensile strength for: a) – the AGW conventional sintered with liquid phase, b) - AGW sintered in 2 steps, including a sintering with liquid phase at 1500 °C for 60 min.

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#### Studiul microstructurii aliajelor grele pe baza de wolfram sinterizate in faza solida si cu faza lichida

**Rezumat:** Aliajele grele pe bază de wolfram cu compoziție 90W-7Ni-3Fe au fost omogenizate timp de 2 ore și compactate bilateral într-o matriță rigidă, după care s-a realizat sinterizarea în fază solidă la 1275 °C, timp de 1h, în curent de hidrogen. Dimensiunile particulelor de wolfram au fost de aproximativ 3,6 µm și densitatea lor aparentă de 4 g/cm<sup>3</sup>. După sinterizarea în fază solidă, aliajul greu a fost sinterizat cu fază lichidă la 1500 °C timp de 30-60 min. Microstructura aliajului greu pe bază de wolfram, sinterizat în 2 faze, indică faptul că particulele sferice de W sunt mai fine la sinterizarea în stare solidă decât la sinterizarea cu fază lichidă, având o maleabilitate de 15 % și o rezistență la întindere de 900 MPa.

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