W-Cr and W-Ti alloys sintered by field assisted sintering with and without carbon diffusion barrier.

Monika Vilémová, Jakub Veverka, František Lukáč, Ksenia Illková

Institute of Plasma Physics, Academy of Sciences of the Czech Republic, Za Slovankou 1782/3, 182 00 Praha, Czechia

**vilemova@ipp.cas.cz*

Highlights

- W-Cr and W-Ti alloys were prepared by mechanical alloying
- W or C foil was placed between sintering mold and consolidated powder
- Significant difference in microstructure and properties was observed between samples sintered in W and C foil

1. Introduction

Tungsten is a refractory metal, widely used in demanding industrial applications, usually in high-temperature environment e.g. heating elements, turbine components, propellers or missile components. This stems out of its high melting point, good thermal conductivity and high strength at elevated temperatures. Recently, tungsten and tungsten alloys are extensively researched for application in thermonuclear fusion owing to the low sputtering yield, low tritium retention and low activation. Nevertheless, tungsten possesses several properties that are not optimal and must be considered during the components design. Significant brittleness at room temperature, along with high ductile-brittle transition temperature is serious drawbacks of tungsten. In addition, the recrystallization temperature is much lower than the melting point and tungsten suffers from rapid oxidation. To overcome these disadvantages, two adjustments of the fabrication process can be made – alloying of tungsten or employing a method known as “Field Assisted Sintering Technique” – FAST, or commonly “Spark plasma sintering” – SPS [1, 2].

For the compaction using SPS, powder (e.g. prepared by mechanical alloying) is poured into graphite die, which is then placed between graphite punches. Conductive foil (commonly graphite) is often placed in the die before pouring of the powder. After that, electric current and pressure is applied through the punches and the powder is compacted. Primary objective of the foil is to prevent the powder adhesion to the die. However, for the examination of the compacts, surface layer affected by the foil is to be grinded off. The experiments have also shown that at elevated temperatures, significant carbon diffusion from the die occurred. This is undesirable, as carbon changes tungsten microstructure, phase composition and worsens mechanical properties.

Generally, very little is known about possibilities of diffusion barrier application and their effect on the sintering process for various materials. Carbon diffusion control might be of highest interest especially for tungsten alloys containing elements with high carbon affinity. In the presented study, two tungsten alloys – W-10wt.% Cr-1wt.% Hf and W-7wt.% Ti-2wt.% Hf - prepared by mechanical alloying were compacted by means of SPS. The effect of foil material (potential diffusion barrier) on the microstructure and basic mechanical properties of the compacts was examined. The behaviour during sintering in different foils was explained using differential thermal analysis and XRD analysis.

2. Methods

The W-Ti and W-Cr powder batches of various compositions (see Table 1) were prepared in a planetary ball mill Pulverisette 5 (Fritsch, Germany). The starting powders were W (99.9% purity, 1.2 µm average powder size), Ti (99.4% purity, 5 µm average powder diameter size), Hf (bimodal powder diameter size distribution, 15 µm and 45 µm) and HfC (bimodal powder diameter size distribution, 5 µm and 20 µm). The powders were loaded in tungsten carbide bowls with tungsten carbide grinding balls in the ball to powder ratio (BPR) 11:1. The sealed bowls were flushed with high purity argon in order to prevent oxidation during the milling process.

The powders were consolidated by pulsed electric current sintering machine SPS 10-4 (Thermal Technology, Santa Rosa, CA, USA) under similar sintering conditions, i.e., sintering temperature 1750 C, pressure of 70 MPa, vacuum of 10 Pa/helium atmosphere of 60 Pa and sintering time of 3 min. Sintering was performed in graphite molds and graphite foils or tungsten foils.

3. Results and discussion

Figure 1 shows the microstructure of the sintered tungsten alloys. The use of foil material is indicated in figure above respective micrographs. Comparing the microstructures it is apparent that foil material significantly influences the result for W-Cr as well as for W-Ti. Most likely the reason for the different results is carbon diffusion into the material during sintering. Further details will be provided and discussed during the workshop.

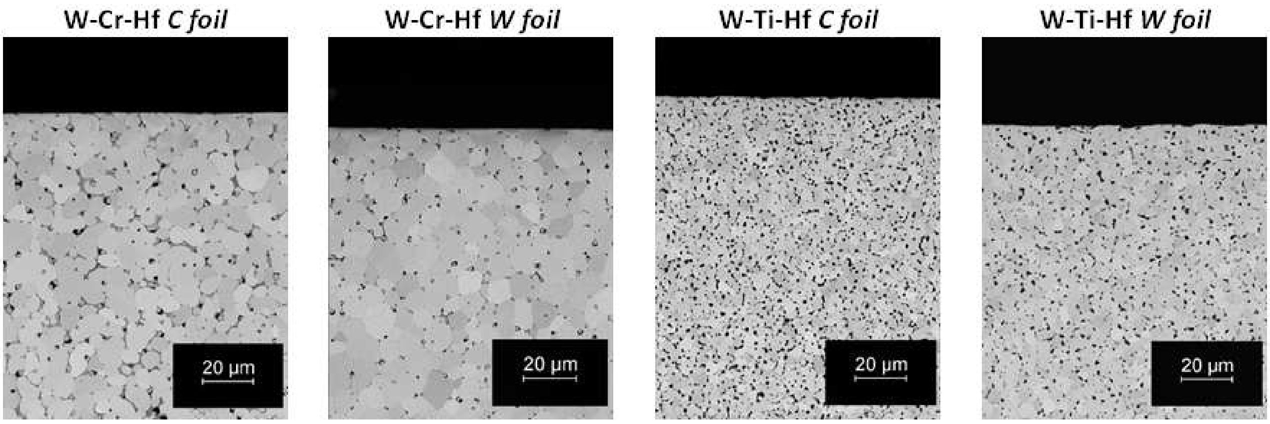

Figure 1. Microstructure of the W-Cr and W-Ti alloys sintered with and without diffusion barrier..

4. Conclusions

W-Cr and W-Ti alloys were sintered using field assisted sintering method. Effect of sintering with two different sintering foils was studied and evaluated. Following conclusion can be drawn:

- Type of sintering foils placed between the mold and powder significantly influences microstructure and properties of resulting compacts.
- Sintering in carbon mold /foil causes in depth carbon contamination.
- Of concern are especially W-Cr and W-Ti alloys due to very little carbon solubility in W, limited solubility of Cr and Ti in W and high affinity of Cr and Ti to C.
- Sintering of W-Cr and W-Ti in carbon molds/foil leads to formation of Cr and Ti carbides.
- Application of carbon diffusion barrier during sintering in a form of W foil placed between the carbon mold and compacted powder minimizes or completely avoids carbide formation.

References

1. A. Litnovsky, T. Wegener, F. Klein, Ch. Linsmeier, M. Rasinski, A. Kreter, B. Unterberg, J. W. Coenen, H. Du, J. Mayer, C. Garcia-Rosales Nucl Fusion. 57 (2017) 66020.
2. A. Calvo, N. Ordás, I. Iturriza, et al. Phys Scr. T167 (2016) 14041.

Keywords
Tungsten alloys; tungsten; carbon contamination; diffusion barrier; sintering foil

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