



Effect of reinforcement phase on the mechanical property of tungsten nanocomposite synthesized by spark plasma sintering



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ABSTRACT

Nanostructured tungsten composites were fabricated by spark plasma sintering of nanostructured composite powders. The composite powders, which were synthesized by mechanical milling of tungsten and Ni-based alloy powders, are comprised of alternating layers of tungsten and metallic glass several hundred nanometers in size. The mechanical behavior of the nanostructured W composite is similar to pure tungsten, however, in contrast to monolithic pure tungsten, some macroscopic compressive plasticity accompanies the enhanced maximum strength up to 2.4 GPa by introducing reinforcement. We have found that the mechanical properties of the composites strongly depend on the uniformity of the nano-grained tungsten matrix and reinforcement phase distribution.

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1. Introduction

Tungsten (W) alloy or pure tungsten metal contacts, electrodes, and wires are used in electrical, electronic, heating, lighting, and welding applications due to their exceptional properties such as high density, hardness, very high melting temperature, relatively high radiation opacity and good thermal conductivity combined with very low thermal expansion [1]. Tungsten is also used to make heavy metal alloys for armaments, heat sinks, radiation shielding, weights and counterweights; superalloys for turbine blades; tool steels; and wear-resistant alloy parts and coatings [1]. The leading use is as tungsten carbide in cemented carbides, which are sintered powder metallurgical parts that are used as cutting tools and wear-resistant components by the construction, metalworking, mining, and oil drilling industries [2].

Tungsten tool materials as carbides are the largest consumption volume of tungsten in end-use sector, which account for about 60% of the tungsten consumption [2]. The higher the volume fraction of carbide particles there are within the material, the harder it is, but the less tough it behaves during loading; and, vice versa, significant increases in toughness can be achieved by a higher amount of metallic binder (i.e., Co, Ni) at the expense of hardness. The cobalt and/or nickel metal powder is

used as a binder to hold together the tungsten carbide particles. The use of coarse W or WC particles/grains, which are preferred for the manufacturing process, promotes the increasing product efficiency but decreasing mechanical property (especially toughness) of the tool materials by coarse W particles [3–5]. Due to limitations in decreasing the particle size as well as processing complications, however, most WC tool materials have not achieved uniform nanometer-scale homogeneity, which is essential to increase toughness.

One noteworthy exception to this are uniformly-layered nanometer-scale tungsten composites, which are obtained by a controlled milling process to yield composite powders that are consolidated by spark plasma sintering (SPS) into fully dense bulk shapes [6,7]. The composites show more preferred shear banding in a nanometer-scale reinforcing metallic glass (MG) phase homogeneously-distributed in a metallic tungsten matrix. Spark plasma sintering (SPS), where the material is bonded by viscous melt flow at the particle surface, can be a suitable compaction technique to maintain the nanostructured metallic glass-tungsten composite that is created by high-energy ball milling since it utilizes low sintering temperatures and short sintering times [8,9].

Typically the processing temperature and time for synthesizing MG composite are determined by the thermal characteristics of the MG, i.e., supercooled liquid region, because crystallization of the MG critically deteriorates the mechanical properties [10]. However, improvement of plasticity in Zr-based MG after dispersion of crystalline nanoparticles by annealing has been reported [11]. There are no reports

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in the literature, however, on the relation between mechanical property and crystallization of amorphous phase in MG reinforced tungsten nano-composite before and after consolidation.

In the current study, we investigated the effect of crystallization of the reinforcing metallic glass phase on the mechanical behavior of tungsten nano-composites, which have uniformly dispersed reinforcements in the ultra-fine grained tungsten matrix exhibiting a novel nano-mixture structure by utilizing SPS.

2. Experiment

A master alloy was prepared by induction melting high-purity elemental constituents (electron beam melted Ni 99.999%, crystal bar Zr 99.99%, Ti sponge 99.99%, Si 99.9%, and Sn 99.9%) in an yttria-stabilized zirconia crucible under an Ar gas atmosphere. The homogenized molten alloy was bottom pour cast into a 5-cm diameter water-cooled Cu mold. The master alloy was re-melted in the atomization furnace. Spherical $\text{Ni}_{59}\text{Zr}_{20}\text{Ti}_{16}\text{Si}_2\text{Sn}_3$ (at.%) metallic glass alloy powders, which prepared by high pressure gas atomization at a dynamic pressure of 2.8 MPa and at a molten temperature of 1350 °C using a close-coupled annulus with a melt delivery nozzle 3.2 mm in diameter at the Ames Laboratory (USDOE), and W powders (>99.9% purity, PerkinElmer/Metco, SPI12430) both with have $\sim 45 \mu\text{m}$ sizes were used as starting materials. Because from the previous study, the atomized Ni-based MG powders were sieved according by conventional sieve analysis and were divided into the following 12 different size ranges: under 10, 10–20, 20–38, 38–45, 45–53, 53–63, 63–75, 75–90, 90–106, 106–125, 125–150 and over 150 μm . The oxygen content of the powder analyzed by an elemental inorganic analysis method decreases as the increase powder size up to 40 μm . The oxygen content is nearly constant (about 450 ppm) for powder sizes larger than 40 μm . For this study, the W contents were selected 50 vol.% (= 65 wt.%) to reveal possible effect of reinforcement to the deformation behavior as a fraction of microstructural change and 50 vol.% of Ni-based MG in the composites. Milling of the Ni-based MG powder blended with W powder was performed with a Retsch Pulverisette 5 planetary ball mill using a rotational velocity of 100 rpm for 144 ks using hardened steel milling tools and a ball to powder ratio of 10:1. Milling conditions with relatively low velocity were chosen to prevent crystallization of metallic glass or excess phase transformation during milling process. All milling and powder handling steps were done in a glove box in purified Ar atmosphere (less than 3 ppm O_2 and H_2O). The consolidation of the milled powders using SPS to form bulk samples with 20 mm diameter and 10 mm thickness was performed with a SPS Syntex Inc. Model Dr. Sinter SPS-515S with tungsten carbide punches and die. SPS was done under vacuum with an external compaction pressure of 300 MPa and a heating rate of 40 K/min. The temperature for sintering and processing time window were determined based on the T–T curve for the onset of crystallization [6]. Considering the heating rate, an SPS temperature of 577 °C was selected, and crystallization of Ni-based metallic glass can be retarded up to about 300 s at this temperature. The sintering temperature and time were varied to optimize the sintering parameters leading to dense (93.3%) sintered parts. The samples were cut and the surface was micro-polished to obtain 2 mm \times 2 mm \times 4 mm rectangular bars for compression testing. Structural characterization was performed using a Philips APD 3520 X-ray diffractometer with monochromatic $\text{Cu-K}\alpha$ radiation and a LEO 1530 scanning electron microscope (SEM). The thermal properties of the samples were measured with a PerkinElmer Pyris-1 differential scanning calorimeter (DSC) using continuous heating at a rate of 40 K/min. Uniaxial compression tests were conducted on an Instron Model 8563 at room temperature at a constant cross head speed and an initial strain rate of $3 \times 10^{-3} \text{ s}^{-1}$. The density of the samples was determined by the Archimedes method in water using a Mettler Toledo-AT261 Delta-Range density measurement system.

3. Results and discussion

The SEM images in Fig. 1 of the transverse cross-section of the sintered samples show two types of nanostructured W composites fabricated by SPS. Both structures show a uniform size and distribution of the Ni-based reinforcement particles (dark contrast area) dispersed within the W matrices (bright contrast area). The dark phase, however, in sample A, corresponds to a Ni-based crystalline phase and in sample B, it corresponds to a Ni-based amorphous phase. Fig. 1a displays a polished transverse section (i.e., the consolidation direction is normal to the plane of the image) of the spark plasma sintered 144 ks milled 50 vol.% Ni-based crystalline (devitrified MG during SPS) reinforced W composite (sample A). Fig. 1b shows the fine layered structure developed in the composite powders during milling is composed of homogeneously dispersed dark layers of the Ni-based MG separated by adjacent lighter W phases.

The observed size range of the layers is relatively broad and varies from ~ 20 to 500 nm, which is similar to the previously reported result in a Cu-based MG and Hf-based MG nanocomposites [7,12]. Due to longer milling time (144 ks), the thicknesses of the layers for the finely mixed structure between W and MG are smaller than previously reported for a 72 ks milled 50 vol.% W composite which has a layer size range from 100 nm to 2 μm thickness [6]. For the W-composite reinforced with the crystallized MG phase (sample A; Fig. 1a), the crystallized MG and the W matrix are intimately mixed in multiple layers similar to the sample B composite. The microstructures of the nano-grained tungsten matrix are mostly uniformly throughout for samples A and B, while the reinforcing phase is different for the two samples. This is not unexpected since both composite samples utilized the same starting

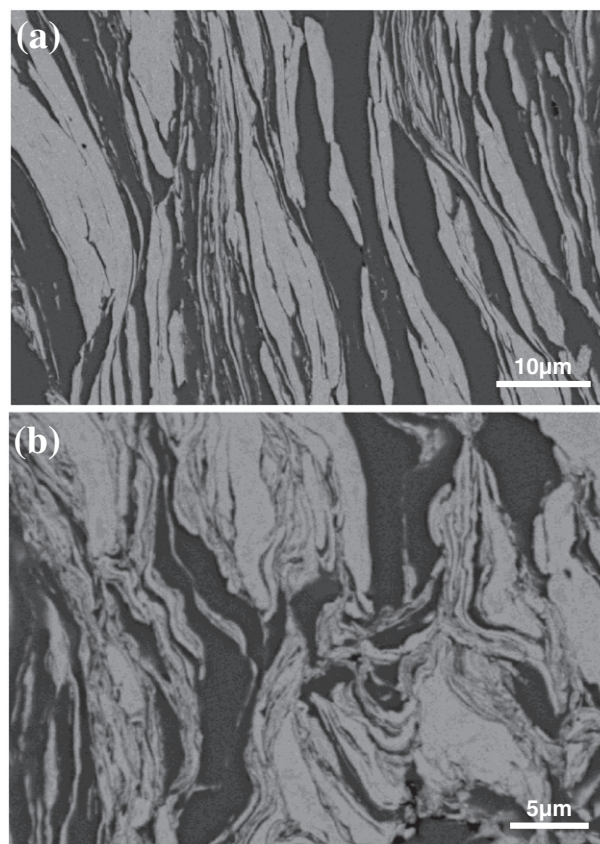


Fig. 1. SEM backscattered electron images obtained from the polished cross section of the spark plasma sintered nanostructured W composites containing 50 vol.% Ni-based alloy reinforcement. (a) Sample A: crystalline phase Ni-based alloy reinforcement particles within the W matrix. (b) Sample B: amorphous Ni-based alloy reinforcement within the W matrix.

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