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Rapid processing & characterization of micro-scale functionally graded porous materials

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ABSTRACT

Spark plasma sintering (SPS) is a process that has stimulated worldwide interest for the rapid consolidation of powder-based materials through the combined effects of electric current and pressure. Recently the localization of SPS has been realized through current activated tip-based sintering (CATS) where electric current is selectively applied to small targeted regions of a green compact/powder bed via a precision controlled electrically conductive small tip. The unique tip-specimen geometry allows for locally controlled temperature and current distributions that can result in microstructural modifications on the micro-scale. The present paper presents for the first time the rapid processing and characterization of micro-scale functionally graded materials in relation to porosity content and size. The effects of initial green density and particle size on the developed micro-scale functionally graded material are discussed.

1. Introduction

Functionally graded materials (FGMs) have been around for many decades, and have the special feature of a spatial change in composition (Yongming et al., 2003), grain size (Traini et al., 2008), or porosity (Holland et al., 2010). The powder metallurgy approach can be well suited for the production of these types of materials. One of the basic approaches is to sequentially deposit or add different powder layers of varying compositions (Omori et al., 1998), particle size and/or pore content (Morsi et al., 2004) followed by full or partial consolidation at high temperatures. Despite its advantage in terms of simplicity, it involves multiple powder deposition steps and the approach is prone to processing defects such as layer delamination/interface cracking (Morsi et al., 2004) upon sintering which may result from differential shrinkage.

One of the recent approaches for the consolidation of FGMs is spark plasma sintering (SPS). The process typically involves the application of pulsed DC current and pressure to powder that is placed within a confined graphite die. SPS boasts much faster sintering and heating rates compared to conventional sintering (Omori, 2000). However, it has so far been largely limited to the production of simple bulk geometries on the macro-scale. Within the context of porous FGMs, SPS has recently been used to produce functionally graded porous tungsten macro-scale structures (which were later infiltrated with copper following SPS) using a prior powder segregation technique of bimodal powders (Janković Ilić et al., 2007). Titanium-sodium chloride (NaCl) composites were also spark plasma sintered after which the NaCl was dissolved out to produce functionally graded porous titanium microstructure ready for subsequent infiltration with biodegradable poly-L-lactic acid fibers (Watanabe et al., 2011). Using reactive mixtures of amorphous boron and carbon powders, macro-scale porous FGM B_4C was produced using an offset powder loading configuration under SPS (Hulbert et al., 2008) which was later infiltrated with aluminum. In another study 15 mm long and 9 mm thick B_4C tubes were produced using an SPS configuration that generates a steep temperature gradient enabling a steep radial microstructural variation across the outer thickness of the cylinder, to produce macro-scale cylinders with functionally graded porous surfaces (Holland et al., 2010).

On the other hand, the localization of SPS has been recently realized through current activated tip-based sintering (CATS). CATS is a new process where electric current is selectively applied to targeted regions of a green compact or powder bed using a macro-, micro- or nano-precision controlled electrically conductive tip. This unique tip-specimen configuration allows for the generation of locally controlled micro-scale (and potentially nano-scale)

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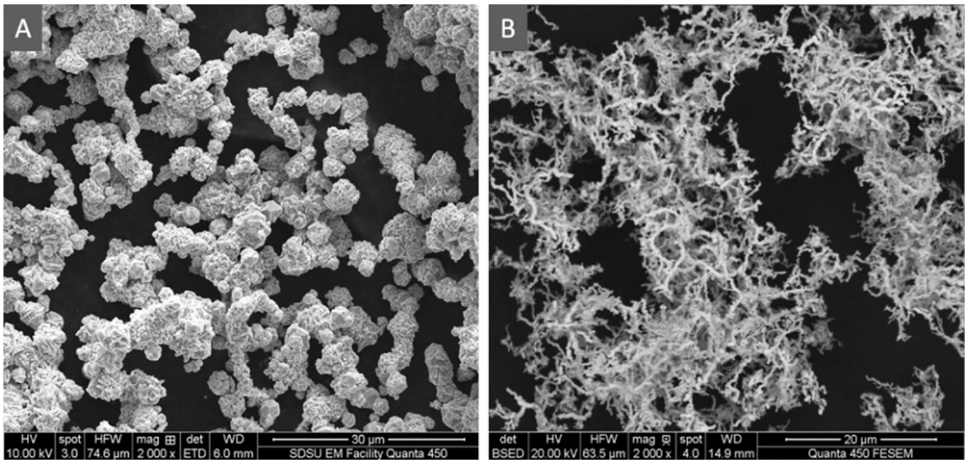

Fig. 1. FESEM micrograph of (a) INCO 123 and (b) INCO 210 nickel powders.

temperature and current distributions that can result in significantly large micro/nano-structural gradients. This ability to selectively apply spatial temperature and current distributions on such a small scale can potentially result in the production of porous "micro-scale" functionally graded materials (μ-FGM) under short electric current exposure times and without the need for sequential powder deposition approaches (which could be more complicated as the size scale is reduced). The CATS approach could open the door for the production of μ-FGM porous components, or larger 2D and more complex lightweight structures if a moving tip-approach is used, where complicated shapes can be sintered. However before such goals can be realized, it is important to confirm that such unique conditions can indeed result in micro-scale variations in pore content and size, and determine if they can be controlled for example by simply changing the initial green density or particle size. Although CATS has so far been largely used with the objective of locally sintering materials to high densities (Morsi et al., 2009), to-date no study has focused on its ability to produce porous μ-FGM features, which could additionally be later infiltrated. Moreover, to the best of the authors' knowledge, no prior work has investigated the porosity profiles and pore size distributions for electrically processed powder-based materials in general. As such, this paper investigates for the first time, the influence of green density and particle size on the micro-scale porosity profile and pore size distribution for porous μ-FGM produced using CATS.

2. Experimental procedure

Nickel powders (Inco Type 123 and Inco 210) with manufacturer quoted particle sizes of 3–7 μm and 0.5–1.0 μm respectively were used in the investigations (Fig. 1a and b are scanning electron micrographs of the powders used). Powders were compacted to different fractional green densities between ~0.57 and ~0.78 of theoretical. Fractional densities were calculated by measuring the specimen mass and dimensions to give the density, which was then divided by the theoretical density of nickel (8.9 g/cm^3). The fractional green densities for the investigated configurations were also confirmed by image analysis. Table 1 shows the investigated green density-particle size combinations.

All green compacts had the dimensions ~12.1 mm diameter and ~1 mm thickness (i.e. squat specimens) in order to minimize density distribution within the compact. Compacts of less than 0.57 fractional density lacked sufficient green strength for handling and processing and were therefore excluded from the investigations. For all CATS experiments a continuous ultra-high nominal current density (current intensity/tip-specimen contact area) of ~101 kA/cm^2 was applied (using a power supply, Power Ten, Model P63C-51000) to the top central region of each green compact using a conductive flat tip of diameter 500 μm for 30 seconds. A tip load of ~4 N was initially applied and maintained throughout the process, and all experiments were conducted in an inert argon environment.

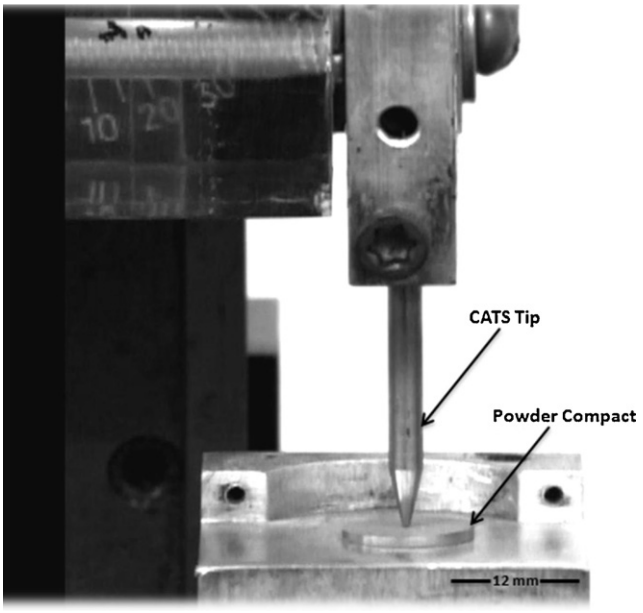

Fig. 2. Tip-specimen setup.

Fig. 2 shows the tip-specimen setup. Following CATS, the surface was slightly ground to remove any tungsten picked up by the surface due to the ultra-high and continuous current densities used. For micro-structural characterization, micro-scale sintered samples were cross-sectioned centrally along the thickness of the compacts and were ground and polished to at least 1-μm finish using diamond suspension. Micro-structural characterization was

Table 1
Investigated fractional green density-particle size combinations (for investigated configurations A–D) in the present study.

Particle Size (μm)	Fractional green density		
	0.57	0.67	0.78
3–7	A	B	C
0.5–1.0		D	