JTTEE 2: 287-294

A Comparison of Techniques for the Metallographic Preparation of Thermal Sprayed Samples

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Metallographic preparation of thermal spray coated samples is often difficult because hard and soft materials, which normally require different polishing techniques, are commonly present in a single spray-coated sample. In addition, the microstructures of many spray-deposited materials make them prone to pull-out damage during cutting, grinding, and polishing operations. This study compares alternative metallographic techniques to prepare three common types of thermal sprayed coatings: (1) a plasma sprayed alumina-titania wear coating, (2) a plasma sprayed zirconia thermal barrier coating, and (3) a high-velocity oxy-fuel (HVOF) sprayed tungsten-carbide/cobalt (WC/Co) hard coating. Each coating was deposited onto a steel substrate and was prepared with metallographic protocols based on silicon carbide (SiC) papers, bonded diamond platens, and diamond slurries. Polishing with SiC papers generally produced edge rounding and significant pull-out, which increased the apparent porosity of the coatings. Polishing with bonded diamond platens produced less edge rounding, but some pull-out was still observed. Preparation by diamond slurry lapping consistently produced the best overall results. Porosity artifacts produced by polishing with SiC papers and bonded diamond platens also resulted in spuriously low hardness values for the WC/Co samples; however, hardness results for the two ceramic coatings were not affected by the polishing method.

1. Introduction

METALLOGRAPHIC examination is a key analytical tool for research, developmental, and production thermal sprayed materials. Qualitative and quantitative metallographic techniques are widely used to investigate various phenomena such as possible cracking in the coating, the amount and distribution of phases (e.g., oxides), the degree of melting of individual particles, the porosity content, and the integrity of coating/substrate or splat boundary interfaces.[1-4] To achieve an accurate analysis, the true microstructure of the coating must be preserved during metallographic sample preparation. Unfortunately, a typical coated sample often contains two or more materials with drastically different properties that would normally require very different grinding/polishing protocols. As a result, developing proper cutting and polishing techniques for specific thermal spray coatings can pose challenging problems. For example, pull-out is a common problem that can substantially increase the apparent porosity in polished coating samples, especially with brittle coating materials such as ceramics and tungsten-carbide/cobalt (WC/Co) cermets. Conversely, for coatings that contain soft, ductile phases, smearing of these ductile materials can produce artificially low apparent porosity values. In some cases, other measured properties, such as microhardness, can also be influenced by the procedures used to prepare a metallographic sample.

In the present work, various procedures have been grouped according to the dominant type of grinding/polishing media. Three common types of grinding/polishing media are silicon carbide (SiC) papers, bonded diamond platens, and diamond suspension slurries. The results for three different thermal spray coatings were compared, each prepared with three different metallographic protocols based on the major types of media described above. The coating materials were selected because they are difficult to polish and because they are of interest for potential project applications unrelated to this metallographic study. The coatings for this study include two ceramics—alumina-titania and partially stabilized zirconia (PSZ). Each ceramic was plasma sprayed onto a mild steel substrate with a nickel-base bond coat. Samples of WC/Co deposited onto mild steel substrates were also prepared using a HVOF spray system.

For each material, apparent microstructures resulting from the three different grinding/polishing methods were compared on the basis of photomicrographs, quantitative image analysis of porosity (% area), and microhardness measurements. Consistent trends in the amounts of artifact produced by each grinding medium were observed, although the polishing procedure for each type of media and each coating material was optimized. Grinding with SiC papers always resulted in edge rounding and substantial pull-out, with a higher apparent porosity. It is noteworthy that edge-retaining plates or other special measures were not used to mitigate edge rounding. The diamond platens produced much less rounding and reduced pull-out. The best results were consistently achieved with the diamond slurries, which provided good overall performance and, in some cases, brought out fine details in the microstructure that were not easily observed in samples prepared with the other polishing media.

Key Words: HVOF materials, image analysis, metallographic preparation, polishing procedures

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2. Experimental Procedure

The following procedures were used to produce, prepare, and analyze the samples in this study. Although some consistent trends were observed in the results of this limited investigation, the results reported here may not be valid for conditions other than those described in this section.

2.1 *Coating Deposition*

The chemical compositions and size ranges of the spray powders for this study are shown in Table 1. The nickel-base bond coating and the two ceramic coatings were plasma sprayed onto mild steel substrates prepared by acetone/alcohol degreasing and grit blasting with coarse (~36 mesh) aluminum oxide. These coatings were sprayed with a Miller Thermal Technologies model SG-100 gun equipped with a 40 kW, subsonic electrode set. Argon was used as the primary arc gas and powder carrier gas, and the auxiliary gas was helium. The gun-to-substrate distance was approximately 9 cm in all cases. The powder feed rates and power levels used to spray the various materials were as follows: 3.1 kg/h bond coat at 27 kW; 2.0 kg/h alumina-titania at 30 kW; and 2.2 kg/h PSZ at 33 kW.

Miller Thermal Technologies provided the HVOF sprayed WC/Co samples for this study. These samples were sprayed with a Miller Top Gun™ HVOF system operated at 307 L/min (650 SCFH) oxygen and 64 L/min (135 SCFH) propylene, with 17 L/min (35 SCFH) of argon as the powder carrier gas. The WC/Co powder was fed at a rate of 1.5 kg/h with a gun-to-substrate distance of 19 cm.

2.2 *Metallographic Preparation**

For each material, samples for the three different metallographic procedures were cut from a single piece of spray-coated steel. This was done to avoid possible sample-to-sample variations in coating microstructure. All samples were sectioned with an Isomet™ low-speed diamond saw. Although sample cutting techniques were not specifically investigated in this study, the authors' past experience has shown that a conventional high-speed abrasive cut-off saw can cause extensive damage to coated samples. This damaged material often is not entirely removed by subsequent grinding operations. Although it requires more time to cut a sample with a low-speed diamond saw, this procedure causes much less damage and therefore reduces the likelihood that artifacts caused by the cutting operation will be carried forward into the final polished surface.

The procedure used for sample mounting is also critically important for some materials. The pressure applied to the sample in a normal mold press can sometimes crack or otherwise damage the coating, significantly altering the apparent microstructure of the sample. This is especially true if there are any small burrs along the edge of the sample that prevent it from resting perfectly flat in the mounting press. Also, porous brittle materials, such as a typical PSZ thermal barrier coating, are easily damaged in a conventional mounting press. The best results were found by mounting with Shell EPON 828 epoxy and Diethanolamine (DEA) mixed in a ratio of 100 parts by weight (pbw) epoxy to 12 pbw DEA. This liquid mixture has excellent flow properties, and it will infiltrate porous coatings with surface-connected porosity. It also has sufficient hardness to provide good edge retention in most cases. The samples cut for this study were mounted in 2.5 cm (1 in.) mold rings that had been coated with Miller-Stephens spray-on fluorocarbon mold release agent to prevent adhesion of the epoxy to the mold ring. Prior to mixing and pouring, the samples, mold rings, and epoxy were all preheated to 85 °C to maximize the flow characteristics during the pour. The epoxy/DEA was mixed and then immediately evacuated in a bell jar at 130 to 400 Pa (1 to 3 torr) for 1 min to remove trapped air before pouring. After pouring in an ambient air environment, the mounts were again evacuated for 1 min in the bell jar to remove trapped air and then vented back to atmospheric pressure. This procedure has the added advantage that atmospheric pressure forces the epoxy into evacuated, surface-connected pores in the samples as the molds are vented back to the ambient atmosphere. The molds were then cured in an air furnace at 85 °C for 8 h.

All samples were prepared on an Ecomet™ IV grinder/polisher equipped with a Euromet™ I power head. The diamond slurries were automatically applied with a Metlap™ I programmable dispenser system. Semiautomatic equipment such as this provides good sample-to-sample uniformity and day-to-day reproducibility. The specific protocols for each coating material were empirically optimized on the basis of several trials, and the procedures that produced the best results are summarized in Table 2. The parameters in Table 2 are largely a product of accumulated experience, and it is difficult to provide a detailed explanation for each of the parameters listed. However, a few general comments may provide guidance for optimizing parameters on other materials or equipment.[5,6] In the initial stages of grinding, the objective is to remove any damage from the cutting operation and to get the sample flat. Hence, relatively coarse abrasives, high platen steels, and counter rotation (i.e., the sample holder rotates in the opposite direction relative to the rotational direction of the grinding platen) are used to rapidly remove material from the sample. As the process proceeds, the objective is to remove the scratches and damage from the previous grinding or polishing step, while minimizing new damage to the surface so that there is a gradual progression to finer abrasives with slower rotation speeds and a transition from counter rotation to concurrent rotation of the sample holder and polishing wheel. The specific number of steps, speeds, etc., varies greatly from one material to the next and must be determined by experimentation, drawing on personal experience or recommended procedures for similar materials.

Table 1 Spray powder compositions and particle sizes

Powder type	Composition, wt%	Particle size, μm
Metal bond coat	88Ni-6Al-6Mo	−106, +44
Alumina-titania	$97Al_2O_3$-$3TiO_2$	−44, +15
PSZ (fused)	$92ZrO_2$-$8Y_2O_3$	−75, +44
Tungsten carbide/cobalt	83WC-17Co	−44, +15

*Except as specifically noted in the following descriptions, all of the metallographic equipment and consumables used in this study were purchased from Buehler Ltd. of Lake Bluff, Illinois. Hence, the tradenames used to describe various equipment or materials refer to Buehler products unless otherwise noted.

Two items in Table 2 that merit a brief explanation are the use of a SiC paper in the bonded platen procedure and the final polishing step for the WC/Co samples. The SiC paper was used instead of a fine diamond plate because the authors' experience has shown that samples such as those studied here rapidly destroy the surface of a 15-μm diamond platen. Such platens work well on a monolithic hard material, such as a piece of ceramic, but the presence of a softer material, in this case the mild steel substrate, results in severe damage to the platen surface. The mechanism responsible for this damage was not studied, but it quite probably is due to hard particles that become embedded in the soft sample material. Therefore, to avoid destroying an expensive platen, the SiC paper was used for this step. With regard to the final lapping step for the WC/Co samples, the hardness of the Metlap™ 2 platen had no bearing on the lapping action. This particular platen was chosen primarily because it had never been used, and it was therefore exceptionally flat from edge to edge. Almost any platen could be used for this step, provided that it is flat, because flatness is critical for the fine lapping operation. The Texmet™ perforated cloth used for this step is a chemotextile material that is useful for hard materials, such as cemented carbides, ceramics, and petrographic samples. In the authors' experience, a perforated cloth results in less pull-out in this type of fine lapping operation.

Final polishing of all samples was performed on a Vibromet™ vibratory polisher. The primary purpose of vibratory polishing is to remove fine scratches (see Fig. 1) from the previous polishing step. Vibratory polishing may also help to mitigate the effects of smearing in some instances, because it is nondirectional and tends to remove thin smeared layers. During vibratory polishing, each sample was weighted with 400 g, and the polisher was adjusted so that the samples made approximately six revolutions per minute (rev/min) around the polishing basin. The ceramic samples were vibratory polished with nylon cloth and a colloidal silica solution for 2 to 4 h. However, attempts to use a similar procedure for the WC/Co samples resulted in severe chemical attack of the cobalt phase, even for relatively short polishing times. For the WC/Co samples, much better results were achieved with a Texmet™ napless cloth using a Masterpolish™ solution for only 15 to 30 min.

Fig. 1 Photomicrograph of WC/Co sample showing fine scratches before vibratory polishing. Compare with the final vibratory polished surface in Fig. 3.

Table 2 Summary of metallographic procedures

Media	Abrasive	Size, μm	Time, min	Speed, rpm	Relative rotation	Dispensing sequence
Grinding/polishing schedule for silicon carbide paper (used for all samples)						
Carbimet™ paper	SiC	60	1.5(a)	400	Counter rotation	Steady H_2O
	SiC	44	1.5(a)	400	Counter rotation	Steady H_2O
	SiC	38	1.5(a)	200	Counter rotation	Steady H_2O
	SiC	20	1.5(a)	200	Counter rotation	Steady H_2O
Texmet™ cloth	Diamond	6	2	150	Counter rotation	None
Nylon cloth	Diamond	1	1	150	Concurrent rotation	None
Grinding/polishing schedule for bonded diamond platens (used for all samples)						
Bonded platen	Diamond	45	Until plane	175	Counter rotation	Steady H_2O
	Diamond	30	4	175	Counter rotation	Steady H_2O
Carbimet™ paper	SiC	20	1.5	150	Counter rotation	Steady H_2O
Texmet™ cloth	Diamond	6	2	150	Counter rotation	None
Nylon cloth	Diamond	1	1	150	Concurrent rotation	None
Grinding/polishing schedule for diamond suspension lapping (used for the ceramic samples)						
Bonded platen	Diamond	45	Until plane	250	Counter rotation	Steady H_2O
Metlap™ 8 platen	Diamond	30	6	200	Counter rotation	1 s on, 10 s off
Metlap™ 4 platen	Diamond	6	10	120	Concurrent rotation	1 s on, 20 s off
Grinding/polishing schedule for diamond suspension lapping (used for WC/Co samples)						
Bonded platen	Diamond	45	Until plane	250	Counter rotation	Steady H_2O
Metlap™ 8 platen	Diamond	30	6	75	Concurrent rotation	1 s on, 10 s off
Metlap™ 2 platen + perforated Texmet™ cloth	Diamond	6	2	50	Concurrent rotation	1 s on, 10 s off

Note: For all grinding/polishing operations, the applied force was 2.3 kg per sample. (a) Each SiC paper was used only 1.5 min. Some steps required more than one paper.

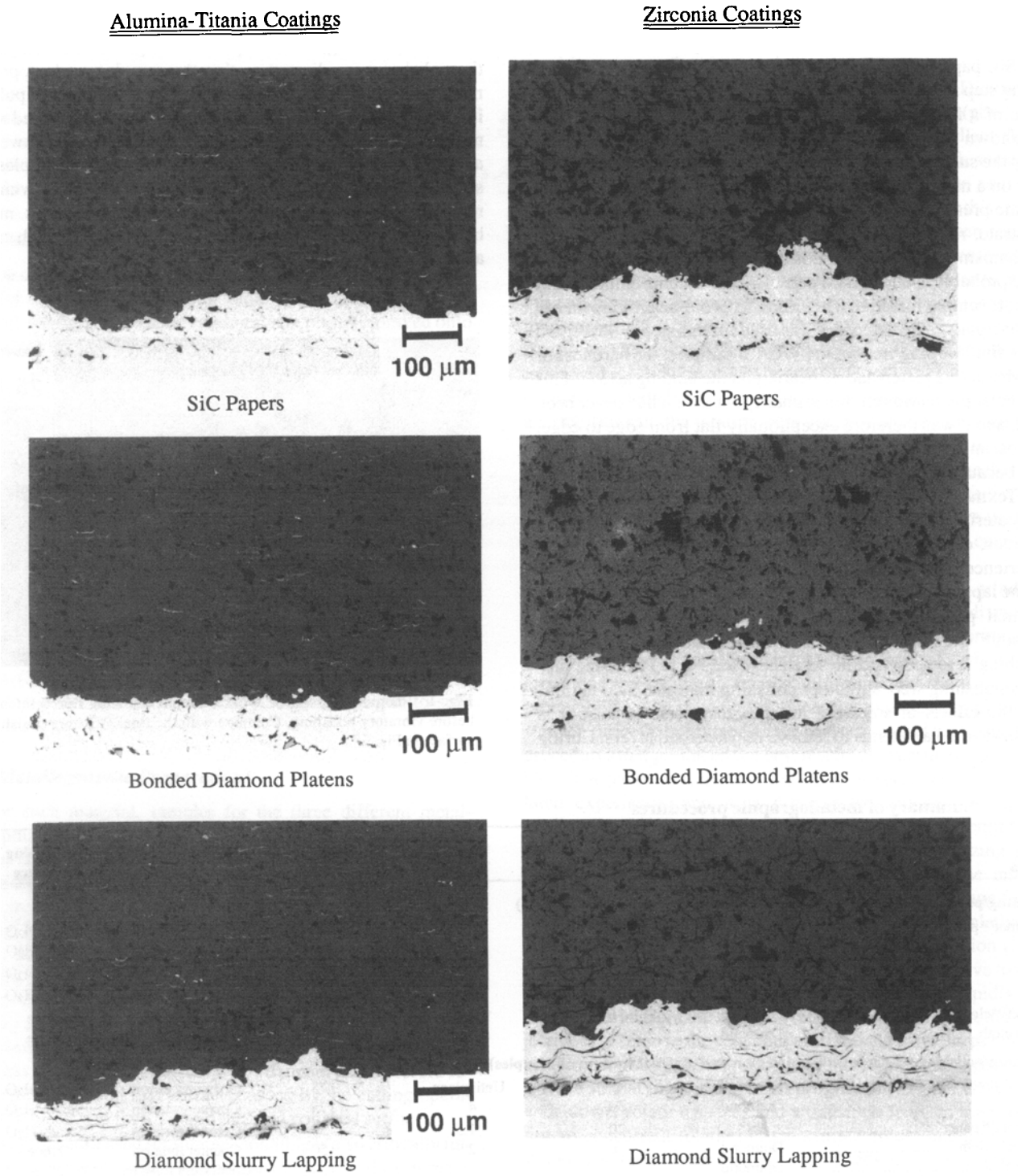

Fig. 2 Comparison of alumina-titania and yttria-stabilized zirconia coatings prepared with three different grinding/polishing media. All micrographs are at the same magnification.

2.3 *Photographs and Image Analysis*

Photomicrographs of each sample were prepared on a Leco model 300 metallograph. Quantitative image analysis of the polished sections for porosity (% area) was performed with a Unitron Versamet II metallographic microscope coupled to a Dapple image analysis system running on an Apple MacIntosh IIX computer. The resolution for the image analysis was 1.6 pixels/μm^2 based on system calibration with a known standard. The average porosity and standard deviation of the measurements for each sample were computed on the basis of measurements for 20 fields of view randomly distributed across the sample.

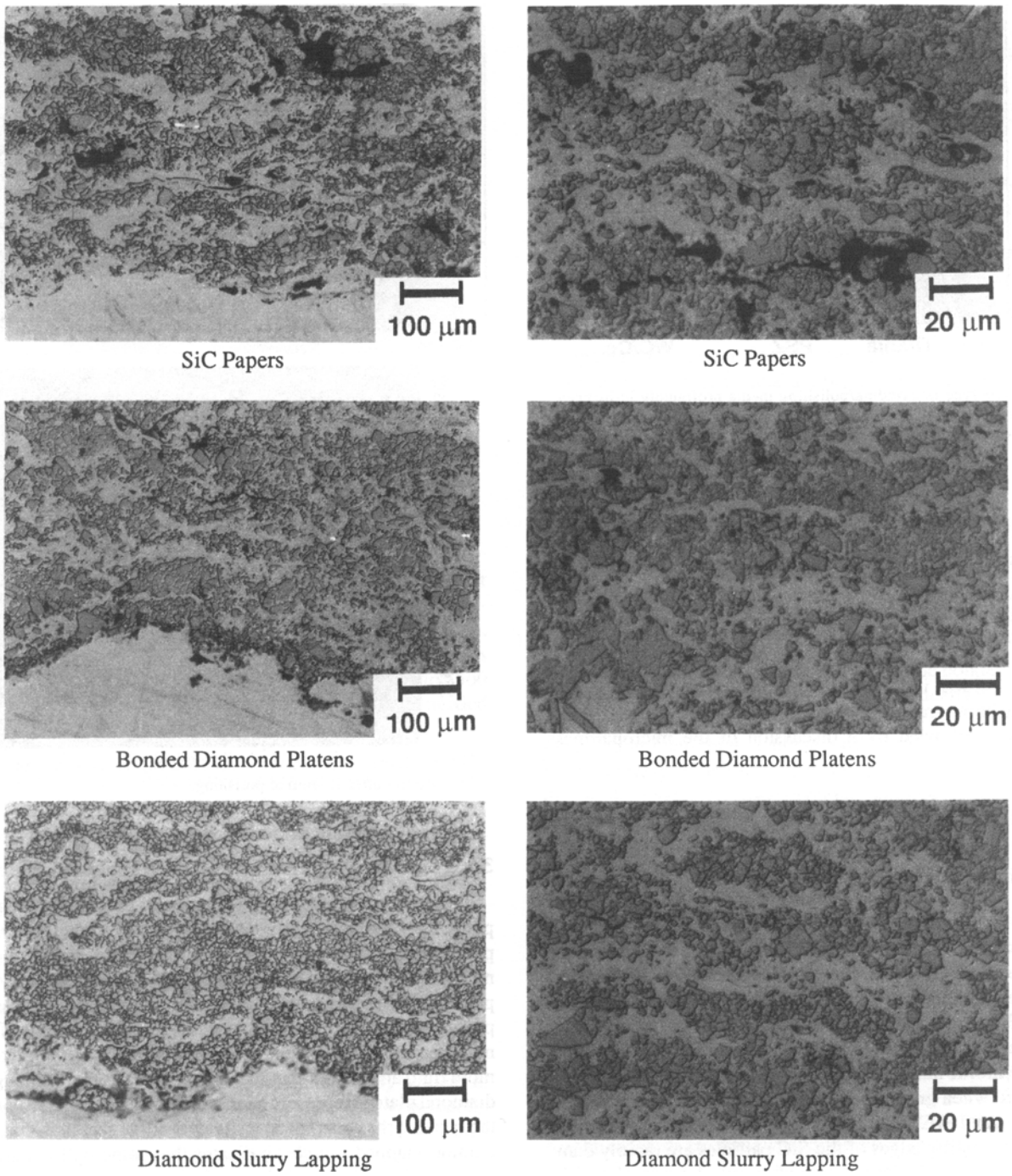

Fig. 3 Comparison of WC/Co coatings prepared with three different grinding/polishing media.

2.4 *Microhardness Measurements*

Microhardness measurements on all of the coatings were made with a Micromet™ digital microhardness tester. Measurements were made with a Knoop indentor using a 200-g load applied for 10 s. Eight measurements were made in random locations on each sample. The purpose of these measurements was to investigate possible variations in apparent microhardness due to differences in the metallographic sample preparation procedures and the consequent variations in artifacts.

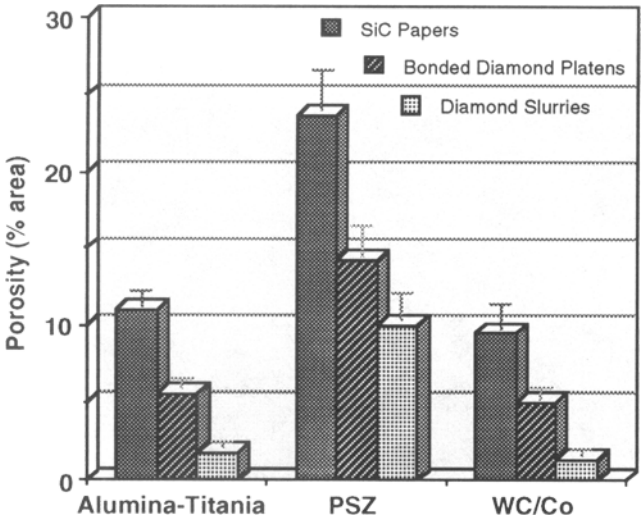

Fig. 4 The type of grinding/polishing media profoundly influences the apparent porosity determined by quantitative image analysis. The error bars represent one standard deviation for 20 measurements.

3. Results and Discussion

For a given sample material, significant differences in the apparent porosity and microhardness of polished samples were observed for different polishing procedures, and some consistent trends are evident in the results for the three materials investigated. The photographic and image analysis results will be presented first, followed by a discussion of the microhardness results.

3.1 *Photographic and Image Analysis Results*

3.1.1 Samples Prepared with SiC Papers

Visual examination of the photomicrographs (Fig. 2 and 3) shows that samples prepared with SiC papers have significantly higher apparent porosity than comparable samples prepared with either of the diamond procedures. The porosity measurements in Fig. 4 confirm that the SiC polished samples are indeed substantially more porous. Because silicon carbide does not have the extreme hardness of diamond, it does not cut as cleanly as diamond when grinding or polishing hard materials, such as the ceramic and WC/Co coatings of this study. As shown in Fig. 5, the sharp cutting edges of the SiC particles are rapidly damaged, with noticeable degradation after only 1.5 min of polishing. The reason for the increased pullout with the SiC paper is not clear, but the inferior cutting action of the SiC may contribute to this problem. Some edge rounding was also clearly evident in microscopic examination of samples prepared with SiC papers. This can probably be attributed to the fact that the paper backing of the SiC abrasive sheets is not perfectly rigid, and a slight flexure of the abrasive surface can occur during grinding/polishing operations. The results indicate that SiC paper is not a desirable method for metallographic preparation under the conditions of this study.

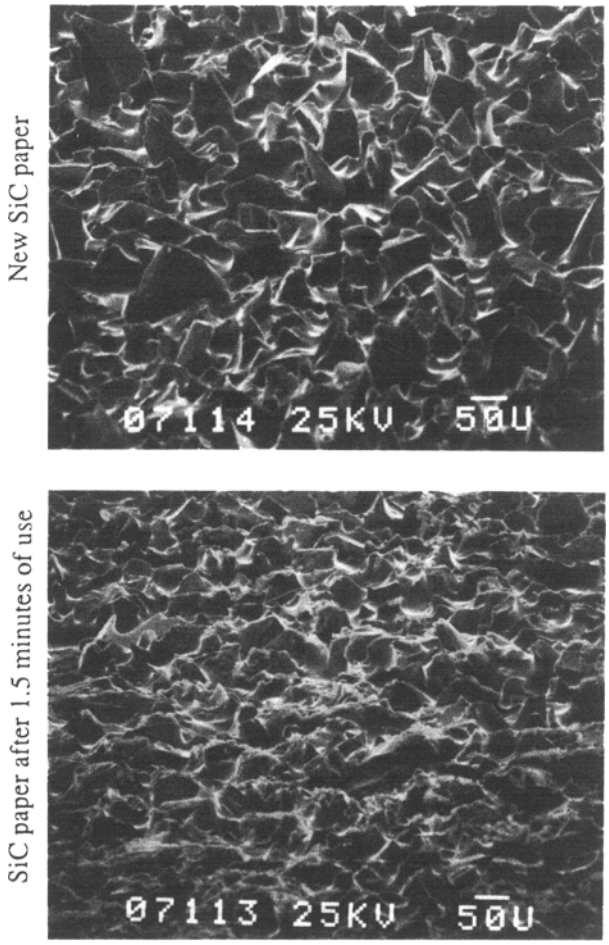

Fig. 5 Comparison of new and used SiC papers showing wear of the SiC particles after 1.5 min of polishing.

3.1.2 Samples Prepared with Bonded Diamond Platens

As shown in Fig. 2 to 4, the degree of porosity produced by preparation is consistently much lower in the bonded diamond platen samples than in comparable SiC samples, but it is still significantly higher than the porosity in the diamond slurry samples. Also, the edge rounding observed with the SiC preparation procedure was not observed with the diamond platens. This is not unexpected, because the abrasive surface of the platens is more rigid and cannot flex like the carbide papers. Although the diamond platens represent an improvement over the SiC papers, the fact that the apparent porosity was still higher than that of the diamond-lapped samples indicates that some pull-out still occurred with the diamond platens. Pull-out of individual WC particles is supported by microscopic examination at higher magnification. At higher magnification, individual carbide particles within a cemented carbide region bear some resemblance to highly angular pieces of a tiny "mosaic," and there are clear examples where one or multiple pieces of the mosaic have apparently been removed, leaving characteristic angular cavities or pores in the cemented carbide material.

Based on accumulated laboratory experience, the authors also noted that wear rates for bonded diamond platens can be quite high when polishing extremely hard materials. Wear of

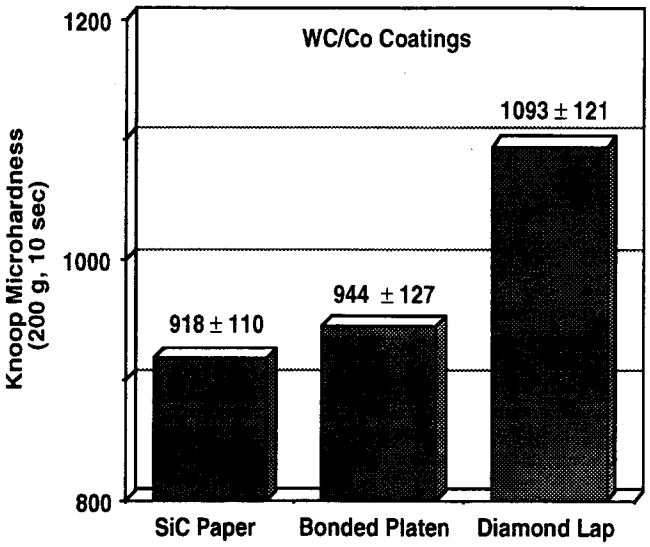

Fig. 6 Comparison of Knoop microhardness results for WC/Co samples. The hardness results for the samples prepared by diamond lapping are significantly higher than the measured results for samples prepared with SiC papers or bonded diamond platens.

these platens appears in the form of scratches, gouges, and/or discoloration (i.e., bluing from local heating), and there are significant increases in polishing time as a platen becomes worn. The useful lifetime of such platens can be as little as 30 samples in extreme cases. Because these platens cost about $400 to $500 each (1992 U.S. dollars) and because several platens are needed for a typical grinding/polishing procedure, bonded diamond platens may not be the most cost-effective method for metallographic preparation of the materials in this study.

3.1.3 Samples Prepared with Diamond Slurry Lapping

Once again referring to the results presented in Fig. 2 to 4, it is apparent that lapping with diamond slurries produced the best results for all of the sample materials in this study. The PSZ thermal barrier coating in this study was deliberately deposited with a high level of porosity to decrease the thermal conductivity of the coating. It is probable that the higher true porosity of this ceramic coating makes it more susceptible to damage by the aggressive grinding action of the bonded diamond abrasive. Microcracking of the PSZ coating, which is clearly evident in the lapped sample in Fig. 2, can only be seen with very careful microscopic examination of the bonded platen and SiC-polished PSZ samples. Hence, the diamond slurry lapping procedure provided better definition of small details in the PSZ microstructure. The higher magnification photos of the lapped WC/Co sample in Fig. 3 also illustrate the striking definition produced with diamond lapping. It is clear from a comparison of the photos in Fig. 3 that the lapping procedure resulted in substantially less pull-out of carbide particles than either the SiC paper or bonded platen methods. The lower porosity values for the lapped ceramic coatings in Fig. 4 are also presumed to be more reflective of the true porosity in the coatings, because pull-out has been reduced, and these hard, relatively brittle ceramics are not prone to smearing.

For the hard materials of interest in this study, lapping with diamond slurries also appears to be cost effective. Although the initial cost for the diamond lapping system is high, the subsequent cost per sample is relatively low. Like the bonded platens, the diamond lapping platens cost about $400 to $500 each. However, unlike the bonded platens, when the lapping platens become worn, they can be dressed with a special accessory kit to restore flatness. With proper care and maintenance, their useful lifetime can thus be extended for many years of service. The cost of the diamond suspension slurries is about $50 to $100 per pint depending on the size of the diamond particles. However, only very small amounts of the slurry are used for a given grinding/polishing operation. Based on the authors' experience, it is reasonable to expect a pint of slurry to last for several hundred samples in typical polishing operations. The automatic slurry dispensing system is very convenient, but it is not an essential component of the lapping system. Excellent results can be achieved by periodic manual application of the diamond slurries from spray dispensers; however, this method does require regular monitoring during the lapping operations.

3.2 *Microhardness Results*

The microhardness results for the WC/Co samples are shown in Fig. 6. A statistical "*t*" test comparison of the average hardness results at a 95% confidence level shows that the samples prepared with diamond lapping have a significantly higher average hardness than samples prepared with either SiC papers or bonded diamond platens. The difference between the average hardness results for the SiC papers versus the bonded diamond platens was much smaller and was not statistically significant at the 95% confidence level. Nevertheless, it is interesting that the low, medium, and high average hardness results in Fig. 6 uniformly correspond to the high, medium, and low porosity results in Fig. 4. Hence, the lower average hardness of samples prepared with SiC papers and bonded diamond platens can probably be attributed to the loss of the hard carbide phase and the attendant increase in porosity, which results from increased pull-out with these two polishing methods.

Differences in the microhardness results for the alumina-titania samples and the PSZ samples were all small in comparison to the measurement uncertainty, with no consistent relationship between the preparation method and microhardness. The observation that these results do not show the same apparent relationship to porosity as the WC/Co results may be related to the fact that these ceramic coatings are inherently more brittle than the metal-matrix WC/Co samples. Therefore, it is probable that the failure mechanisms under the highly localized point load of a Knoop indentor are somewhat different in these materials.

4. Summary

Metallographic preparation methods based on SiC papers, bonded diamond platens, and diamond slurries for grinding and polishing of alumina-titania, partially stabilized zirconia, and WC/Co thermal sprayed coatings have been compared. The SiC procedure caused extensive pull-out in all of the sample materi-

als, produced some edge rounding, and resulted in a comparatively lower apparent microhardness for the WC/Co samples. The bonded diamond platens produced slightly better results than the SiC papers, but these samples were still inferior to the diamond slurry lapping method. For the hard ceramic and cemented carbide materials of this study, rapid wear of bonded diamond platens can also result in a very high cost per sample. The diamond slurry lapping method consistently produced the best results for all three coating materials. The lapping procedure minimizes pull-out, provides excellent definition of subtle microstructural details, and can be performed at a very reasonable cost per sample. The measured microhardness of the WC/Co coating prepared by diamond lapping was also higher than the hardness measured for the samples prepared with either the SiC papers or the bonded diamond platens. This difference in apparent microhardness may be related to the significantly reduced level of carbide pull-out with the lapping method.

For the coating materials and metallographic procedures investigated here, diamond slurry lapping appears to offer substantial advantages for minimizing damage during grinding and polishing operations, resulting in a more accurate representation of the true coating microstructure and properties. Diamond slurry lapping may be beneficial for other thermal sprayed materials as well, but further work is needed to verify and extend the applicability of these results to other materials and conditions. It should also be noted that the rudimentary cost comparison presented here is based solely on the experience of the Process Development Metallographic Laboratory at Sandia National Laboratories. Actual relative costs will obviously vary somewhat from one laboratory to the next, depending on the type and volume of materials polished, differences in equipment and consumables costs, local labor costs, etc.

Acknowledgments

The authors wish to thank Miller Thermal Technologies for providing the HVOF sprayed samples for this study. This work was performed at Sandia National Laboratories, operated for the U.S. Department of Energy under contract number DE-AC04-76DP00789.

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