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Strength of functionally designed cellular cemented carbides produced by coextrusion

Sean E. Landwehr · Gregory E. Hilmas · Anthony Griffo

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Abstract In an effort to improve the wear characteristics of petroleum drill bit inserts, a series of cemented carbide materials with a functionally designed cellular (FDC) architecture were fabricated by a coextrusion process. The FDC architecture characterized in this study was comprised of cemented carbide cells surrounded by a ductile cobalt cell boundary. Property evaluation employed transverse rupture strength (TRS) testing to characterize their mechanical behavior. It was determined that the presence of $Co_{2+x}W_{4-x}C$ in the material greatly affected the bonding of the cell to the cell boundary and therefore the strength of the material. Fractography of the FDC materials supported the hypothesis that the interface between the cell and cell boundary was affected by the $Co_{2+x}W_{4-x}C$ phase and the consequential reduction in cobalt content of the cell.

Introduction

Cemented carbide materials are widely used in the petroleum drilling industry because of their high wear resistance and superior toughness in this physically demanding application. The tungsten carbide provides the material with wear resistance while the ductile binder imparts toughness. It is well known that increasing the cobalt content of the material results in an increase in toughness, however it comes with a concomitant decrease in wear resistance [1]. Thus in traditional cemented carbide materials, a balance must be struck between wear resistance and toughness. A new class of functionally designed materials, using engineered architectures, seeks to minimize the loss of wear resistance while gaining the desirable fracture characteristics associated with additional metallic material [2–4]. One class of these materials is known as ‘‘double cemented’’ carbides [2]. The double cemented carbide materials contained spherical WC-Co globules surrounded by a continuous cobalt phase. These materials have seen limited use in petroleum drilling applications and the materials wear characteristics, as well as its fracture behavior, have been studied [3]. The other engineered architecture is a functionally designed cellular (FDC) structure. These materials trace their roots back to a class of materials known by the ceramic materials community as ‘‘fibrous monolithic’’ materials [5–11]. Fibrous monolithic materials are meant to impart non-brittle fracture to otherwise brittle materials. A particular class of these materials consisted of a brittle ceramic surrounded by a metal cell boundary [9, 11]. This concept is the basis of the FDC materials currently being developed in cemented carbides. The brittle and more wear resistance material (WC-Co) is surrounded by the tougher but less wear resistant material (Co). These materials have been the subject of a study to characterize their wear resistance (Landwehr S, Hilmas G, Whie B, Griffo A, Bitler J, University of Missouri-Rolla, Rolla, MO, Unpublished

S. E. Landwehr (✉) · G. E. Hilmas
Materials Science and Engineering Department,
University of Missouri-Rolla, 1870 Miner Circle, 222
McNutt Hall, Rolla, MO 65409, USA
e-mail: landwehr@umr.edu

A. Griffo
Smith Bits, Houston, TX 77032, USA

research, 2004). This study examines the fracture behavior of these materials. Transverse rupture strength (TRS) results along with fractographic analysis were used to gain an understanding of the mechanical behavior of FDC cemented carbides.

Experimental procedure

The FDC materials in this study were produced using WC-Co powders produced by Kennametal Energy Group (Rogers, AR). Two powders were used in the study: powder A (WC APS 5 μm, 6 wt% Co), and powder B (WC APS 4 μm, 14 wt% Co). These powders were chosen to investigate the effect of changing cobalt content of the cell phase. The cobalt powder used in the cobalt cell boundary was a commercially available grade from CERAC, Inc. (C-1111, –325 mesh, Milwaukee, WI).

A novel coextrusion process was used to produce the FDC materials in this study. A more detailed account of coextrusion processing and its development has been presented previously for producing fibrous monolithic materials and prior FDC hard materials [10–12]. The FDC materials in this study, employed a cell material consisting of WC-Co, making up 82.5 vol% of the composite architecture, while the surrounding cobalt cell boundary constituted the remaining 17.5 vol%.

The filaments from the coextrusion process in the current study were aligned unidirectionally in a rectangular die and warm pressed into billets with respect to the cell orientation at a temperature of 150 °C, and a pressure of ~30 MPa. From these unidirectionally aligned billets, samples with specific orientation were produced for testing.

The binder was removed from the samples by thermolysis. The heating schedule was approximately 160 h long, and utilized an oxidizing atmosphere of compressed air at temperatures below 280 °C and a reducing atmosphere of Ar/5%-H_2 above 280 °C to prevent oxidation of the parts. Samples were heated to 1050 °C in the reducing atmosphere after thermolysis was complete to impart handling strength through partial sintering prior to rapid omnidirecional compaction (ROC).

The final step in the processing of these materials was ROC. Details of this process have been outlined elsewhere [13–15]. For the materials in this study the ROC process was performed at 1240 °C and a pressure of 840 MPa. These conditions allow for sintering in the solid state and prevent the breakdown of the FDC architecture due to cobalt migration from the cell boundary into the cell.

Since the materials spent a considerable amount of time in an oxidizing atmosphere, the materials were examined for the presence of a $Co_{2+x}W_{4-x}C$ phase after processing. Samples were etched for 3.5 min with Murakami's solution and examined with optical microscopy (Richter MFe3, Germany). The micrographs were then analyzed using Image J (National Institutes of Health, Washington, DC), which used areal analysis to determine the relative amount of cobalt depleted from the cell during the formation of the $Co_{2+x}W_{4-x}C$ phase.

TRS testing was conducted in three-point bending, in accordance with the standards of the carbide industry (ASTM B406-96). The TRS samples were 0.2 × 0.25 × 0.75 in (5.1 × 6.4 × 19.1 mm) and were ground to a surface finish (RMS) of 0.38 μm. The flexure fixture was mounted on a mechanical load frame, where the samples were tested until failure. Five samples were tested for each condition or cell orientation as described in the standard.

Since the FDC materials are anisotropic, they were tested in two different orientations with designations adopted from a previous wear study [Landwehr S, et al. Unpublished research, 2004]. These orientations can be found in Fig. 1a and b. Figure 1a shows the longitudinal orientation, in which the cells of the FDC architecture were perpendicular to the loading direction. The second orientation, the perpendicular orientation, is shown in Fig. 1b where the cells of the FDC architecture were oriented parallel to the loading direction.

In addition to testing the FDC materials, the constituent materials, both monolithic cemented carbide grades as well as the cobalt that makes up the cell boundaries of the FDC composites, were also tested. This additional testing allowed for a baseline comparison of the strength of the FDC materials. These materials were also produced using the ROC process under the same conditions.

Fractographic analysis of the FDC materials was carried out using a scanning electron microscope (SEM; JEOL, Model T330, Tohoku, Japan). Fracture specimens were obtained from sample testing in the TRS studies.

Results and discussion

Presence of $Co_{2+x}W_{4-x}C$

The presence of the $Co_{2+x}W_{4-x}C$ phase was suspected due to the extended hold at elevated temperatures in an oxidizing atmosphere. Optical microscopy (micrographs not pictured here) showed that a large

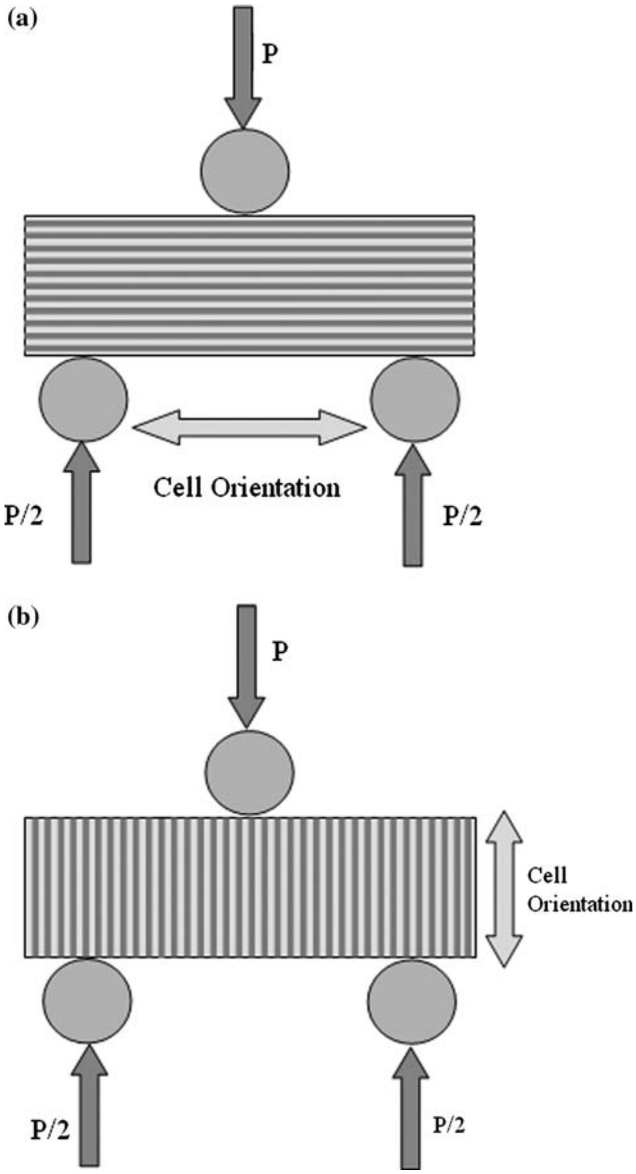

Fig. 1 Schematic representations of TRS test set-up for (**a**) the longitudinal orientation and (**b**) the perpendicular orientation for FDC materials

portion of the cobalt in the cell material was consumed; however the cobalt in the cell boundary appeared unaffected by the reaction. When image analysis was performed on the micrographs it was determined that 6 wt%, or all, of the cobalt in the cell of the FDC A materials was consumed, while the FDC B materials had 8 wt% of the cobalt consumed in the cell material, leaving 6 wt% cobalt in the cell. While the formation of $Co_{2+x}W_{4-x}C$ was unintended, it gives an interesting comparison of fracture behavior as it pertains to cobalt content in the cell material of the FDC cemented carbides.

TRS testing

The TRS testing results are shown in Fig. 2. Included in this chart are the results of the monolithic WC-Co materials, the FDC materials produced with each WC-Co grade as the cell material, and the cobalt material that makes up the cell boundary. The error bars in Fig. 2 represent the standard deviation of each set of tested samples. The standard deviation was taken to be the variability in the strength measurements.

When examining the TRS results it is straightforward to consider the strength of the FDC materials as a degradation in strength from the strength of the monolithic ROC materials for the longitudinal orientation. The longitudinal orientations were then ~50 and 45% weaker than the monolithic ROC materials for the FDC A and B materials respectively. Since these values are similar, it suggests that in this orientation, the strength is controlled by the FDC structure which degrades the strength by almost an equivalent amount from the monolithic ROC materials.

Testing in the perpendicular orientation gave a good approximation of how well the cell was bonded to the cell boundary. How well the cell was bonded to the cell boundary was determined by how close the strength of the perpendicular orientation was to the strength of pure cobalt. The drop in strength, as compared to the pure cobalt, was ~70 and 20% for the FDC A and B perpendicular materials, respectively. This suggests that the bonding between the cell and cell boundary was much better in the FDC B materials. The major difference in these two materials, as described in the previous section, is that the FDC A materials had no cobalt remaining in the cell, whereas the FDC B materials had ~6 wt% remaining. It can be inferred from this that the formation of the $Co_{2+x}W_{4-x}C$ phase, and the subsequent loss of cobalt, resulted in

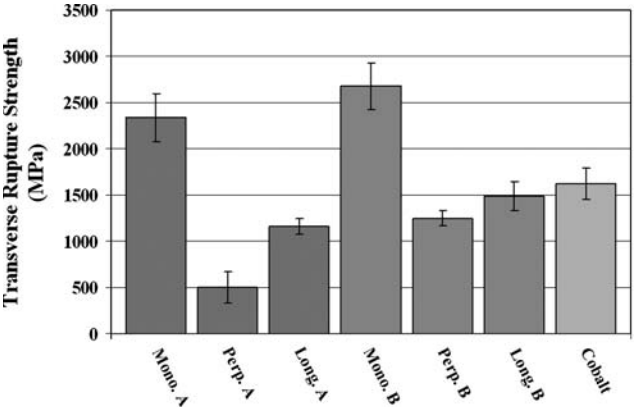

Fig. 2 Chart of TRS results for this study

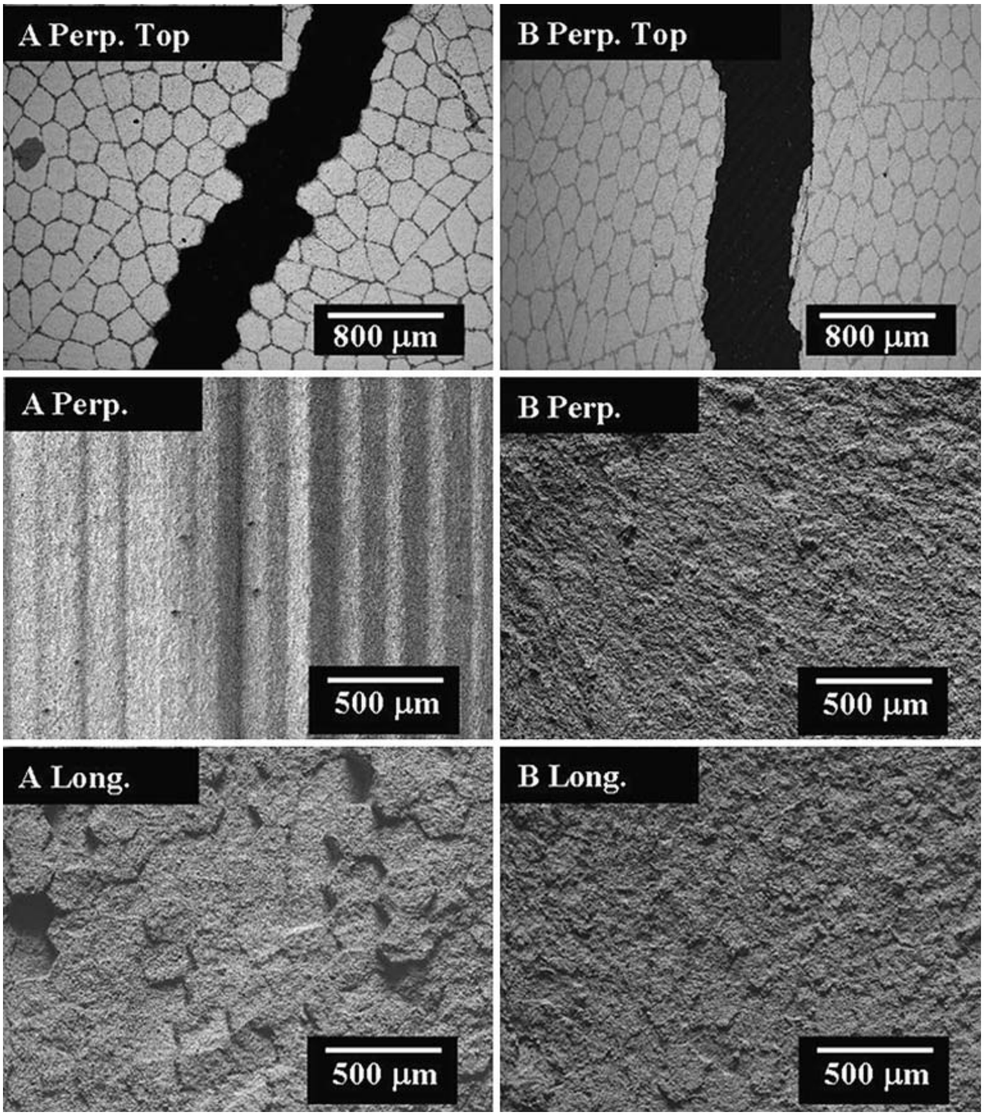

Fig. 3 SEM micrographs of the top view of fracture for the perpendicular orientation and the fracture surface views for both the perpendicular and longitudinal orientation in the FDC A and B materials

poor bonding between the cell and cell boundary of the FDC materials.

Fractography

The fracture behavior of the FDC TRS samples was examined using SEM. The manner in which the FDC materials fractured seems to confirm what was hypothesized previously in regard to the reduced relative strength of the bond between the cell and cell boundary. In Fig. 3, the FDC A materials show extensive fracture along the interface of the cell and cell boundary. In the case of the perpendicular orientation this is evident by the lack of fracture through the cells, and in the longitudinal orientation it is evident by the cells being in relief from the fracture surface. All of these features are noticeably absent from the FDC B materials, and the fracture of the material seems to be unaffected by the FDC architecture. This suggests that the bonding between the cell and cell boundary is weak for the FDC A materials due to the lack of cobalt remaining in the cell, while the bonding between the cell and cell boundary is much stronger due to the 6 wt% cobalt not consumed during the formation of $Co_{2+x}W_{4-x}C$.

Summary

1. WC-Co materials having a FDC architecture were produced in an attempt to improve the performance of cemented carbide materials for petroleum drilling applications. The fracture characteristics of these materials were investigated using optical microscopy, TRS testing, and fractography with SEM.

2. The FDC A and B materials lost 6 and 8 wt% cobalt from the cells in the FDC structure, respectively, due to the formation of a $Co_{2+x}W_{4-x}C$ phase. This left no cobalt in the cell for the FDC A materials and 6 wt% remaining in the FDC B materials.
3. The FDC A and B materials, in a longitudinal orientation, showed a 50 and 45% decrease, respectively, in strength from their constituent monolithic materials suggesting the FDC architecture controls the strength in this orientation.
4. The FDC A and B materials in the perpendicular orientation showed a decrease of 70 and 20%, respectively, as compare to the strength of pure cobalt. This result suggested that the bond between the cell and cell boundary in the FDC materials was greatly influenced by the presence of cobalt in the cell.
5. Fractography confirmed that the FDC A materials had weaker bonding between the cell and cell boundary than the FDC B materials, due to the absence of cobalt in the cell.
6. Further research should be performed to eliminate the presence of $Co_{2+x}W_{4-x}C$ and determine the optimal amount of cobalt in cell material. Once the fracture behavior has been tailored to mitigate crack growth, wear, and fatigue testing of the optimized composition should be conducted to determine its suitability for petroleum drilling applications.

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