A COMPARISON OF TECHNIQUES FOR THE METALLOGRAPHIC PREPARATION OF THERMAL SPRAYED SAMPLES*

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ABSTRACT

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. We have compared 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) an HVOF (High Velocity Oxy-Fuel) sprayed tungsten-carbide/cobalt (WC/Co) hardcoating. Each coating was deposited onto a steel substrate and was prepared with metallographic protocols based on: a) silicon carbide (SiC) papers, b) bonded diamond platens, and c) diamond slurries. Polishing with SiC papers generally produced edge rounding and significant pull-out that increased the apparent porosity of the coatings. Polishing with bonded diamond platens produced better results, but some pull-out was still observed. In addition, this method is typically the most expensive due to the limited lifetime and high cost of the platens. Preparation by diamond slurry lapping produced the best overall results at a reasonable cost per sample. Porosity artifacts produced by polishing with SiC papers and bonded

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diamond platens also resulted in spuriously low hardness values for the WC/Co samples, but hardness results for the two ceramic coatings were not affected by the polishing method.

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. In order 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 many brittle coatings, 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.

We have grouped various procedures according to the dominant type grinding/polishing media. Three common types of grinding/polishing media are silicon carbide (SiC) papers, bonded diamond platens, and diamond suspension slurries. In this paper, we compare results for three different thermal spray coatings, each prepared with three different metallographic protocols based on the major types of media just described. 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 an HVOF (High Velocity Oxy-Fuel) 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. Although every effort was made to optimize the polishing procedure for each type of media and each coating material, it was possible to identify clear trends in the amounts of artifact produced by each grinding medium. Grinding with SiC papers always resulted in edge rounding and substantial pull-out with a higher apparent porosity. The diamond platens produced less rounding and pull-out, but in some applications the cost per sample may be high due to the expense and limited useful lifetime of the platens. The best results were consistently achieved with the diamond slurries, and the unit cost for the slurry protocols was much lower than for the diamond platens.

EXPERIMENTAL PROCEDURES

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 hr^{-1} bond coat @ 27 kW; 2.0 kg hr^{-1} alumina-titania @ 30 kW; and 2.2 kg hr^{-1} PSZ @ 33 kW.

Table 1: Spray Powder Compositions and Particle Sizes

Powder Type	Composition (wt.%)	Particle Size (μm)
metal bond coat	88 Ni / 6 Al / 6 Mo.	-106, +44
alumina-titania	97 Al_2O_3 / 3 TiO_2	-44, +15
PSZ (fused)	92 ZrO_2 / 8 Y_2O_3	-75, +44
tungsten carbide/cobalt	83 WC / 17 Co	-44, +15

Miller Thermal Technologies provided the HVOF sprayed WC/Co samples for this study. These samples were sprayed with the Miller Top Gun™ HVOF system operated at 307 ltr min^{-1} (650 SCFH) oxygen and 64 ltr min^{-1} (135 SCFH) propylene, with 17 ltr min^{-1} (35 SCFH) of argon as the powder carrier gas. The WC/Co powder was fed at a rate of 1.5 kg hr^{-1} with a gun-to-substrate distance of 19 cm.

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, our past experience has clearly 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, it causes much less damage and therefore produces metallographic specimens with fewer artifacts.

The procedure used for sample mounting is also critically important for some materials. In some cases, the pressure applied to the sample in a normal mold press can crack or otherwise damage the coating, significantly altering the apparent microstructure of the sample. This is especially true for porous brittle materials, for example, a typical PSZ thermal barrier coating. We have found the best results 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 sufficient hardness to provide good edge retention. The cut samples 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

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†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.

to maximize the flow characteristics during the pour. The epoxy/DEA was mixed and then immediately evacuated at 1-3 Torr for 1 minute to remove trapped air before pouring. After pouring, the mounts were also evacuated at 1-3 Torr to remove trapped air, and they were then cured at 85 °C for 8 hours.

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. Semi-automatic equipment such as this provides good sample-to-sample uniformity and day-to-day reproducibility that is difficult for anyone but a highly experienced metallographer to achieve with traditional manual grinding/polishing methods. 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. Final polishing of all samples was performed on a Vibromet™ vibratory polisher. Each sample was weighted with 400 grams during the vibratory polish. The ceramic samples were vibratory polished with a nylon cloth and a colloidal silica solution for 2-4 hours. 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 15-30 minutes.

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 twenty fields of view randomly distributed across the sample.

Table 2: Summary of Metallographic Procedures††

Media	Abrasive	Size (μm)	Time (minutes)	Speed (rpm)	Relative Rotation	Dispensing Sequence
Grinding/Polishing Schedule for Silicon Carbide Paper (Used for all Samples)						
Carbimet™ paper	SiC	60	1.5*	400	counter rotation	steady H_2O
Carbimet™ paper	SiC	44	1.5*	400	counter rotation	steady H_2O
Carbimet™ paper	SiC	38	1.5*	200	counter rotation	steady H_2O
Carbimet™ paper	SiC	20	1.5*	200	counter rotation	steady H_2O
Texmet™ cloth	diamond	6	2	150	counter rotation	none
nylon cloth	diamond	1	1	150	concurrent rotation	none
* Each SiC paper was used only 1.5 minutes. Some steps required more than one paper.						
Grinding/Polishing Schedule for Bonded Diamond Platens (Used for all Samples)						
bonded platen	diamond	45	until plane	175	counter rotation	steady H_2O
bonded platen	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 sec on, 10 sec off
Metlap™ 4 platen	diamond	6	10	120	concurrent rotation	1 sec on, 20 sec off
Grinding/Polishing Schedule for Diamond Suspension Lapping (Used for the 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 sec on, 10 sec off
Metlap™ 2 platen + perforated Texmet™ cloth	diamond	6	2	50	concurrent rotation	1 sec on, 10 sec off

†† For all grinding/polishing operations on this table, the applied force was 2.3 kg per sample.

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 seconds. 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 resulting variations in artifacts.

RESULTS AND DISCUSSION

A simple visual examination of the photomicrographs in Fig's 1 and 2, 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. 3 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 pure ceramic and WC/Co coatings of this study. The inferior cutting action of SiC is the apparent cause of the pull-out and increased porosity in these samples. Some edge rounding was also clearly evident in microscope 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 of the coating materials in this study.

As shown in Fig's. 1-3, the degree of porosity produced by preparation is consistently much lower in the fixed diamond platen samples than in comparable SiC samples, but it 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, since the abrasive surface of the patens is more rigid and cannot flex like the carbide papers. Although the diamond platens represent an improvement over the SiC papers, the resulting metallographic samples still had apparent pull-out. Based on accumulated laboratory experience, we have also noted that wear rates for bonded diamond platens can be quite high when polishing extremely hard materials. The useful lifetime of such platens can be as little as 30 samples in extreme cases. Since these platens cost roughly $400-$500 each, and since several

platens are needed for a typical grinding/polishing procedure, bonded diamond platens may not be a very cost effective method for metallographic preparation of the materials in this study.

Once again referring to the results presented in Fig's 1-3, it is apparent that lapping with diamond slurries produced the best results for all of the sample materials in this study. PSZ thermal barrier coatings are deliberately sprayed with high porosity levels 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 photo of Fig. 1, can only be seen with very careful microscopic examination of the bonded platen and SiC polished PSZ samples. Hence, the diamond slurry lapping procedure provides better definition of small details of the PSZ microstructure. The higher magnification photos of the lapped WC/Co sample in Fig. 2 also show the striking definition produced with diamond lapping. It is clear from a comparison of the photos in Fig. 2 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.3 are also presumed to be more reflective of the true porosity in the coatings, since 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. The diamond lapping platens also cost $400-$500 each, but unlike the bonded diamond platens, they can be dressed to restore flatness as they wear. 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 roughly $50-$100 per pint depending upon the size of the diamond particles. However, only very small amounts of the slurry are used for a given grinding/polishing operation. Based on our own experience, it is reasonable to expect a pint bottle of slurry to last for several hundred samples in typical polishing operations. The automatic slurry dispensing system is a very convenient, but 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.

The microhardness results for the WC/Co samples are shown in Fig. 4. 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. 4 uniformly correspond to the high, medium, and low porosity results in Fig. 3. 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 resulting 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 preparation method and microhardness. The fact 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.

SUMMARY

We have compared 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. The SiC procedure caused extensive pull-out in all of the sample materials, 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 clearly 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.

Based on the coating materials 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.

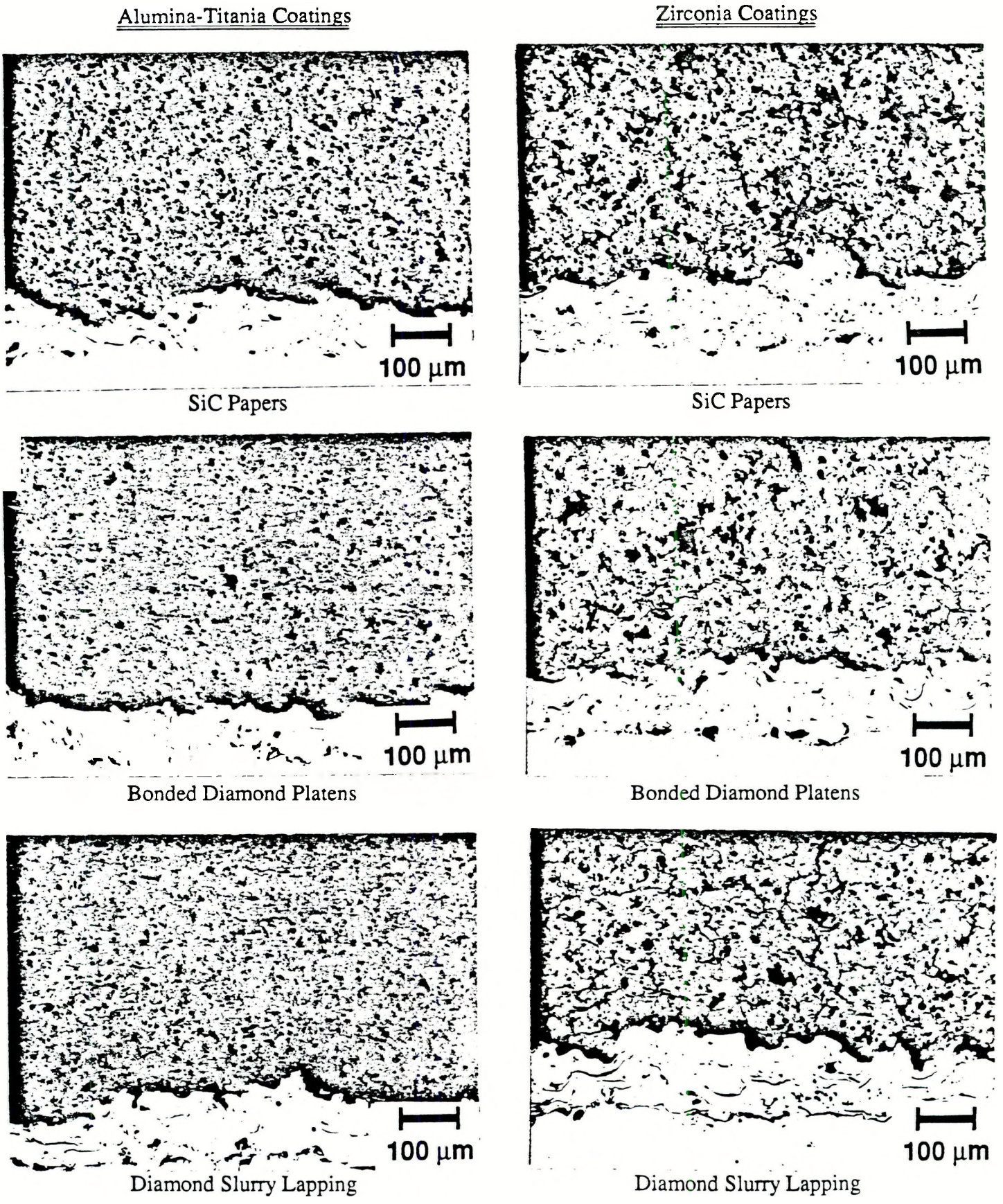
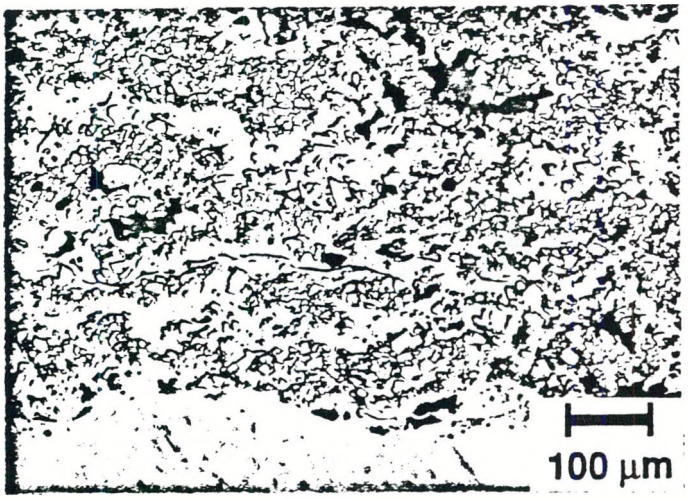
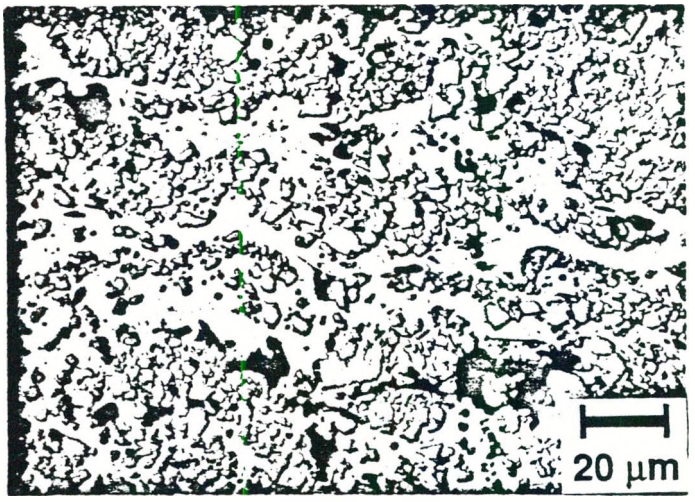


Fig. 1: Comparison of ceramic coatings prepared with three different grinding/polishing media.

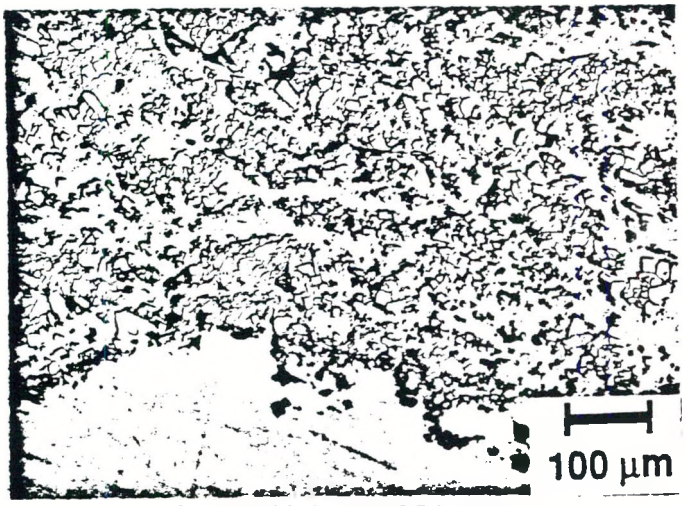
WC/Co Coatings



SiC Papers

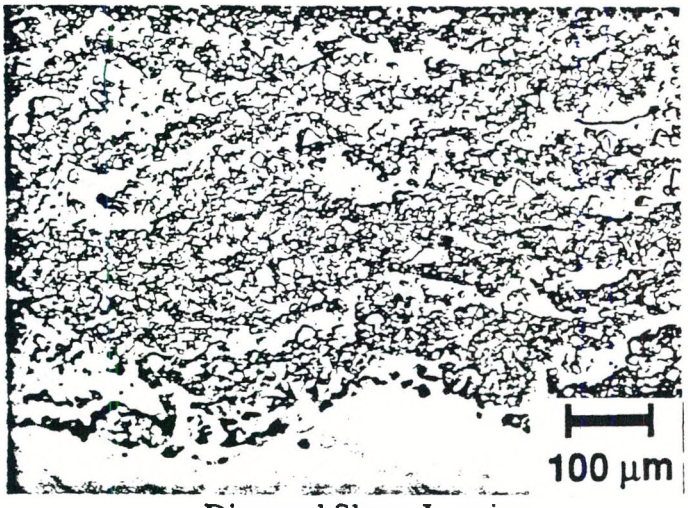


SiC Papers

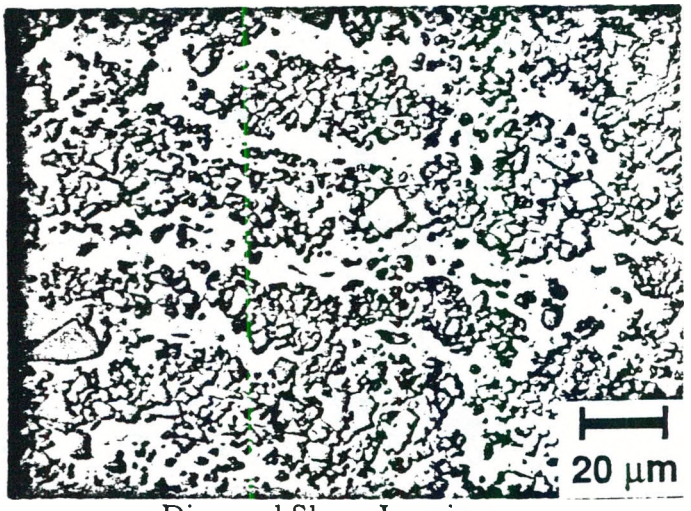


Bonded Diamond Platens

Bonded Diamond Platens



Diamond Slurry Lapping



Diamond Slurry Lapping

Fig. 2: Comparison of WC/Co coatings prepared with three different grinding/polishing media.

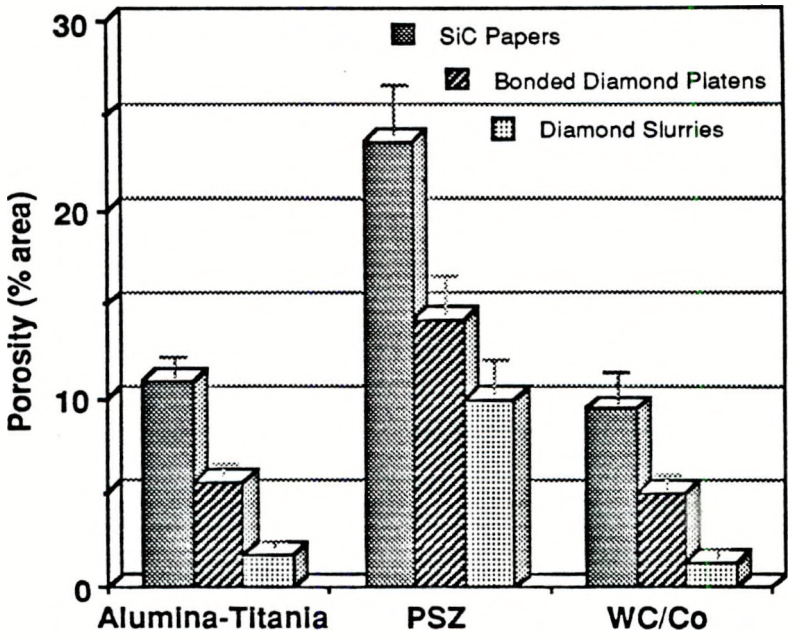


Fig. 3: The type of grinding/polishing media profoundly influences the apparent porosity determined by quantitative image analysis. The indicated error bars represent one standard deviation for twenty measurements

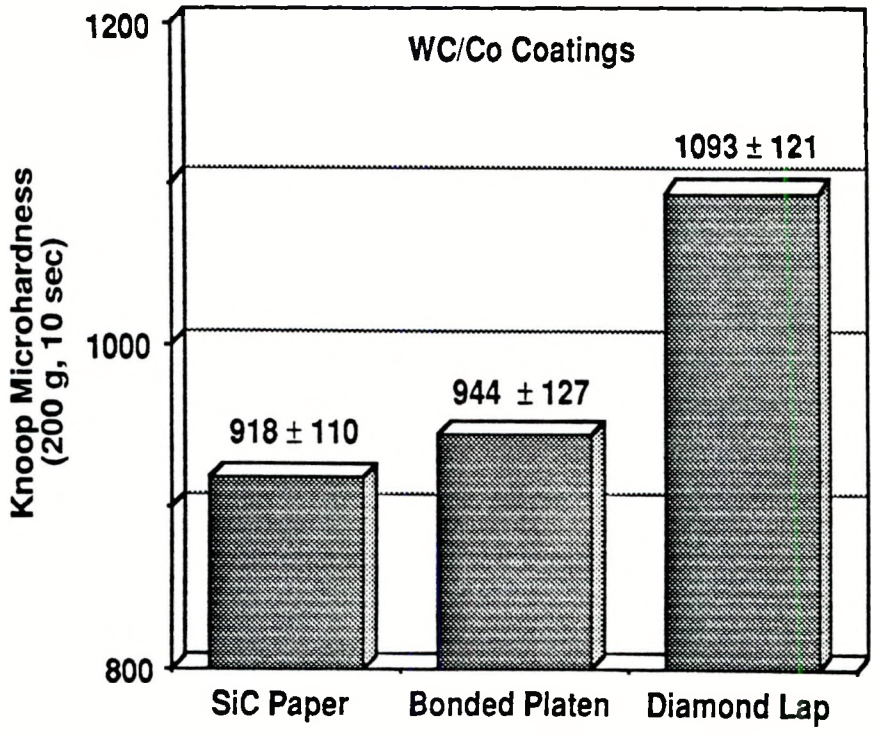


Fig. 4: Comparison of microhardness results for WC/Co samples.