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Full Length Article

# Microstructure and thermal properties of mechanically alloyed W-1%TiC powder consolidated via two-step HIPping

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ABSTRACT

In this work an influence of a fabrication route on the microstructure, thermal and mechanical properties of the W-1%TiC was studied. The W-1%TiC powder was mechanically alloyed using the stainless steel milling media. Two-step HIPping of the powder at 1300 °C (1573 K) and then at 1750 °C (2023 K) was performed. After second-step HIPping, in spite of 13.5% of porosity, bending stress and strain values were 300 MPa and 2%, respectively. On the contrary, the grain growth (from 0.7 up to 22.0 μm) and coarsening mechanisms of the TiC nanoparticles were observed. Up to 1200 °C (1473 K), thermal expansion parameters of two-step HIPped W-1%TiC alloy exhibited thermally unchanged values, comparable with a pure W rod, however, the thermal conductivity of the W-TiC alloy at RT was 26.5 W/m K, and at 1200 °C (1473 K) this parameter increased up to 44.8 W/m Km K.

## 1. Introduction

Tungsten (W) and W refractory alloys are promising candidates for structural materials and plasma-facing components of the future fusion reactor, owing to their good refractory and high temperature properties, high thermal conductivity, low thermal expansion coefficient as well as low activation under neutron irradiation [1–3]. Out of the various W alloy compositions, the W-TiC refractory alloy seems to have properties that are sufficient for the application in question [4–6]. According to many authors, the TiC constituent improves embrittlement of pure tungsten by strengthening interface bonding between tungsten particles and the matrix [7,8]. However, the high ductile-to-brittle transition temperature (DBTT) of the body-centred cubic (BCC) phase structure of tungsten and tungsten alloys are still an issue to be solved as far as thermonuclear reactor application is concerned.

At present, powder metallurgy (PM), particularly mechanical alloying (MA), is broadly applied in manufacturing of W refractory alloys [4–6,9]. One of the problems encountered during the process is contamination coming from the milling system (jar and balls). WC-Co and Ti-Zr-Mo (TZM) are the materials commonly used for this purpose [3,10,11]. WC-Co jar and balls are widely applied in W alloy manufacturing, this system, however, is inadequate for the proposed application. Cobalt, a highly activated element, transmutes easily under neutron irradiation and therefore does not fulfil the low activation requirements. Since the TZM milling system is commercially unavailable, it is difficult to obtain milling jar and balls made from this alloy. To solve this issue, the authors decided to use a common stainless steel (Fe-14%Cr-0.5%C) milling system to analyse its influence on the composition, powder consolidation, microstructure and second phase precipitations as well as mechanical properties of the W-TiC alloy. It is also expected that Fe and Cr elements may facilitate liquid phase sintering of the W-TiC powder, whereas Cr may also improve oxidation resistance and thus enhance the properties of the alloy. Since quaternary phase diagrams of W-Fe-Cr-TiC are complex it is impossible to predict whether this approach is feasible on the basis of purely theoretical considerations.

Thus, an experimental study was performed to develop of a dispersion strengthening W-Fe-Cr-TiC composite alloy as an alternative material for the W-TiC (in wt.%) alloy through mechanical alloying of W and TiC powders in a stainless steel milling system. A metallurgical aspect of the influence of Fe, Cr on the W-TiC alloy manufactured by means of two different powder metallurgy (PM) routes; mechanical alloying, cold compaction and sintering vs. HIPping is reported and discussed. The proposed W-Fe-Cr-TiC composite material can be considered as a structural element in the European helium-cooled divertor concept [12]. On the basis of the results presented by Kurishita et al. [5,13], 1% of TiC was chosen to ensure grain refinement of W during high temperature HIPping.

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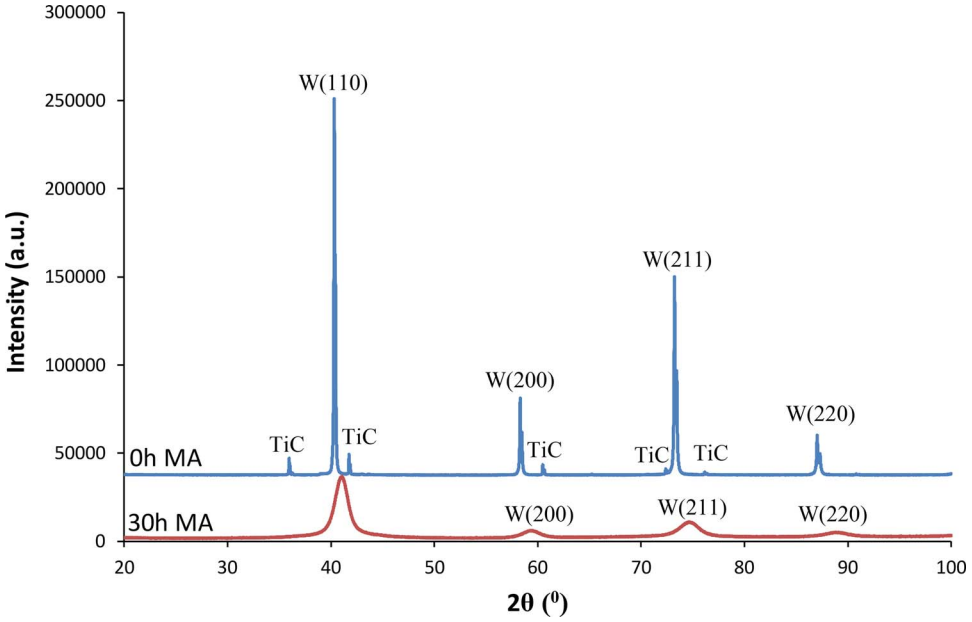


**Fig. 1.** XRD pattern of the W-1%TiC powder MA up to 30 h.

## 2. Material and methods

Pure tungsten powder (99.9% purity, metal base) with an average particle size of 2.5 μm and 1% of TiC (in wt.%) was mechanically alloyed in a planetary ball mill (Pulverisette 5, Fritsch) in a stainless steel system (ball diameter of 10 mm) at 380 rpm for 30 h. An average particle size of the TiC powder (99.5% pure) was ~5 μm. The ball-to-powder weight ratio (BPR) was 8:1. To prevent oxidation, the milling jar was filled with pure argon (99.999). Loading and unloading of the powders were performed under a glove box protected under argon atmosphere. Particle size distribution was measured by means of a Laser Particles Sizer Analysette 22 (Fritsch), using the dry method.

The time of MA was optimized by studying the structural evolution of the powder by means of X-ray diffractometry (XRD), D8 ADVANCE, Bruker, using Cu-Kα X-ray. Crystallite size was determined by measuring the Bragg peak width at half the maximum intensity (HWFM) and by using the Williamson-Hall method [14]. The MA process was conducted until the solute elements' peaks in X-ray diffraction patterns disappeared.

The W-1%TiC powder was cold compacted into a cylinder of 20 mm in diameter and 6–8 mm in height, using a uniaxial hydraulic press, with a compaction pressure in the range of 150–350 MPa. No lubricant was applied. The density of the green compacts was measured using the geometrical and weight methods. Sintering at 1250 °C (1523 K), for 2 h, in a $10^{-3}$ Pa vacuum with an average heating and cooling rate of 350 °C/h were utilized. The W-1%TiC powder after MA was also consolidated by means of HIPping. Prior to HIPping, the powder was degassed at 450 °C (723 K) for 2 h under a vacuum of $10^{-2}$ Pa and closed in a low-carbon steel capsule. A two-step HIPping process was applied, first at 1300 °C (1573 K) and then at 1750 °C (2023 K), under an isostatic pressure of 300 MPa, for 3 h. The heating rate for HIPping was similar to the sintering process, whereas the cooling rate was 600 °C/h (873 K/h). The second-step HIPping, at 1750 °C (2023 K), was performed after removing the steel capsule. The density of the HIPped material was measured by the Archimedes method.

The microstructure and composition of the as-sintered and HIPped specimens were observed using the SEM-EDS (Hitachi 3500), SEM-FIB Hitachi NB5000, and STEM Hitachi HD2700. Three-point bending tests at room temperature (RT) were performed using specimens with a span of 18 mm, a cross section of $3 \times 4\ mm^2$ and a strain rate of $1 \times 10^{-3}\ s^{-1}$. Bending stress and strain were calculated according to ASTM Standard [15]. Fracture surfaces were examined by SEM. Vickers hardness (HV) tests were performed under loads of 0.98 N and 98.1 N.

The W–W contiguity of the HIPped specimens was observed by SEM and calculated using the equation given by [16].

The thermal expansion coefficient of the W-1%TiC was measured by the dilatometric method (DIL 402E Netzsch Gerätebau GmbH, Germany). A pure tungsten road was used as a reference. The dimensions of the specimens were 6 mm in diameter and 14 mm in length. Linear changes in the temperature function of W and W-1%TiC were measured under helium, in a graphite sample holder with a heating rate of 2 K/min.

Thermal diffusivity was measured by the laser flash method (LFA 427 Netzsch Gerätebau GmbH, Germany), where the bottom side of the plane-parallel sample was heated by a short energy pulse coming from neodymium laser and the increase of temperature was observed on the upper side of the sample and measured by an infra-red detector. The measurements were performed in argon, using samples with a diameter of 10.0 mm and a thickness of 2.0 mm, in the range of RT to 1400 °C (1673 K). Thermal diffusivity (TD) was determined with the use of the Eq. (1) given by Parker et al. [17]:

$$\alpha = 0{,}1388 \cdot L^2 / t_{0,5} \quad (1)$$

where: $t_{0,5}$ is the time required for back surface temperature rise to reach half of its maximum, $L$ – sample thickness.

Thermal conductivity (TC) was calculated on the basis of the following formula, equation (2):

$$\lambda = \rho \cdot C_p \cdot \alpha \quad (2)$$

where: λ – thermal conductivity, α – thermal diffusivity, ρ – density of the measured sample, $C_p$ – specific heat of the material.

These results represent the average value of three independent tests.

## 3. Results and discussion

### 3.1. Mechanical alloying process

The XRD plots of the W-1%TiC powder in the initial state and after 30 h of mechanical alloying presented in Fig. 1 confirmed that the MA process was completed. The only main peaks detected were those of W. This means that the solute elements from the milling system (Fe, Cr), as well as the TiC powder, dissolved in the W matrix. The crystallite size, calculated from the main XRD peak, was 15 nm, which is similar to the value reported by Ishijama [7].