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Non-destructive measurement of the tungsten content in the binder phase of tungsten heavy alloys

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ABSTRACT

Measurement of the magnetic properties is well-established to non-destructively monitor composition and microstructure of cemented carbides. The present study demonstrates that this method can also be applied to ferromagnetic tungsten heavy alloys. Compositional changes during heat treatment may have effects on the magnetic properties of W-Ni-Fe heavy alloys, as an example, which would allow for simple non-destructive measurement. We report that increasing tungsten content in Ni-Fe(-W) samples and higher annealing temperatures for *Densimet 180®* grade 95W-Ni-Fe heavy alloy samples cause a decrease of their respective weight-specific saturation magnetization. XRD analyses show that higher heat treatment temperatures result in larger lattice parameters of the binder phase. Likewise, larger amounts of tungsten dissolved in the nickel-iron samples also widen the γ-Ni-Fe lattice. This suggests that the decrease in saturation magnetization of heavy alloys annealed at higher temperatures is caused by more dissolved tungsten in the binder phase. As EDX measurements may overestimate the tungsten concentration in the binder phase, another method for determining the amount of dissolved tungsten was developed. A relationship between saturation magnetization and tungsten content in the binder could be established through the Ni-Fe(-W) samples with known amounts of tungsten. In view of these results, we propose saturation magnetization measurements as a simple non-destructive tool for additional quality control in the production of ferromagnetic tungsten heavy alloys.

1. Introduction

Liquid-phase sintered composite materials made of tungsten, nickel and other transition metals are commonly known as Tungsten Heavy Alloys (WHAs). Due to their unique combination of very high density, outstanding mechanical properties and excellent machinability, they are used in a wide range of high-performance applications, e.g. for balancing weights in aerospace and automotive industry, collimators and radiation shielding components, vibration-damping tool holders, kinetic energy penetrators, oscillating weights and sports equipment [1,2]. WHAs are composed of nearly pure body-centered cubic (bcc) tungsten grains embedded in a ductile face-centered cubic (fcc) binder phase (Fig. 1). This work will focus on W-Ni-Fe heavy alloy, one of the most commonly used WHA types, which contains γ-Ni-Fe as binder phase.

Although full density may be achieved through solid-state sintering, the presence of a liquid phase during sintering of WHAs is essential for obtaining their typical structure and properties [3]. Among the multitude of processes occurring during liquid-phase sintering, solution-reprecipitation of tungsten plays a major role. Upon melting of the binder powders, tungsten is dissolved in the newly formed liquid phase. Dissolution occurs preferentially at small grains and convex surfaces, whereas reprecipitation of tungsten is favored at large grains and concave surfaces [4]. In the long run, this effect causes growth and rounding of the tungsten grains. When the liquid phase solidifies during cooling, some tungsten remains dissolved in it and will therefore affect the properties of the binder.

Chemical composition measurements are often performed using energy-dispersive X-ray spectroscopy (EDX). While EDX is generally a simple and fast tool for qualitative analysis, the biphasic nature of tungsten heavy alloys obstructs precise quantification of the binder composition. Especially in high-tungsten grades containing only thin layers of binder phase between tungsten grains, the three-dimensional excitation volume from which the X-rays are generated does not necessarily reflect the composition of the surface visible in the two-dimensional SEM image. For instance, tungsten grains may be present closely underneath a small layer of binder and could affect the results of EDX analysis despite not being observable in the SEM image. This commonly leads to an overestimation

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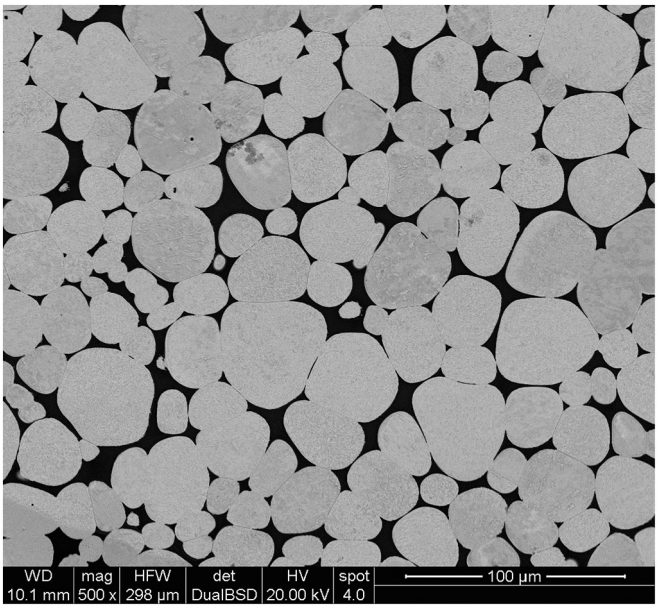

Fig. 1. Typical microstructure of a 95W-Ni-Fe heavy alloy (BSE image, tungsten particles are bright, binder phase is dark).

of the tungsten content. In fact, tungsten contents calculated from EDX analyses may considerably exceed the solubility limits of tungsten in γ-Ni-Fe, with values over 30% tungsten content in the binder phase having been reported in literature [5,6].

W-Ni-Fe is weakly ferromagnetic [7] and therefore, considering its structure, in some way comparable to cemented carbides, typically composed of hard tungsten carbide grains in a ductile cobalt matrix. For cemented carbides, coercivity and saturation magnetization measurements are well-established non-destructive methods of monitoring chemical composition and microstructure [8]. A correlation between tungsten grain size and coercivity (as known for tungsten carbide in cemented carbides) was reported for W-Ni-Fe heavy alloys by Danninger et al. [9]. However, very little information can be found in the literature about their magnetization behavior [7,10] and, to the best of the authors' knowledge, no connection between saturation magnetization and applied heat treatment has been described for W-Ni-Fe. In this work, we intend to fill this gap by investigating the effects of heat treatment on the saturation magnetization of W-Ni-Fe and exploring whether saturation magnetization measurements may therefore be used for determining the tungsten content in the binder phase of ferromagnetic WHAs.

2. Experimental

Small *Densimet 180®* grade 95W-Ni-Fe heavy alloy samples (approximately $10 \times 10 \times 5\,mm^3$ in size) were produced by *Plansee Composite Materials* via cold-isostatic pressing of tungsten, nickel and iron powder mixtures at 210 MPa pressure. Two types of commercially available tungsten powders from different manufacturers (labeled powder A and powder B) were used, both having a nominal grain size of 4 µm and a log-normal particle size distribution. SEM images of the metal powders used are shown in Fig. 2. Liquid-phase sintering of the powder compacts was performed in hydrogen atmosphere at an

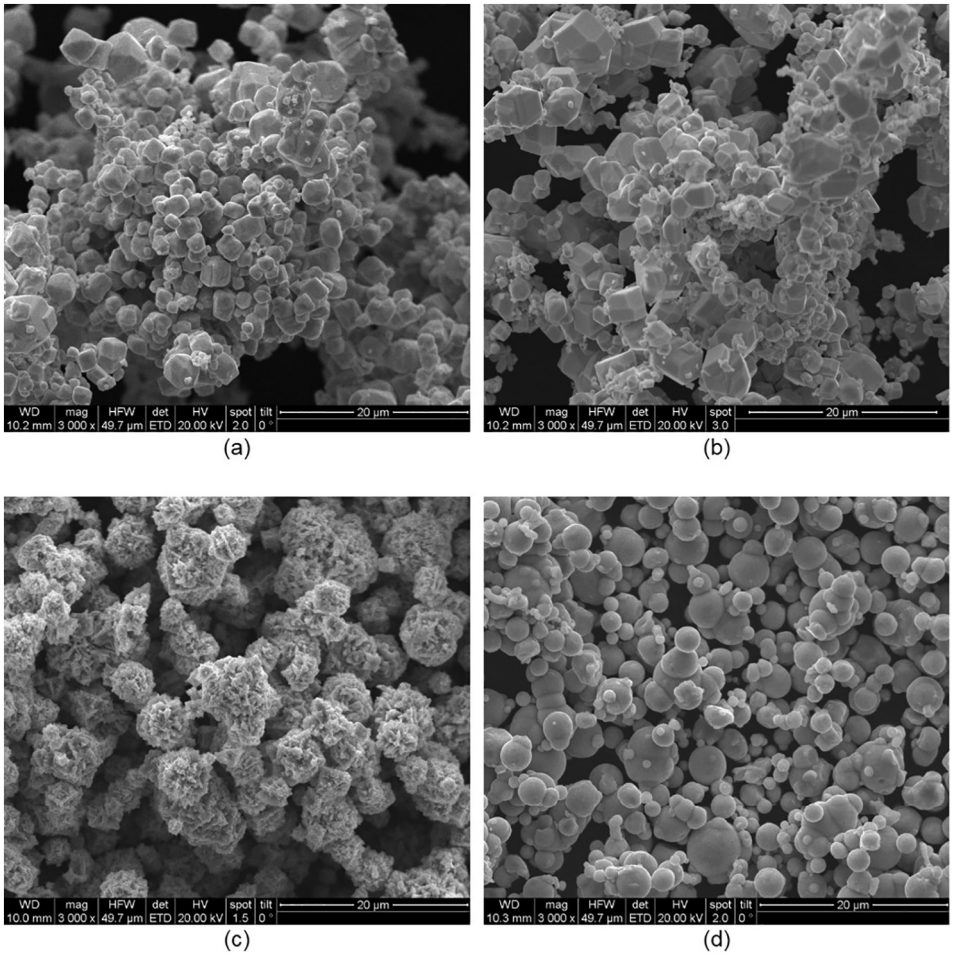

Fig. 2. SEM images (SE) of metal powders used: (a) tungsten powder A, (b) tungsten powder B, (c) nickel, (d) iron.