Int. Journal of Refractory Metals and Hard Materials 61 (2016) 173–178

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

SANS characterization of particle dispersions in W-Ti and W-V alloys

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ARTICLE INFO

Article history:
Received 29 March 2016
Received in revised form 10 September 2016
Accepted 12 September 2016
Available online 13 September 2016

Keywords:
ODS alloys
Refractory metals
SANS
HIP

ABSTRACT

SEM analyses of W-Ti and W-V alloys produced by a powder metallurgy route have revealed the presence of Ti or V pools with different sizes and shapes. The larger pools have sizes between ~0.3 and several microns and are embedded between the matrix grains. The smaller Ti-rich (V-rich) particles, with sizes below ~0.3 μm, are spherical and are dispersed inside the matrix grains. The characteristics of the second phase nanoparticles have been studied using the SANS technique. The scattering curves have been analyzed in terms of a polydisperse particle system following the Beaucage approach, and the maximum entropy method to obtain the particle size distributions. Beaucage analyses suggest the presence of two particle populations, characterized by radii of gyration from 50 to 80 Å (population I) and between 650 and 760 Å (population II). The volume distribution obtained by the maximum entropy approach reveals a prominent peak with particle diameter of 60 Å and ~70 Å for the W-Ti and W-V alloys, respectively. Secondary peaks in the size range 100–300 Å could be associated with the particle population with the larger radius of gyration.

1. Introduction

An important issue in the design of the future fusion reactor DEMO is the materials selection for a helium-cooled system of the first wall and blanket. W and its alloys are among the most promising structural and armor materials for building plasma facing components (PFCs) [1–6]. For accomplish their functions with reliance the properties of these materials such as the thermal conductivity, mechanical behavior at high temperature, recrystallization temperature, the ductile to brittle transition temperature (DBTT) and ductility have to be enhanced. Nevertheless, recent studies have demonstrated the difficulty of developing W alloys for PFCs [7]. Different strategies have been approached for achieving enhanced ductility in W alloys: solid solution alloying, grain refinement and nanostructuring as well as the use of W composites [8].

In the last years, W and several W-Ti and W-V alloys, with grain sizes of hundreds of nanometers and densification very close to 100%, have been produced by mechanical alloying and consolidation by hot isostatic pressing (HIP) [9–11]. In spite of the submicron grained microstructure, and a possible dispersion of second phase nanoparticles, these alloys do not exhibit a significant ductility enhancement. For instance, the W-4Ti alloy did not exhibit any sign of plastic flow up to 1273 K while unalloyed W processed following the same route did it at 673 K. For W-4V the DBTT is also very high, between 1073 and 1273 K [9,12]. A relevant microstructural feature in the W-4Ti alloys that might give account for the mechanical behavior is the Ti segregation as large pools and dispersed particles with a wide range of sizes [13]. The same has been found for the W-V alloys despite the complete solid solution of V in W [11]. The aim of the present study has been to disclose the characteristics of the second phase nanoparticles dispersed in W-Ti and W-V alloys using the small angle neutron scattering (SANS) technique. As this technique samples a macroscopic volume of material, it provides a more reliable and complete information about the size distribution of submicron-sized particles in a matrix than others as high-resolution transmission electron microscopy and atom probe tomography. In order to obtain reliable quantitative results the SANS data have been analyzed applying the Beaucage unified model [14,15] and the maximum entropy approach [16,17].

2. Experimental procedures

W-*x*wt%Ti and W-*x*wt%V alloys ($x = 2$ and 4) were produced by a powder metallurgy route consisting in mechanical alloying of an elemental powder blend with the target composition and consolidation by HIP. The starting powders were: 99.9% pure W, 99.9% pure Ti and 99.5% pure V with particle sizes between 1 and 5 μm for W, <106 μm for Ti and <45 μm for V. The powder blends were mechanical alloyed for 20 h in a high-energy planetary mill. Then, the alloyed powder was canned and degassed, and consolidated for 2 h at 1575 K and 195 MPa. The details of the powder processing and consolidation have been reported elsewhere [11,13]. During the mechanical alloying X-ray diffraction measurements were performed on powder samples to

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http://dx.doi.org/10.1016/j.ijrmhm.2016.09.009
0263-4368/

monitor the alloying process. The oxygen content was also measured by IR absorption using a LECO ON836 analyzer. The density of the consolidated materials was determined with a He ultrapycnometer (Ultrapycnometer 1000). This device allows a very accurate volume measurement of the porous solids. This task is accomplished by employing Archimedes' principle of fluid displacement and the Boyle law for an ideal gas. The morphology of the raw W, Ti and V powders was analyzed with a PHILIPS XL-30 scanning electron microscope. The microstructure of the alloys was studied using a high-resolution HITACHI S4800 J scanning electron microscope equipped with an energy dispersive spectrometer (EDS). Fig. 1 shows the morphology of the starting elemental powders.

The SANS experiments were carried out in the KWS-2 spectrometer of the research reactor FRMII of the Jülich Centre for neutron Science (JCNS) at Garching (Germany) using a neutron wavelength of 7 Å. The samples had an area ~1.5 × 2.5 cm^2 and a thickness of ~2 mm. Data were acquired for sample-detector distance positions of 2, 8 and 20 m. These experimental conditions allowed cover the scattering vector range $0.002 < Q < 0.190$ $Å^{-1}$ corresponding to the angular range $0.068° < 2\theta < 4.80°$ and scattering center sizes between ~30 and 3000 Å. The measured scattered intensity was corrected by subtracting the background counts and taking into account the detector efficiency. The resolution function of the instrument was also considered in the fitting procedure of the experimental data. The intensity was calibrated in absolute units using a Plexiglas sample as secondary reference standard to obtain the scattering cross section value $\Delta\Sigma/\Delta\Omega$ from the scattering centers embedded in the matrix.

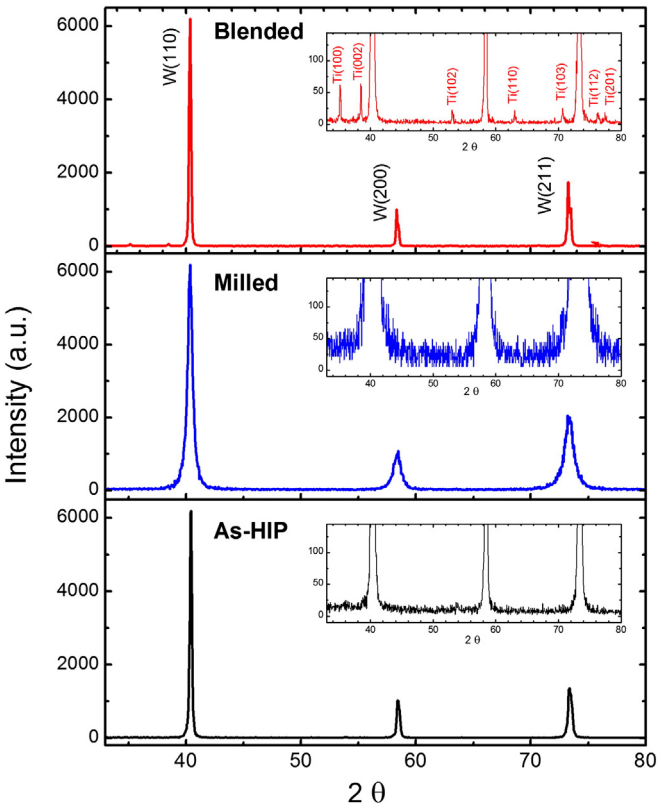

Fig. 2. X-ray diffraction patterns of W-4Ti blended powder, milled powder and consolidated sample. Inset: Enlargement of the diffraction pattern.

3. Results and discussion

3.1. Sample characterization

Figs. 2 and 3 show the evolution of the X-ray diffraction patterns for W-4Ti and W-4V, respectively, during the fabrication process of the materials. After milling for 20 h, as well as in the consolidated materials, the diffraction peaks corresponding to α-Ti, (or V) observed in the starting blended powders were undetected and only those from bcc W were apparent. The same occurred for the W-2Ti and W-2V alloys.

The O contents in the alloys during the steps of the fabrication process, along with the densities, are presented in Table 1. The O content in the W-V milled powders is significantly higher than in the W-Ti ones, although it becomes similar after the consolidation process. This can be attributed to the higher O affinity of V. No evidence for oxides was detected in the X-ray diffraction patterns. The density measurements indicate that the consolidated alloys are virtually fully dense materials.

The microstructures of the W-2Ti and W-2V alloys are shown in Figs. 4 and 5, respectively. The matrix of the alloys exhibits a bimodal grain size distribution with modal values larger and smaller than 1 μm. A homogeneous distribution of irregular shaped pools of practically pure Ti or V appears in the consolidated alloys (dark areas in the images) as

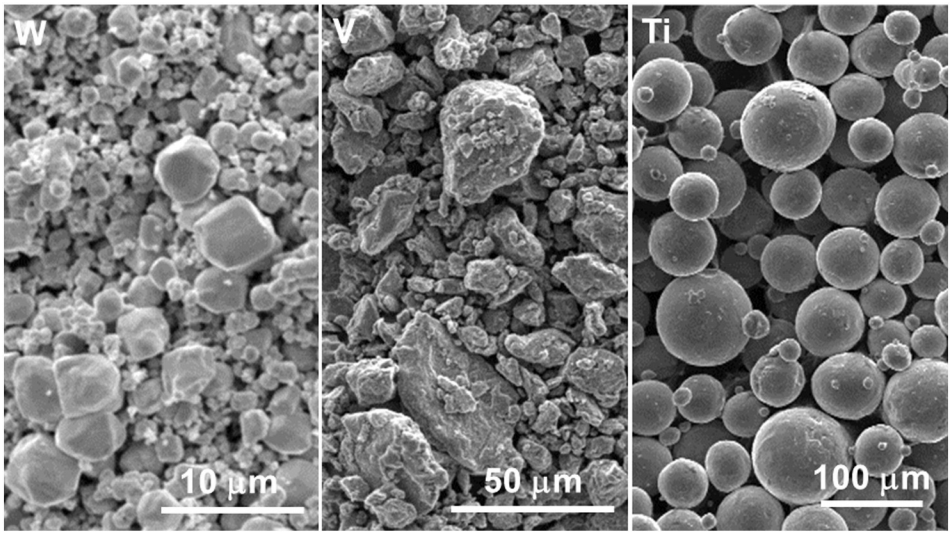

Fig. 1. SEM images of the starting elemental powders.