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IMPROVING MECHANICAL PROPERTIES OF SINTERED WOLFRAM BASED ALLOY WITH LIQUID PHASE TROUGH CONTROLLED COOLING PARAMETERS

POPRAWA WŁASNOŚCI MECHANICZNYCH STOPÓW WOLFRAMU SPIEKANYCH Z UDZIAŁEM FAZY CIEKŁEJ POPRZEZ KONTROLOWANE CHŁODZENIE

Heavy alloys with 90 and 93 w/o W and a 7:3 Ni:Fe ratio are usually produced by the liquid phase sintering of W, Ni, and Fe powder mixtures. The result is a two-phase microstructure of spherical W solid solution grains embedded in a matrix of Ni rich solid solution. UTS, elongation, and microstructure strongly depend on the composition of the atmosphere during liquid phase sintering, on the cooling conditions and/or on the composition of the protective atmosphere during the heat treatment and its cooling conditions if treatment was applied after sintering. The sintering atmosphere is usually hydrogen. This can assure a proper densification but in the same time it can give rise to embitterment of W/matrix boundaries accompanied by a drastically decreasing ductility. In order to avoid this effect, heat treatments are applied in a neutral atmosphere (Ar, N_2, Ar+N_2, etc.) to completely or partially remove the hydrogen from the sintered material. This paper studies the effect of protective atmosphere and cooling conditions on the UTS, elongation and microstructure of the above-mentioned two heavy alloys.

Keywords: Tungsten heavy alloy (THA), W-Ni-Fe composites, liquid phase sintering (LPS), sintering atmosphere

Ciężkie stopy o zawartości 90 i 93%.wag W i stałym stosunku Ni do Fe wynoszącym 7:3 są zwykle wytwarzane przez spiekania z udziałem fazy ciekłej mieszaniny proszków W, Ni i Fe. Rezultatem jest dwufazowa mikrostruktura sferycznych ziaren roztworu stałego W, osadzonych w matrycy bogatego w Ni roztworu stałego. Wytrzymałość na rozciąganie, wydłużenie, i mikrostruktura silnie zależą od składu atmosfery podczas spiekania z udziałem fazy ciekłej, od warunków chłodzenia i/lub składu atmosfery ochronnej podczas obróbki cieplnej i warunków chłodzenia, jeśli zostało zastosowane po spiekaniu. Spiekanie prowadzone jest zwykle w atmosferze wodoru, która zapewnia odpowiednie zagęszczenie, ale jednocześnie może doprowadzić do zadrażnień na granicach ziaren W/osnowa, czemu towarzyszy drastycznie zmniejszenie ciągliwości. Żeby uniknąć tego efektu, obróbka cieplna prowadzona jest w atmosferze obojętnej (Ar, N2, Ar + N2 itp.), żeby częściowo lub całkowicie usunąć wodór ze spiekanego materiału. W pracy badano wpływ atmosfery ochronnej i warunków chłodzenia na wytrzymałość na rozciąganie, wydłużanie i mikrostrukturę wyżej wymienionych stopów.

1. Introduction

The mechanical behaviour of the tungsten heavy alloys (THA) from the W-Ni-Fe system is usually evaluated by tensile strength and elongation, giving a special feature consisting of the fact that in determined processing conditions both tensile strength and elongation could be simultaneously obtained at high level values. The above mentioned THA behaviour is based on the fact that these materials are two-phase composites of inclusion microstructure type, in which the included phase consists of tungsten and the matrix of a W-saturated Ni-Fe solid solution. The W-phase is hard, refractory, with high mechanical strength, while the matrix is more ductile, depending of composition and processing parameters. The performance of THA, as composite materials, depends on the exploiting degree of the intrinsic phase characteristics and on the strength of phase connection.

So, it can be said that the bulk mechanical response of a heavy metal composite has the following components [1], [2], [3]:

- Intrinsic strength of the W-spheroids;
- Strength and ductility of the binder (matrix) phase;
- Strength of the interfaces:
  - W/matrix;
  - W/W;
  - matrix/matrix.

The most important and critical component that determines a more or less plenary manifestation of intrinsic

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mechanical properties of structural phases is the quality of W/matrix interface (its integrity or the presence of porosity, impurities, gas inclusions, etc.). There are a lot of factors that can influence the W/matrix interface quality like composition, the purity of the original powders and technological parameters of THA processing. The cooling conditions and the type of protective atmosphere are the most important.

Therefore, the aim of this paper is to study the influence of cooling conditions after sintering and heat treatment on the tensile strength, elongation and microstructure of two THA compositions from the W-Ni-Fe system having 90 W, respectively 93 w/o W and a 7:3Ni:Fe ratio from the multitude of processing parameters.

The cooling conditions were chosen as research topic because according to several authors including the author of this paper, these conditions could affect the characteristics of the W/matrix interface the most [4], [5], [6].

Besides the above-mentioned aspects it could be yet underlined that THA are obtained by MP procedures. Sintering is a special procedure, a so-called liquid phase sintering. But, the presence of a liquid phase by sintering determines specific reactions with sintering atmosphere, depending on the type of atmosphere. The microstructure of the resulted THA and their properties are strongly dependent on the reactions between the liquid phase and sintering atmosphere. Gas solubility in the liquid phase and its diffusivity in liquid phase, as well as the possibility of removing dissolved gases during the cooling period from sintering to room temperature are especially important [7], [8].

2. Experimental part

Two THA compositions were chosen for this study: 90W-7Ni-3Fe and 93W-4,9Ni-2,1Fe, starting from the elemental powder mixtures of W, Ni, Fe. The powder characteristics, the blending and pressing parameters were presented elsewhere [9], [10].

Specimens for tensile testing and metallographic analysis were prepared from the two powder mixtures. After pressing, the specimens were presintered and sintered in a tunnel-type furnace in hydrogen atmosphere, applying the diagrams schematically presented in Fig. 1.

After sintering at 1500°C, 1 h in H_2 followed by quenching directly in the cooling zone of the furnace, the specimens were tested on the ZD-10 universal testing machine to measure UTS and elongation (5 specimens/composition). Metallographic specimens were prepared as well by a method presented in [7].

The remaining specimens, 15/each composition were divided into 3 lots for different heat treatments, to remove the dissolved hydrogen. 3 variants of heat treatments in neutral atmosphere were applied (TT_1, TT_2, TT_3) presented herein by the schematic diagram in Fig. 2.

The effect of these heat treatments on tensile properties of the two THA, 90W-7Ni-3Fe and 93W-4,9Ni-2,1Fe, was monitored with the aid of UTS, elongation and microstructures of THA specimens.

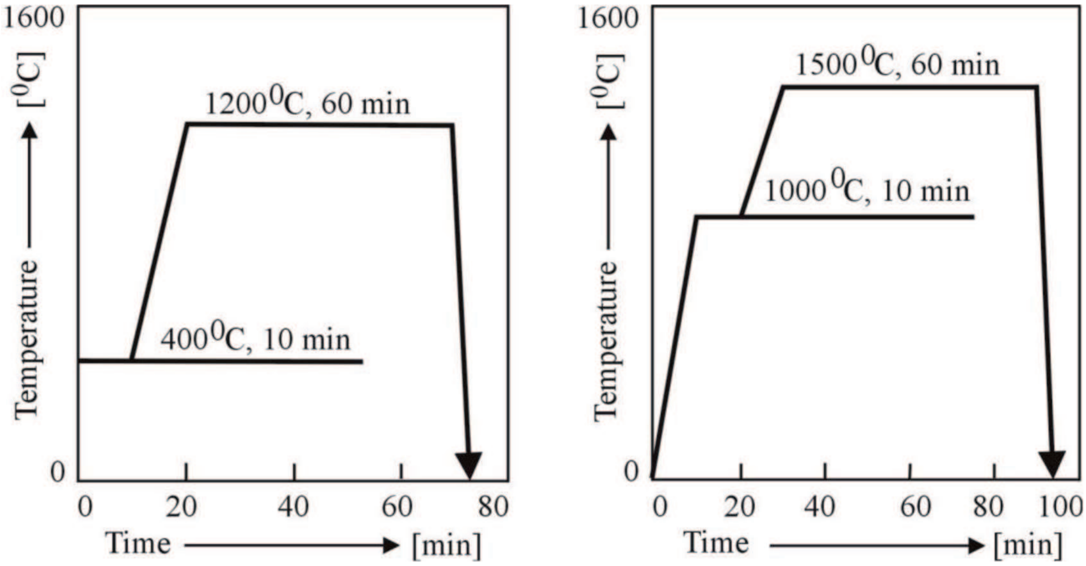


Fig. 1. Schematic presintering and sintering diagrams for 90W and 93W – THA specimens

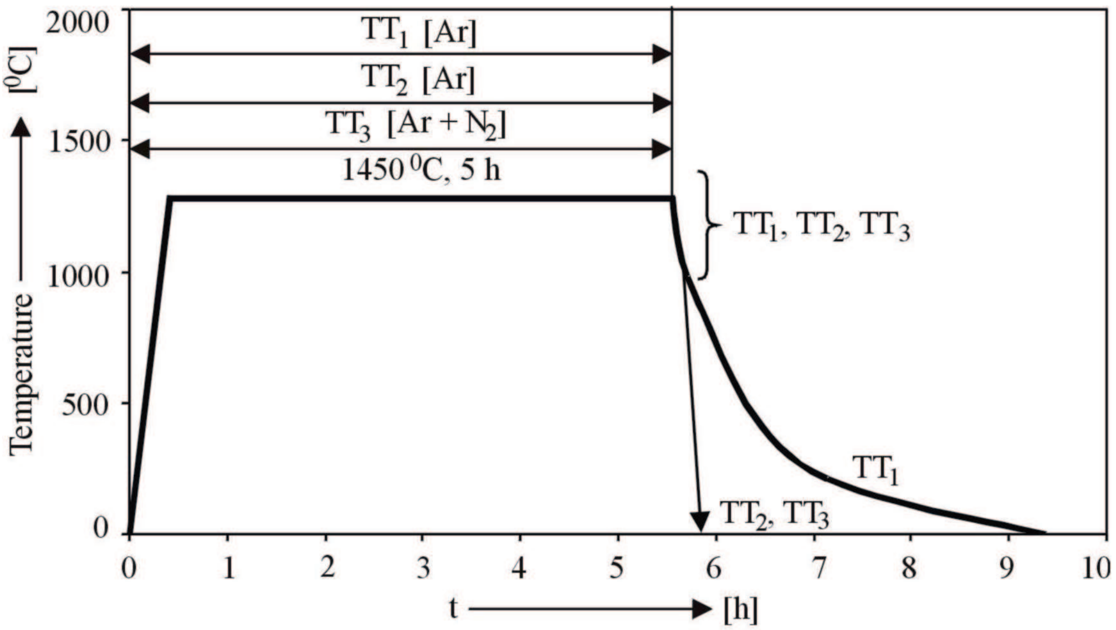


Fig. 2. Diagram for the thermal treatments applied after sintering to the samples made of AGW 90W-7Ni-3Fe and 93W-4,9Ni-2,1Fe

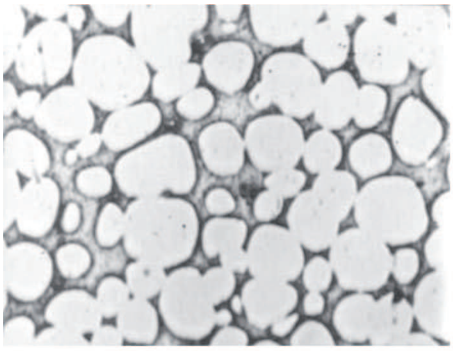


Fig. 3. Microstructure of THA 90W-7Ni-3Fe after sintering. Murakami's etch

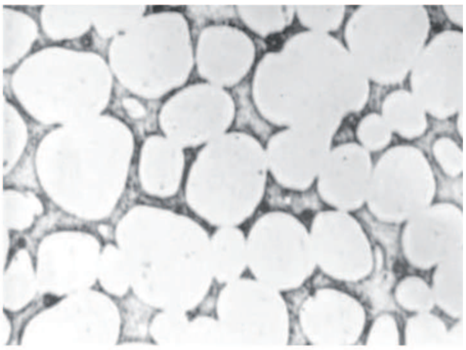


Fig. 4. Microstructure of THA 93W-4,9Ni-2,1Fe after sintering. Murakami's etch

3. Results

Table 1 presents the values of tensile strength (UTS) and elongation measured on the specimens of 90 W and 93 W THA after sintering and figures 3 and 4 show their microstructures.

The effect of the heat treatments, that is of the protective atmosphere and the cooling mode is given by the histograms in Figures 5 and 6 and could be observed on microstructures in Figures 7 and 8.

TABLE 1

Tensile strength and elongation for THA 90W-7Ni-3Fe and 93W-4,9Ni-2,1Fe

THA compositions	Sintering conditions	UTS, [MPA]*	Elongation, [%]*
90W-7Ni-3Fe	T_s =1500 ^{0}C, t_s =60 min, atm. H_2	740	1,4
93W-4,9Ni-2,1Fe		850	11

Remarks: * average values

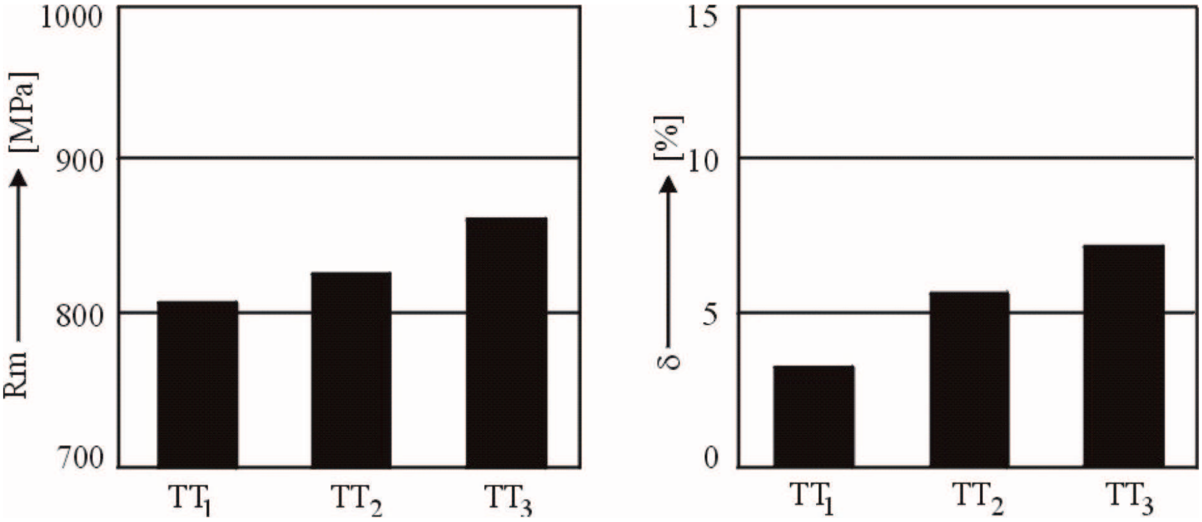


Fig. 5. UTS and elongation of the THA 90W-7Ni-3Fe after different heat treatments

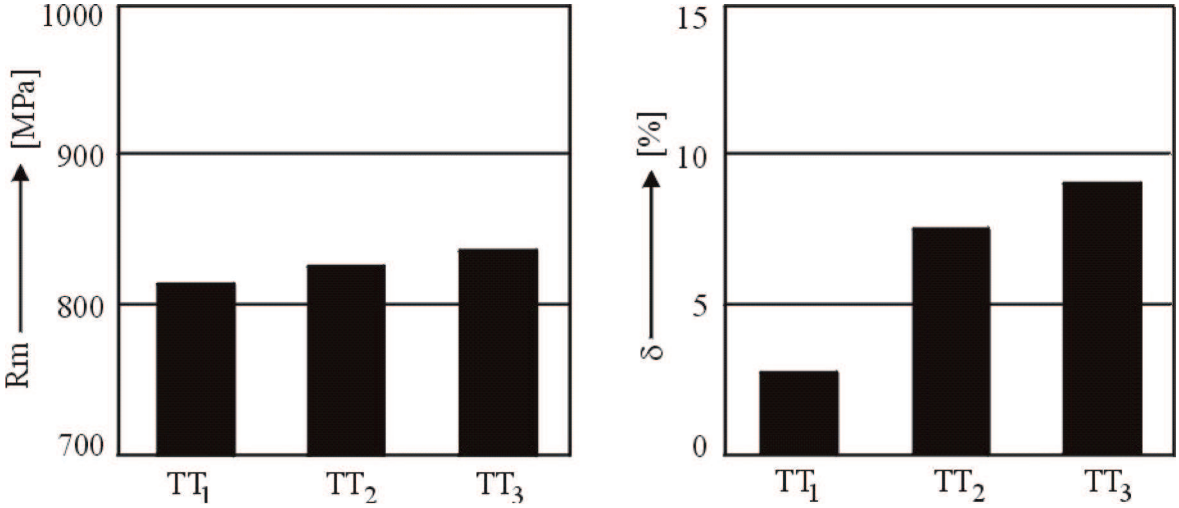
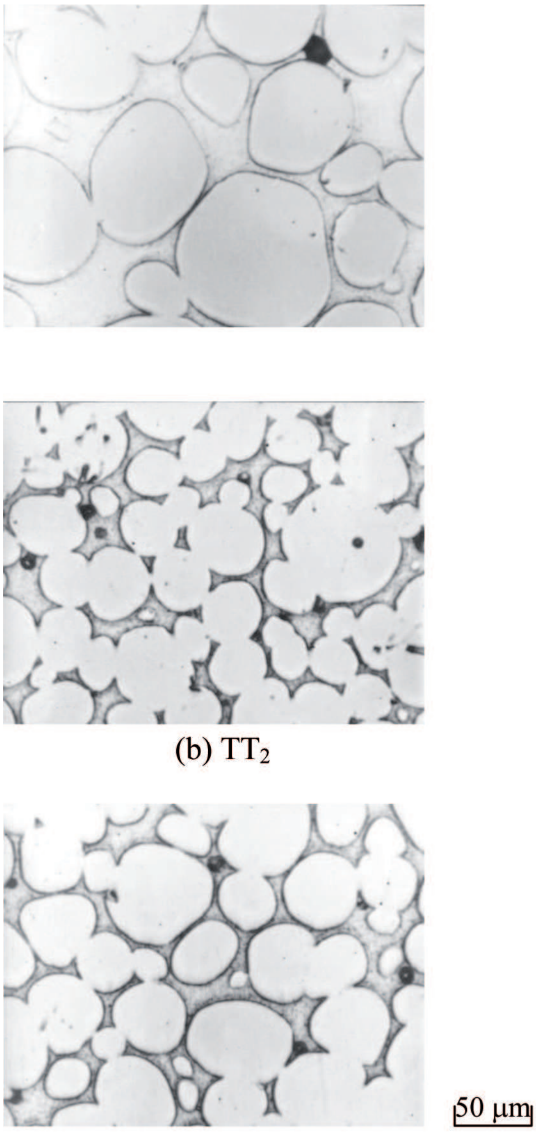


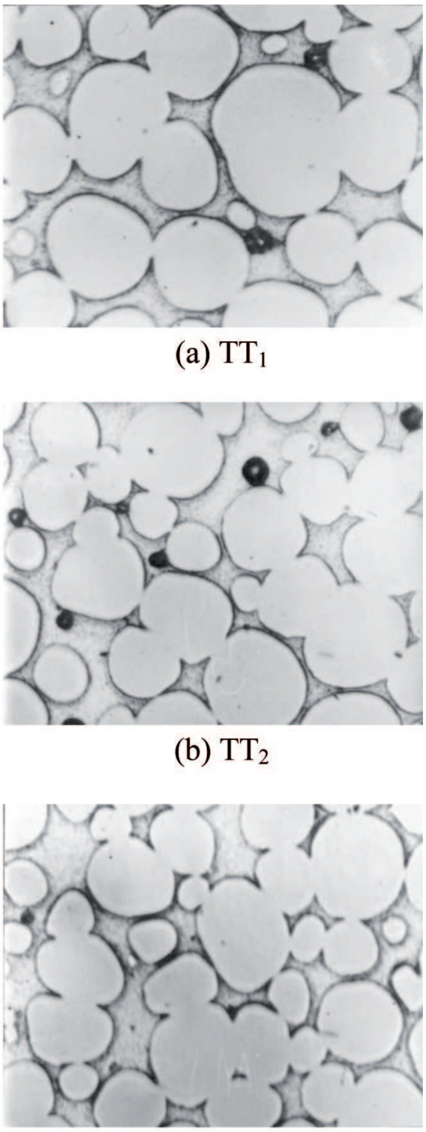
Fig. 6. UTS and elongation of the THA 93W-4,9Ni-2,1Fe after different heat treatments



(b) TT_2

(c) TT_3

Fig. 7. Microstructure of 90W-7Ni-3Fe after heat treatments in different conditions. Murakami's etch



(a) TT_1

(b) TT_2

(c) TT_3

Fig. 8. Microstructure of 93W-4,9Ni-2,1Fe after heat treatments in different conditions. Murakami's etch

4. Discussions

The UTS and elongation values after sintering were smaller at 90W THA in comparison with those of 93 W THA, although a smaller W content assures higher ductility. The cause of this result could be an excessively high sintering temperature for 90W THA determining an increase of W-contiguity, on the one hand and a greater quantity of dissolved H_2 in liquid matrix, on the other hand, resulting in a drastically decreasing of THA-elongation.

The three thermal treatments applied after sintering on THA have different effects upon the microstructure development and implicitly on the mechanical properties. Thus, TT_1 treatment performed in Ar atmosphere, due to the long cooling duration, has determined a more accentuated growth of the W-spheroids-size, alongside with a negative influence of mechanical properties. The best results for mechanical properties and microstructure could be obtained with TT_3-treatment in which an $(Ar+N_2)$-atmosphere and the two step-cooling were used, the first being the cooling together with the furnace followed by quenching.

From the two heavy alloys that with 93 W showed the highest property degree, because the other alloy was sintered at an excessively high temperature that resulted in a gravitational segregation of the phases.

5. Conclusions

- The application of some thermal treatments in neutral protective atmosphere (Ar, N_2, Ar+N_2) after sintering with liquid phase in hydrogen of THA with 90 W and 93 W, has improved their mechanical properties by removing the hydrogen that was dissolved in material at LPS.
- A slow cooling in the furnace to room temperature gave rise to an enhanced growth of W-spheroids, determining the decrease of mechanical properties of both THAs.
- The best values of mechanical properties were obtained by TT_3-treatment with a two-step cooling: the first in the furnace till 1000°C, followed by quenching, in a mixed atmosphere of Ar+N_2.

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