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A comprehensive microstructural analysis of Al–WC micro- and nano-composites prepared by spark plasma sintering

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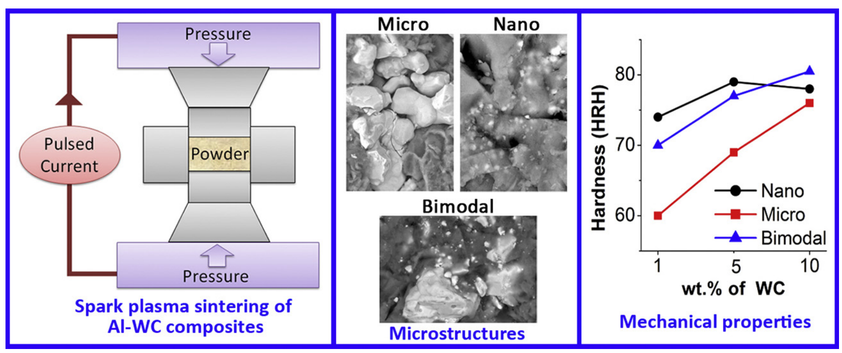
HIGHLIGHTS

- Aluminum matrix composites containing 1–10 wt.% WC micro-, nano- and bimodal-particles are consolidated by spark plasma sintering (SPS).
- The highest and lowest densification levels are obtained in 1 wt.% microcomposites (99.8%) and 10 wt.% nanocomposites (94.4%), respectively.
- Hardness increases by SPS temperature (400 → 500 °C) in 5 and 10 wt.% microcomposites, but slightly decreases in 1 wt.% microcomposites.
- Nanocomposites achieve largest hardness at 5 wt.% WC concentration, but micro- and bimodal-composites are hardest at 10 wt.% WC concentration.
- Interplay between WC particles distribution homogeneity and composite compressibility during SPS dictate the hardening level achievable in the composites.

GRAPHICAL ABSTRACT



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ABSTRACT

There have been many investigations on metal matrix microcomposites produced by conventional casting routes; however, in the past decade, the focus has shifted more toward nanocomposites produced via solid state routes. To have a realistic view of performance prediction and optimum design of such composites, in this work Al matrix composites (AMCs) reinforced with WC microparticles, nanoparticles, and bimodal micro-/nano-particles were prepared by spark plasma sintering. The effects of particle size and concentration, and process variables (i.e. sintering temperature, duration, and pressure) on the evolution of microstructure, density and hardness of the composites were studied comprehensively. Full densification of AMCs with high particle concentration was problematic because of ceramic cluster formations in the microstructure. This effect was more emphasized in AMCs containing nanoparticles. AMCs with microparticles were more easily densified, but their hardness benefits were inferior. On the other hand, the mixture of micro- and nano-particles in Al-WC bimodal composites led to better matrix reinforcement integrity and an overall improvement in the microstructural properties. Finally, increasing the sintering temperature improved the microstructural features

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and hardness of the composites (more enhanced in high wt.% samples), but sintering duration and pressure did not have a big impact on the composite properties.

1. Introduction

One of the most common means to evaluate an industry's vitality is to look at its product demand. For composite materials, the picture is promising as demand for end products reached $22.2 billion in 2015, with the top market segments in transportation/automotive, construction, aerospace, and pipe and tank applications [1]. The common aim in designing and using composite materials is to go beyond property boundaries attributed to common classes of materials. In this regard, a big interest has been given to the development of metal matrix composites (MMCs) in which rigid ceramic reinforcements embedded in a ductile metal or alloy matrix can result in high strength and toughness as well as excellent resilience and hardness. Aluminum matrix composites (AMCs) are a good example in this case as they benefit from excellent ductility, corrosion resistance, recyclability, and formability of aluminum as well as high stiffness, strength, hardness, and wear resistance of the ceramic component [2].

Among the ceramic materials implemented to reinforce the AMCs so far, carbides (SiC, TiC, WC, TiC) [3–14], borides (TiB_2, AlB_2) [15–18], nitrides (Si_3N_4, AlN) [19–24], and oxides (Al_2O_3, SiO_2) [25–31] have been more commonly investigated. However, there are several challenges associated with the introduction of these particles into the metallic matrix. For example, liquid-state processing techniques (such as stir casting, squeeze casting, infiltration, and die casting) [32,33] suffer from poor wettability of the ceramic reinforcements by the molten metal, as the melt has a very high surface tension (of the order of ~1000 mJ m^{-2}) [34]. Also the specific weight of the ceramics and molten metals are often considerably different. As a result, non-uniform distribution, agglomeration, and weak interfacial bonding of the particles in the metallic matrix are often unavoidable [35–39]. In addition, in the case of carbide and oxide particles in molten aluminum unwanted and brittle phases of Al_4C_3, $Al(OH)_3$, and Si may form due to processing at high temperatures [40,41].

An effective solution to overcoming the aforementioned problems in MMC manufacturing is the application of solid-state processing techniques. For instance, the use of mechanical milling followed by consolidation techniques, such as hot pressing, extrusion, forging, rolling, and cold isostatic pressing, can lead to more uniform distribution of the hard ceramic phase into the relatively soft metal matrix [39,42–48]. However, these consolidation techniques may have some adverse effects on the composite properties as well. For example, the applied high temperatures and long sintering time can lead to unfavorable interfacial reactions, oxidation, and grain growth that can deteriorate the mechanical properties [49–51]. Recently, spark plasma sintering (SPS), a non-conventional sintering process, has proved itself as an excellent technique for the consolidation of micro- and nano-composite powders. In this method, uniaxial pressure and pulsed DC current are applied to the powder simultaneously, as schematically shown in Fig. 1(a). The uniaxial pressure can greatly reduce (or even completely eliminate) porosity and also break up the otherwise intrusive oxide layer on aluminum particles [52,53], while the applied current heats the powder rapidly via Joule effect and promotes enhanced diffusion rate of the elements [54]. Moreover, short processing time in SPS can minimize microstructural coarsening, and effective bonding between particles can enable the synthesis of highly dense materials [55–57].

Given these beneficial traits, this research aims to conduct a comprehensive study on the SPS consolidation of AMCs reinforced with tungsten carbide (WC) particles, and to analyze the effect of process variables (SPS temperature, pressure, and time) as well as ceramic particle size (nano, micro, and mixed) and volume fraction on the microstructure and hardness of the composites. In this work, WC particles were the material of interest for reinforcement due to their remarkable hardness and stiffness as well as their good wettability by aluminum and capability to undergo small amount of plasticity [3]. Furthermore, there has rarely been a systematic study on the interrelationship between particle size/quantity and microstructural characteristics and mechanical properties of Al–WC composites.

2. Materials and methods

The powders employed in this work were aluminum (Wako Pure Chemical Industries, Ltd., Japan, average size ~44 μm) and tungsten carbide (Global Tungsten & Powders Corp., USA, average size ~200 nm and ~20 μm). Micro- and nano-composites consisting of 1, 5, and 10 wt.% of WC were fabricated by blending and mixing the matrix and reinforcement powders in an agate mortar for 30 min to get a homogeneous mixing followed by spark plasma sintering (Dr. Sinter 1080,

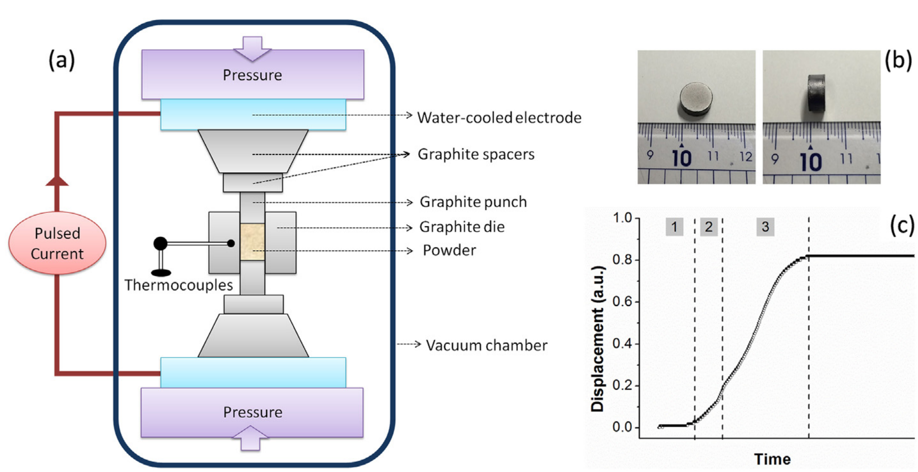

Fig. 1. (a) A schematic representation of spark plasma sintering (SPS) processing of composite powders. (b) Image of a typical sintered disc. (c) Punch displacement versus sintering time of an Al–WC microcomposite.