Materials Letters 211 (2018) 153–156

Contents lists available at ScienceDirect

Materials Letters

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

Powder roll-compaction process for controlling grain orientation texture and size in spark plasma sintered carbides

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A R T I C L E I N F O

Article history:
Received 15 June 2017
Received in revised form 25 September 2017
Accepted 1 October 2017
Available online 3 October 2017

Keywords:
Carbide
Roll-compaction
Gradients
Powder technology
Sintering
Texture

A B S T R A C T

The objective of this work is to develop and evaluate a powder roll-compaction procedure for fabricating cemented carbides that enables control over its surface/sub-surface grain orientation texture and grain size. The primary motivation for this work is to gain the ability to impart spatial material (property) gradients in bulk hard materials and composites for the realization of bio-inspired designs of material-systems. For this study, measured mixtures of tungsten carbide (WC) and cobalt (Co) powders were subjected to pressurized roll-compaction operations to create (malleable) thin sheets, which were then stacked and spark plasma sintered to obtain bulk cemented WC-Co specimens. When using this procedure, it was observed through XRD results that the basal plane orientation texture was increased by about 21%, and that transitions in layer-by-layer carbide grain sizes could be reliably achieved.

1. Introduction

Many biological organism features exhibit unique combinations of functionalities which are achieved via spatial and temporal compositional and structural gradients [1]. When considering functionally-graded natural material-systems, the static gradients are usually attained through continuous transitions in the hardness and/or moduli. A major hurdle in translating such bio-inspired designs to synthetic material-systems is our inability to reliably and consistently impart such (property) gradients when fabricating them in bulk. This paper outlines a powder roll-compaction procedure that strives to impart such gradients through the layer-by-layer control of (*i*) dominance in carbide basal plane orientation texture, and (*ii*) grain sizes [2,3].WC grains primarily grow on the prismatic and basal surfaces during sintering [4]; these are commonly observed in the form of two types of prismatic {1010} and one type of basal {0001} facets [5]. When considering grain orientation texture, the significant difference in hardness between the basal and prismatic planes of hexagonal WC crystals could be leveraged to impart property gradients (measured basal plane hardness is ~1.6 times that on prismatic planes [6,7]). Kim et al. [8] has shown (through simulations) that the young's modulus is affected by WC grain texture, and that its fracture resistance could be improved by 2–13% by texturing the sample with $(100)^C//[10]^S$ or $(100)^C//[100]^S$. Thus, imparting certain layer-by-layer grain textures could offer control over the mechanical and tribological responses. Previous related efforts include obtaining a textured WC-Co surface by manually positioning large WC crystals with the aid of a stereomicroscope and forceps [9]. Efforts on texturing bulk composites (from powder) include an extrusion process for bulk ZnO ceramics [10], and a rolling-extended method utilizing templated grain growth for bismuth-layer-structured ferroelectrics, MgO ceramics and $Ca_{0.85}(LiCe)_{0.075}$-$Bi_4Ti_4O_{15}$ ceramics [11–13].

Carbide grain size affects the properties and performance of WC-Co composites by impacting the hardness (relatable to strength and wear resistance) and transverse rupture strength (measure of toughness). It is known that with decreasing grain size, the yield strength (and hardness) increases within limits as per the Hall-Petch relationship. Thus, it would be advantageous to be able to fabricate bulk functionally graded structures having grain size gradients having certain hardness-toughness combinations.

2. Materials and methods

In order to fabricate bulk cemented carbides with graded structures, sets of WC and Co powders (in 9:1 wt ratio) were first ball-milled to obtain consistent mixtures. These were mixed with a

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https://doi.org/10.1016/j.matlet.2017.10.002
0167-577X/

temporary binder, *viz.*, polyvinyl alcohol in a weight ratio of 4:1, so that the 'dough' was malleable into thin sheets. After damp drying, these were placed between separating films and subjected to multiple pressurized rolling operations. From these sheets (having ~0.5 mm thickness), a number of discs were cut, stacked, thermally de-bound (to remove the temporary binder), and then spark plasma sintered to obtain bulk cemented WC-Co samples using a time-temperature-pressure recipe for minimum grain growth and high densification [14]. The general procedure is outlined in Fig. 1.

For imparting dominant grain orientation textures, measured mixtures of WC and Co powders along with the binder were processed via roll-compaction, and the resulting sheets cut, stacked, thermally de-bound, and sintered. Subjecting these sheets to pressurized rolling operations was deemed responsible for 'aligning' the carbide grains in order for them to grow to have a dominant orientation. For imparting grain size control, sets of WC powder mixtures having different grain sizes were processed in a similar fashion but separately, and the respective sheets stacked in the number/order desired, and then sintered.

3. Results and discussion

3.1. Imparting dominant carbide basal plane orientation texture

For imparting dominant surface and sub-surface textures in carbides, WC (average grain size ~4 μm) and Co (average grain size ~0.5 μm) powders were obtained from commercial cutting tool manufacturers. Both randomly oriented powder and roll-compacted sheets were included within the same specimen to minimize any sintering-related variability. After sintering, suitable sections were made. The SEM images of these sections are shown in Fig. 2. On comparison, it was observed there the average grain size of the roll-compacted region was generally larger than that of the randomly oriented powder region within the same specimen (although the original powder grain sizes were the same). When examining the elemental distribution (via EDS) of these surfaces, a fairly uniform distribution of Co around the WC grains was observed as well.

Global textures of these sintered surfaces were then measured using X-ray diffraction (XRD), and these patterns are shown in Fig. 3. The commonly observed peaks in WC-Co composites, *i.e.*, those belonging to the carbide basal (0001) plane and two prismatic planes, (1100) and (1011) are visible. By defining the relative intensity of the highest XRD pattern peak as 100%, the relative intensities of the carbide basal planes (0001) for the randomly oriented powder (A) and roll-compacted discs (B) were found to be 46% and 67% respectively, *i.e.*, a 21% increase. This implies that the relative surface area of carbide basal planes is higher for the roll-compacted discs within the same sintered specimen, thus offering the potential to control both the dominant texture as well as the resulting mechanical properties (higher hardness of carbide basal planes).

3.2. Imparting grain size transitions (and gradients) in sintered carbides

For fabricating bulk WC-Co specimens with transitions in grain sizes, four sets of WC powder mixtures having different average grain sizes (0.4 μm, 7 μm, 4 μm, and 3.35 μm) were subjected to the above procedure. Each of these WC powder samples were mixed with Co (having an average grain size of 1.7 μm) in the ratio

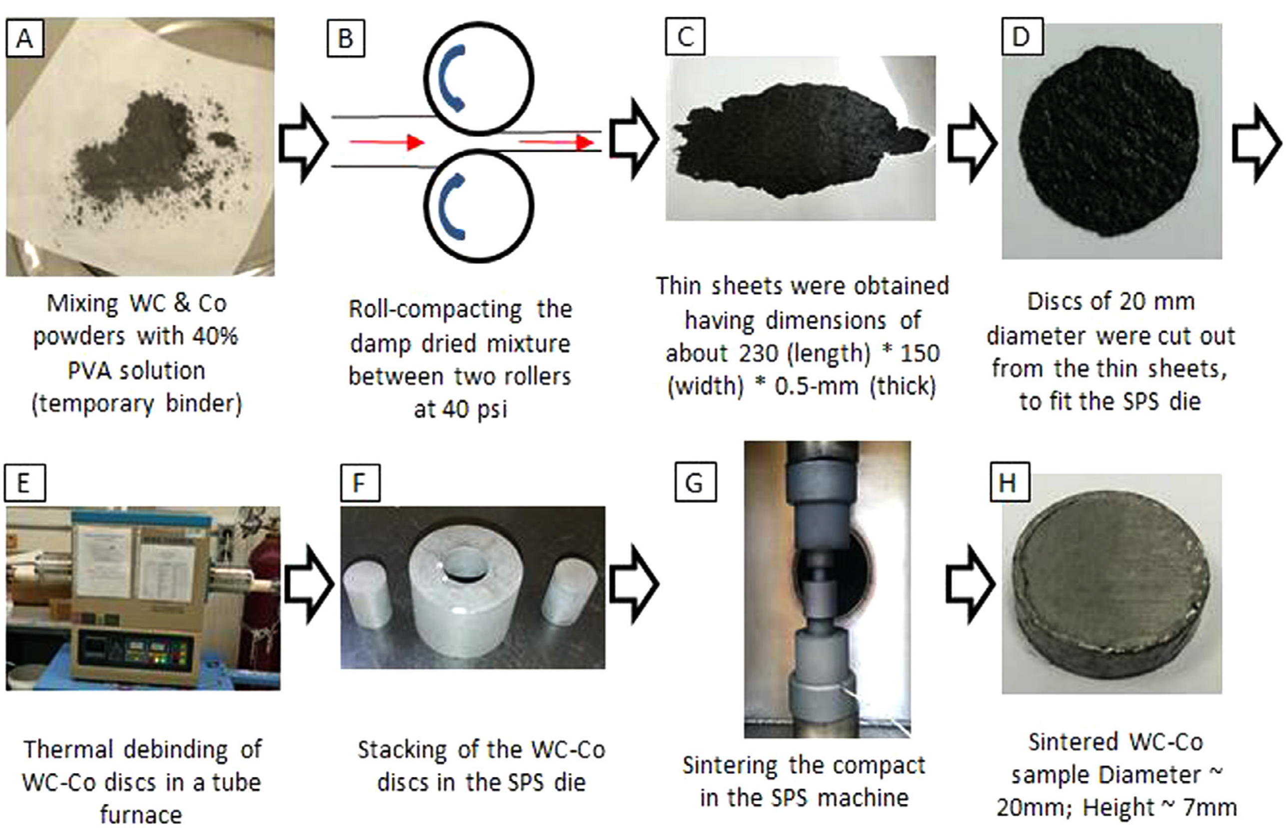

Fig. 1. Modified fabrication procedure for WC-Co composites that involves the roll-compaction of powder mixtures, stacking, and sintering to obtain functionally graded cemented carbides.