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MICROSTRUCTURE CHARACTERIZATION OF TUNGSTEN BASED ALLOYS FOR FUSION APPLICATION

CHARAKTERYZACJA MIKROSTRUKTURY STOPÓW NA OSNOWIE WOLFRAMU PRZEZNACZONYCH DO ZASTOSOWAŃ W PROCESACH SYNTEZY JĄDROWEJ

The microstructure of two tungsten based alloys (W-1.1%TiC and W-1.7% TiC) was characterized using light microscopy, analytical electron microscopy and electron tomography. These alloys represent a class of W based dispersion strengthened alloys with TiC used as strengthening particles. Addition of TiC leads to improved creep resistance and tensile strength of the W based alloys. The results show that the W-1.7%TiC alloy exhibits large scatter in grain size, much higher porosity and contains also Ti-O particles. The W-1.1%TiC alloy has fine grained microstructure with uniformly distributed fine TiC particles within the matrix and low porosity. As a result of the different microstructure, the W-1.1%TiC alloy exhibits better mechanical properties, when compared to the W-1.7%TiC alloy.

Keywords: fusion, tungsten based alloys, electron microscopy, electron tomography

W pracy przedstawiono wyniki badań mikrostruktury dwóch stopów wolframu: W-1.1%TiC oraz W-1.7%TiC. Badania przeprowadzono z wykorzystaniem technik mikroskopii świetlnej, analitycznej mikroskopii elektronowej oraz tomografii elektronowej. Przebadane stopy reprezentują grupę stopów wolframu umacnianych dyspersyjnie cząstkami TiC. Dodatek TiC poprawia odporność na pełzanie oraz wytrzymałość tych stopów. Badania wykonane dla stopu W-1.7%TiC wskazują na duże różnice w wielkości ziarna, dużą porowatość oraz występowanie cząstek TiO. Stop W-1.1TiC posiada drobnoziarnistą strukturę, niską porowatość oraz równomiernie rozmieszczone cząstki TiC w osnowie W. Stop W-1.1%TiC charakteryzuje się znacznie lepszymi właściwościami mechanicznymi niż stop W-1.7%TiC.

1. Introduction

Tungsten based materials, presenting interesting combination of functional and mechanical properties, gained in importance with the start of the International Thermonuclear Experimental Reactor (ITER) project [1-3]. ITER project should demonstrate feasibility of gaining energy by fusion processes. Tungsten and its alloys are proposed for structural application and plasma facing material (e.g. in the divertor) due to their high melting temperature (3422°C), thermal conductivity (174 W/(m·K)) and low erosion caused by isotopes of hydrogen [1]. The function of the divertor is to extract heat and ashes – products of fusion reaction from the plasma and therefore it will be exposed to high heat loads. Due to extremely high temperature (up to 3000°C), there are only few materials considered as the surface part of divertor plasma facing elements: carbon fibre-reinforced carbon composite and tungsten or tungsten alloys. Currently, due to costs, it is planned to use tungsten as a plasma facing part of the divertor [1].

For tungsten based alloys, as structural materials, there are two important temperatures: Ductile to Brittle Transition Temperature (DBTT) which occurs between 100-600°C and recrystallization temperature (T_R) [1-4]. Those temperatures are limiting factors for the application of these alloys. In order to increase W-base alloys high temperature strength, the strengthening particles (dispersoids) might be added to the alloy matrix, but their introduction should not increase DBTT [3]. From selected dispersoids, such as La_2O_3, Ce_2O, ThO_2, HfC and TiC, only TiC was reported to improve the ductile behaviour [3], therefore tungsten based alloys strengthened by addition of TiC particles are promising materials for fusion applications.

The aim of the performed investigations was microstructural characterization of tungsten based alloys strengthened by addition of TiC particles. Two alloys with different amount of TiC dispersoids were chosen for detailed analyses: W-1.7%TiC and W-1.1%TiC. The W-1.1%TiC alloy exhibited higher ductility [5] at similar temperatures, when compared to W-1.7%TiC alloy.

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2. Materials and experimental details

Two tungsten based alloys were investigated: W-1.1%TiC and W-1.7%TiC (wt%). Both alloys were produced by mechanical alloying and sintering. The W-1.7%TiC alloy was produced using classical mechanical alloying of powders in Ar atmosphere, following by hot isostatic pressing (HIP) and recrystallization annealing.

The W-1.1%TiC was produced using special procedure of mechanical alloying (MA), consisted of powder handling in H_2 followed by mechanical alloying in purified H_2. Then the powder was degassed. In the next step, the MA powder was HIP'ed at 1350°C. Finally the specimen was processed by so-called superplastically based microstructure control route (SPMM) in order to achieve the desired microstructure. This processing route should assure enhanced toughness of the alloy.

The microstructural investigation was performed by light microscopy (LM) as well as scanning- and transmission electron microscopy (SEM, TEM, respectively). Microanalysis of chemical composition was performed using energy dispersive X-ray spectrometry (EDS) using SEM and TEM. The SEM investigation was conducted using NEON 40EsB FEG CrossBeam of Zeiss equipped with EDX microanalysis system Quantax 200. The TEM analysis were performed by Tecnai G2 20 of FEI equipped with EDAX/TIA microanalysis system. Specimens for TEM observations were successfully prepared by electro-polishing in NaOH solution using TenuPol-5 of Struers. Preparation methods were described in detail in Ref. 6.

Image analysis of LM, SEM and TEM micrographs was conducted by AnalySIS 3.2 software.

Electron tomography investigation (FIB-SEM) was carried out using dual beam workstation equipped with a Focused Ion Beam (FIB) column (Ga ions) by means of NEON 40EsB FEG CrossBeam of Zeiss. Serial FIB sections in situ milling was performed by Ga-ion beam. For each slide, SEM images were taken with the ESB detector. The acquired stack of 300 images was transformed directly into a 3D data volume. The three-dimensional visualization of reconstructed volume of investigated material for FIB-SEM tomography was performed using ImageJ and Avizo Fire 6.3 software. The procedure of FIB-SEM tomography was described in detail in Ref. 7.

3. Results

W-1.7% TiC alloy

Fig. 1 shows the microstructure of the W-1.7%TiC alloy (Fig. 1) produced by a classical MA route revealed by LM. Roughly bimodal grain size (large grains as well as areas with small grains) and a high number of pores were observed. In the microstructure a large number of dispersoids was present, however majority of particles were identified as titanium oxides (not as TiC, as expected)

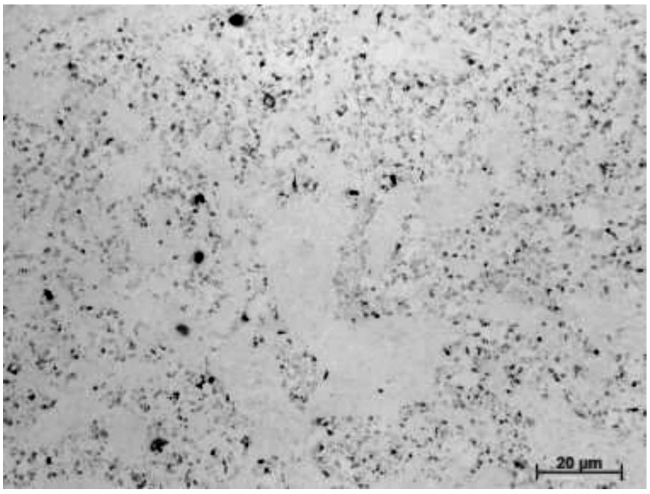

Fig. 1. Microstructure of W-1.7%TiC alloy, visible large scatter in grain size, LM

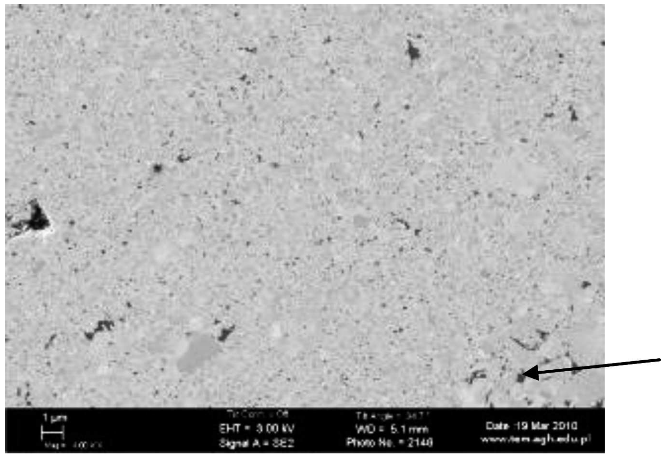

Fig. 2. Microstructure of W-1.7%TiC alloy. Large scatter in grain size, pores and particles, mainly oxides are visible, SEM

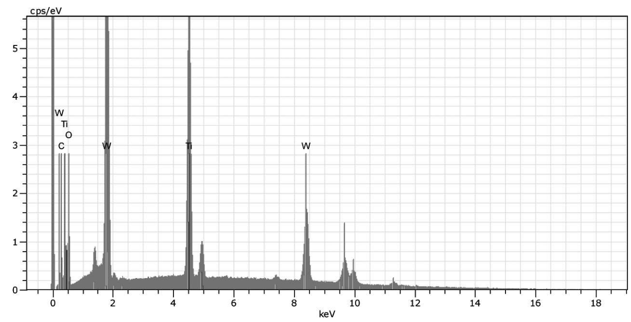

Fig. 3. SEM-EDS spectrum taken from particle marked with an arrow in Fig 2.

The microstructure of the alloy as seen by TEM is presented in Figs 4a,b. Tomographic reconstruction of the investigated volume (4.5×2.5×1.7μm^3) is presented in Fig. 5, where high porosity, areas of large grains and areas with small grains are visible.

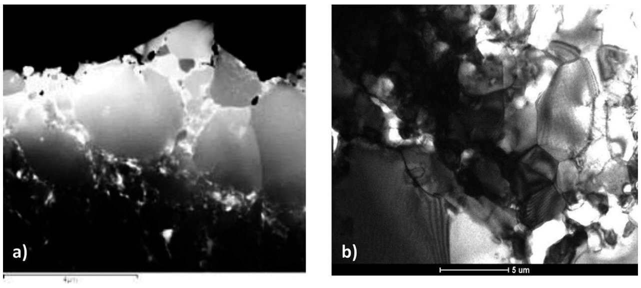

Fig. 4. Microstructure of the Ti-1.7%TiC alloy exhibiting large scatter in grain size, STEM

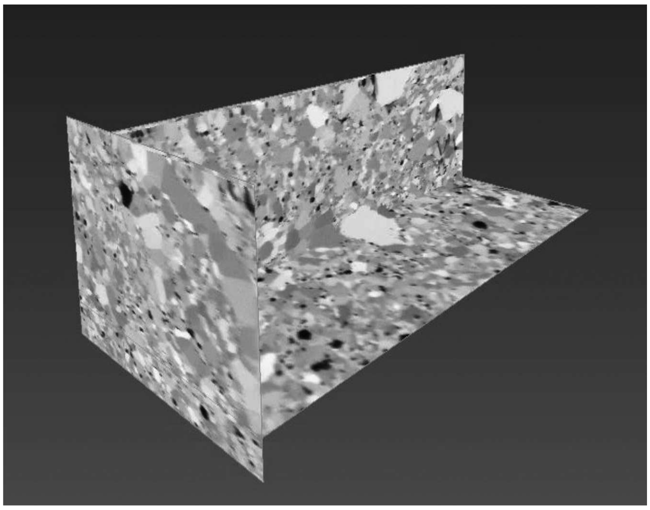

Fig. 5. Tomographic reconstruction of the Ti-1.7%TiC alloy

Based on FIB-SEM tomography results, the volume fraction (V_v) of particles was estimated to be V_v = 4.4±0.6%. and the mean diameter was measured for D=48.6±11 nm.

W-1.1% TiC alloy

Microstructure of W-1.1%TiC produced by SPMM route is presented in Figs 6a,b. Fine, fairly equal grain size and low number of pores were observed, especially when compared to previously described microstructure of W-1.7%TiC alloy (Fig. 1).

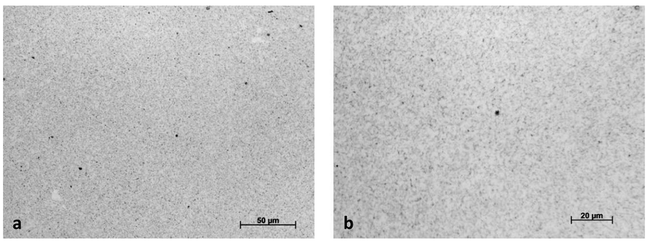

Fig. 6. Microstructure of the W-1.1%TiC alloy, fine microstructure with low porosity, LM

SEM investigation confirmed fine grained microstructure with high number of dispersoids (Figs 7a,b). Chemical composition of particles shown in Fig. 8a, investigated by TEM-EDS indicated for the presence of TiC (Fig. 8) however unambigous identification of all precipitates was not possible.

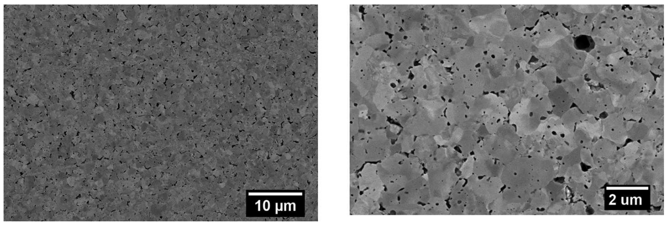

Fig. 7. Microstructure of the W-1.7%TiC alloy; high number of TiC particles within the W matrix, SEM

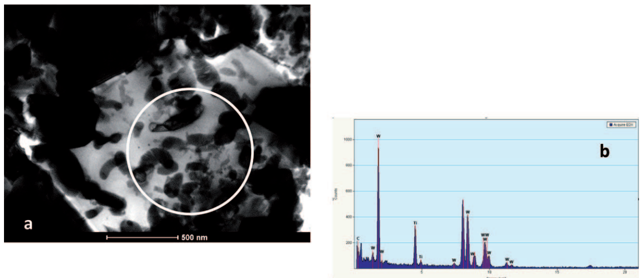

Fig. 8. Microstructure of the Ti-1.1%TiC alloy (a), visible large number of particles, TEM, and (b) corresponding EDS spectrum taken from marked area, indicating for TiC particles

FIB-SEM tomography revealed 3D microstructure of the investigated alloy and distribution of dispersoids (Figs 9 and 10).

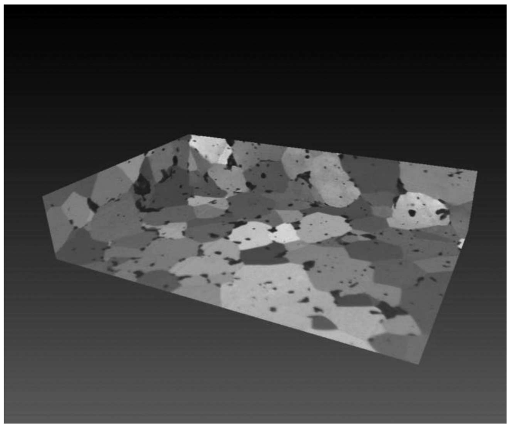

Fig. 9. Tomographic reconstruction of the W-1.1%TiC alloy

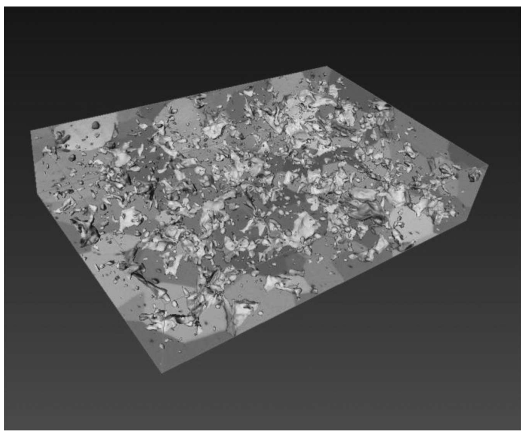

Fig. 10. Microstructure of W-1.1%TiC, reconstructed volume, large number of TiC particles is visible

Based on this investigation, the volume fraction of particles (dispersoids) was estimated to be V_v^{TiC} =3.5±1.3%. The mean diameter of TiC particles was measured as D=32.7±9 nm.

4. Summary

Two tungsten-base alloys were investigated using light microscopy, electron microscopy and electron tomography techniques. The main difference in their chemical composition was content of TiC particles used in the MA process. The processing routes of both alloys consisted of MA and HIP. For W-1.1%TiC alloy, additionally, superplastically based microstructure control process was applied. As the result, the W-1.1%TiC alloy exhibited a fine, equal grain size microstructure, consisting of W matrix and a large number of TiC dispersoids as well as low porosity.

The microstructure of W-1.7%TiC alloy was not so uniform comparing to W-1.1%TiC. It consisted of W matrix and dispersoids, which were identified mainly as TiO_2. Although the process of MA was carried in argon, only few TiC dispersoids were found. Areas with large grains and areas with small grains were observed, what suggests that composition of powders (particle size) used for MA of W-1.7%TiC alloy should be optimised.

Application of FIB-SEM tomography allowed for three dimensional imaging of the microstructure of both alloys. The volume fraction of TiC particles in the W-1.1%TIC alloy was estimated for 3.51±1.32%; a mean diameter of TiC was measured as D=32.7 nm. The volume of TiO_2 particles in the W-1.7% TiC alloy was estimated for 4.41±0.62%; a mean diameter of TiO_2 particles was established for D=48.6 nm.

The results of performed microstructural investigation, showing fine and uniform grain microstructure and low porosity of W-1.1%TiC alloy explains its better mechanical properties in comparison to W-1.7%TiC alloy.

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