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Metallographic Preparation of Titanium Diboride Coatings

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Mark E. McAllaster

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METALLOGRAPHIC PREPARATION OF
TITANIUM DIBORIDE COATINGS

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ABSTRACT

A method is described for preparing metallographic cross sections of thin, hard, chemically vapor deposited titanium diboride coatings on various softer substrates. Standard metallographic preparation techniques were found to result in fracturing and edge rounding of the coatings. It is shown that these problems can be avoided by unidirectional grinding on worn 600 grit silicon carbide abrasive paper. Typical photomicrographs of chemically vapor deposited titanium diboride coatings are shown along with photomicrographs of intermediate phases that form at the titanium diboride - substrate interfaces.

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METALLOGRAPHIC PREPARATION OF TiB_2 COATINGS

Thin coatings of erosion resistant titanium diboride are currently being studied by Sandia National Laboratories for application in components ranging from valve parts in coal liquifaction systems to beam limiters in Tokamak fusion reactors. The TiB_2 is chemically vapor deposited as a hard (VHN 3300) 10 to 75 μm thick coating on a variety of softer substrates including steels, copper alloys, iron-nickel alloys and graphite. The major emphasis of the work at Sandia is to characterize the effects of chemical vapor deposition parameters on microstructure and erosion resistance of the coating.

Initial attempts to grind and polish cross sections of TiB_2 coatings were unsuccessful due to severe rounding and fracturing of the coatings. It is the intent of this paper to describe a procedure that was developed to prepare high quality samples of TiB_2 coatings for metallographic examination.

The initial attempts to prepare metallographic samples utilized standard preparation techniques. The samples were sectioned using a diamond wafering blade, mounted in epoxy, ground on 120 thru 600 grit silicon carbide abrasive papers, and polished on nylon cloths using 30 micron, 6 micron, and 1 micron diamond paste. Optical examination revealed that the substrates were adequately polished, however, the TiB_2 coatings were fractured with less than 5% of the coating being suitable for examination. Additional polishing to improve the condition of the coating resulted in edge rounding to the degree that simultaneous optical examination of the edges and center of the coating was not possible as illustrated in Figure 1. It was apparent that standard metallographic techniques were inadequate and that an alternate method was needed to obtain a flat highly polished coating.

As part of an attempt to understand the cause of the fracturing, an assumption was made that the brittle coating was being fractured, in part, due to insufficient support from the epoxy mount. This assumption was tested by grinding the samples in predetermined directions as illustrated in Figures 2 and 3. It was found that extensive fracturing occurred at the interface of the TiB_2 coating and the epoxy mounting material when the samples were held such that the abrasive particles passed from the substrate material across the TiB_2 coating and into the mounting material as illustrated in Figure 2. Significantly less fracturing occurred when the grinding direction was reversed, as illustrated in Figure 3, resulting in more support of the coating by the stiffer substrate materials which were in intimate contact with the coating. This confirmed the earlier assumption that the lack of support of the epoxy mount was contributing to the fracturing of the coating. It also revealed that the fracturing could be minimized by holding the sample in the proper orientation on the grinding wheel.

This technique allowed a flat surface to be obtained on the TiB_2 coating however, a small amount of fracturing still occurred as illustrated in Figure 4. It was found that all of this fracturing could be eliminated and a high quality, flat, unfractured surface of chemically vapor deposited TiB_2 could be obtained by holding the sample in a stationary position on the same 600 grit silicon carbide abrasive paper for five minutes, as illustrated in Figure 5. Any subsequent grinding on new 600 grit silicon carbide paper resulted in fracturing of the coating similar to that shown in Figure 4. Any subsequent polishing using diamond paste resulted in edge rounding.

A scanning electron microscope was used to examine the new and the worn silicon carbide abrasive papers, Figures 6 and 7 respectively, in an effort

to understand why the worn abrasive paper works well and the new abrasive paper fractures the coating. The new abrasive particles have sharp points and edges, whereas the worn particles are rounded and flattened. This suggests that the reason the new abrasive particles fracture the coating is that the sharp points and edges of the new particles result in highly stressed point loading conditions, causing localized fracturing of the hard brittle coating. As the abrasive particles become rounded and flattened this point loading condition is eliminated and the fracturing no longer occurs.

After a high quality polish was obtained, the samples were chemically etched in 2 mL HF, 2 mL HNO₃ and 96 mL H₂O. Electrolytic etching in the same solution for up to 100 seconds at 2-1/2 volts was used for the TiB₂ on graphite samples. Examination using interference contrast techniques revealed a columnar grain structure as shown in Figure 8. Banding, apparently as a result of fluctuations in gas flow during the chemical vapor deposition process could also be seen in the coating.

Examination of the interface between the TiB₂ coating and metallic substrates was performed in a few cases to determine if intermetallics or other borides were being formed during the deposition process. Polishing on nylon and microcloths using diamond paste and an oil extender was needed to remove the scratches from the substrate. This polishing was limited to an absolute minimum amount of time due to edge rounding that occurs with longer polishing times. The intermediate phases that formed between Kovar and TiB₂ are shown in Figure 9. These were identified as (Fe, Ni, Co)₃B and (Fe, Ni, Co)₂B by electron probe microanalysis.

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Figure 1. TiB₂ coating on graphite substrate showing severe edge rounding as a result of standard metallographic procedures.

Figure 2. Rotation direction of silicon carbide grinding wheels resulting in severe fracturing of TiB₂ coatings.

Figure 3. Rotation direction of silicon carbide abrasive wheels minimizing fracturing of TiB₂ coatings.

Figure 4. TiB₂ coating on graphite substrate showing flat coating with some fracturing still occurring.

Figure 5. Highly polished, flat TiB_2 coating on graphite substrate, no fracturing occurring.

Figure 6. Scanning electron micrograph of fresh 600 grit silicon carbide abrasive showing sharp points and edges that fracture TiB_2 coatings.

Figure 7. Scanning electron micrograph of worn silicon carbide abrasive showing rounded and flattened points and edges that result in polishing of TiB_2 coatings.

Figure 8. Polished and electrolytically etched TiB_2 on graphite substrate showing columnar grain structure and banding.

Figure 9. Intermetallic phase at the interface of the TiB_2 coating and the Kovar substrate.

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