

Revealing True Porosity in WC-Co Thermal Spray Coatings

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The principles underlying composite material behavior during metallographic preparation of coating cross-sections are generally not well understood. This study of the effect of extended fine polishing on apparent porosity shows that adequate polishing times, using a fine abrasive (3 μm) and low force, are required to remove prior deformation in the section surface and to reveal the true porosity of the underlying composite material. Insufficient polishing times can result in considerable underestimation of porosity. A model is described which proposes that the deformation induced in the material during grinding and polishing, even at low applied force, results in smearing of material into voids that exist in the plane of the section.

Keywords HVOF coatings, metallography, porosity, WC-Co composites

1. Introduction

Though much progress has been made in understanding the specific requirements for the proper metallographic preparation of composite thermal spray coatings, there are fundamental principles that are still poorly understood throughout the industry. One firmly established misconception that requires serious consideration is the tendency for producers to accept the metallographic preparation that provides them with the most dense or "best" looking coating, regarding evidence of porosity as being due to "particle pullout."

The problem stems from the structural inhomogeneity of the composite materials, which can be composed of materials of quite different mechanical properties. For example, in the case of a WC-Co composite coating, the difference in properties of the component phases is considerable. Tungsten monocarbide is a very brittle material with a hardness between 1300 and 2200 Vickers hardness (HV), depending on crystallographic direction (Ref 1). The cobalt binder material is much more ductile and likely to have hardness below 300 HV, taking into account the hardening effect of the dissolved tungsten and carbon and the deformation induced during spraying. A further complication is the hardness of the coated substrate material (usually mild or stainless steel), which is usually less than 250 HV (Ref 2). Given the structural complexity of this system, great care must be taken in preparing cross sections to achieve a true representation of the microstructure.

Traditional methods of grinding and polishing, incorporating SiC abrasive papers, are generally not recommended for WC-Co coating layers due to low cutting and cost efficiency and smearing of the coating material into pores (Ref 3). Other reported disadvantages include poor edge retention and significant hard particle pullout (Ref 4). Bonded diamond grinding disks have produced better results with better edge retention and

less particle pullout (Ref 4), but the disks induce considerable smearing deformation in the coating section due to the fixed orientation of the diamond abrasive particles (Ref 5). They are also very expensive and, in the authors' experience, have a short life when applied to materials containing a relatively ductile component such as the ferrous substrate.

There are a number of significant developments that have been made in this field that have contributed to a more reliable and economical determination of coating microstructure. The first development is the substitution of lapping techniques for bonded diamond grinding disks. These techniques ensure the most efficient removal of material with minimum deformation of the coating (Ref 6) and provide a more cost effective alternative to bonded diamond platens. The second development involves the vacuum impregnation of coatings with cold-mounting resin. This has the effect of filling the pores prior to metallographic preparation, making it more difficult for hard particles to smear over (or become embedded in) preexisting pores (Ref 3). However, it should be noted that this is a significant benefit in the case of porosity open to the coating surface through which resin can penetrate into the coating, and that even resin filled pores can be closed during grinding and polishing.

It was suggested that the apparent porosity of a coating section should become stable after smearing from grinding and/or coarse polishing steps is removed (Ref 7). However, adequate fine polishing times must be employed, and methods that use short (less than 2 min) stages are not recommended (Ref 6). The goal of the present work is to define an adequate polishing time for a WC-12%Co coating by investigating the effect of lapping time on the apparent porosity in the coating section.

2. Experimental

WC-12%Co coatings produced by high-velocity oxygen fuel (HVOF) spraying were vacuum impregnated using alumina-filled cold-mounting epoxy resin. The epoxy resin components were mixed according to the manufacturer's recommendations (15 parts resin to 2 parts hardener by volume), and the alumina filler material, used for better edge retention, was added to the

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mixed resin in the volume proportion 1:1. Coating sections were cut from the resin impregnated samples using a bakelite-bonded alumina cut-off wheel at 3000 rpm and a cutting rate of 0.02 mm/s. Sections were then mounted in the alumina-filled epoxy resin and mixed as previously described. Table 1 outlines the metallographic method employed in the present work. The samples were lapped on a Struers Rotopol automatic polishing machine using a single sample loading mode. This negated the need for a planar grinding stage. The coarse lapping stage was performed using a Struers MD-Allegro lapping disk, while the fine lapping used a Struers DP-Plan napless cloth. Abrasives were polycrystalline diamond in the form of a liquid suspension and were added to the lapping surface prior to each 3 min polishing interval. The oil-based lapping lubricant (Struers Blue Lubricant) was added regularly for one second at ten second intervals. No coarse grinding stage was employed, as it has been the authors' experience that a sufficiently good section can be obtained by the previously stated cutting method enabling direct progress to a coarse lapping stage. This has further reduced the number of stages required and consequently reduced associated costs. (Struers Rotopol automatic polishing machine, Struers MD-Allegro lapping disk, Struers DP-Plan napless cloth, and Struers blue lubricant, all belong to Struers, Rodovre, Denmark.)

3. Results

The mounted section was coarse lapped (9 μm) using the specified conditions for approximately 15 min until the entire

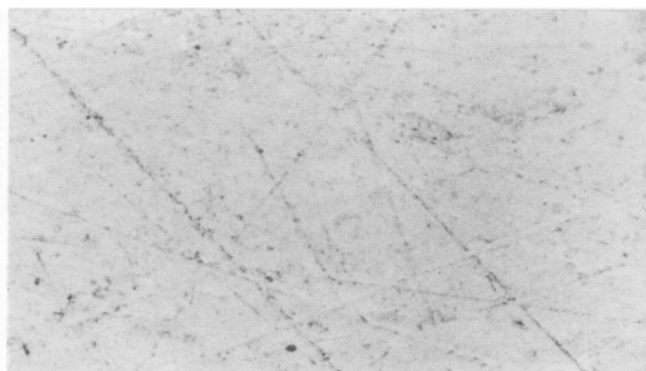
sample surface was plane and obvious deformation from the cutting stage was removed. The coarse-lapped section shown in Fig. 1(a) reveals a structure almost porosity free. After 3 min of fine-lapping using 3 μm diamond abrasive (Fig. 1b), the coating appears to be porosity and crack free, and an excellent polished surface is apparent.

At this stage, operators might be satisfied and cease polishing. However, as the following micrographs show, continuation of the polishing process reveals a progressive increase of apparent porosity, which reaches a maximum after approximately 15 min. This is consistent with the theory that the cutting and grinding stages smear material over voids in the coating, which are only exposed after significant material removal at low force using fine abrasive size.

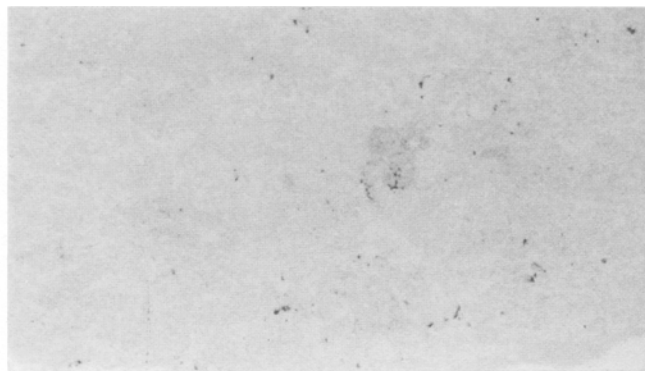
Figure 2 is a schematic illustration of the subsurface deformation, which is responsible for altered structure in lapped porous composite materials. Figure 2(a) shows that after 9 μm

Table 1 Method for preparation of WC-Co coating

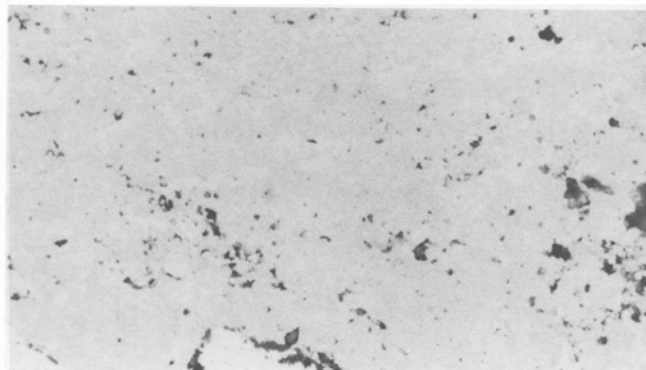
Parameter	Lapping stages	
	Coarse	Fine
Disk/cloth	Hard composite	Napless cloth
Abrasive type	Diamond	Diamond
Abrasive size, μm	9	3
Force, N/sample	30	20
Speed, rpm	150	150
Rotation	Concurrent	Concurrent
Lubricant	Water-based	Water-based



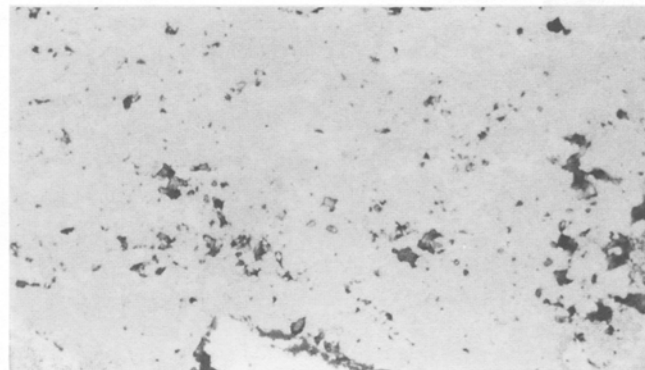
(a)



(b)



(c)



(d)

Fig. 1 Appearance of coating cross section after (a) coarse-lapping (9 μm) for 15 min and fine-lapping (3 μm) for (b) 3, (c) 9, and (d) 15 min. 605 \times

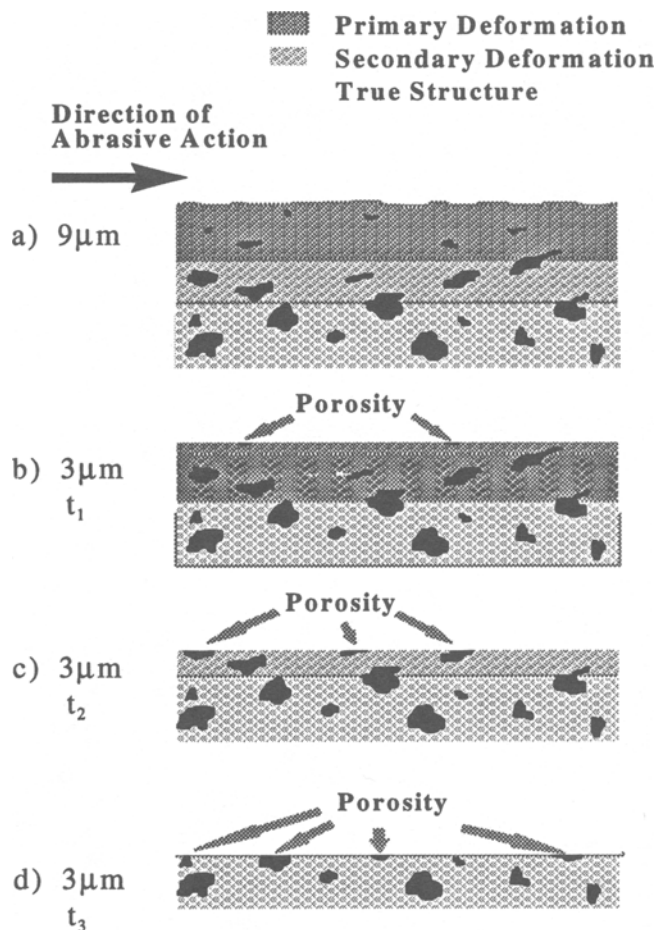


Fig. 2 Schematic illustration of the stages of metallographic preparation necessary to reveal true porosity in the coating cross section; $t_3 > t_2 > t_1$

lapping, the layer(s) of material immediately below the abraded surface is deformed to an extent dependent on the force applied during lapping.

This deformed layer is drawn as two distinct layers of primary and secondary deformation, respectively, to illustrate the lessening of deformation effects with depth below the surface. In reality, there is a continuous transition from heavy to light deformation with increasing depth below the abraded surface. Within the layer of primary deformation, the preexisting pores were filled by material being smeared by the abrasive action of the diamond particles. The result is a surface with very little apparent porosity.

After a short period of 3 μm lapping, a smooth polished surface is apparent, again with little apparent porosity (Fig. 1b). However, as illustrated in Fig. 2(b), the reason for this low apparent porosity is that the period of 3 μm lapping was long enough to provide a smooth polished surface but not long enough to remove the layer of primary deformation. The result is a significant underestimation of coating porosity.

For the purpose of simplification, the amount of deformation introduced by the 3 μm lapping is considered negligible. Given a longer period of 3 μm lapping, the primary deforma-

tion is removed, as illustrated in Fig. 2(c), and apparent porosity increases (Fig. 1c). However, it is not until the remainder of the secondary deformation is removed with longer periods of 3 μm lapping, as illustrated in Fig. 2(d), that a valid estimation of true porosity can be made. In the present work, no further increase in apparent porosity was evident after approximately 15 min of 3 μm lapping under the specified conditions, suggesting that smearing deformation was removed and true porosity revealed.

These results were repeated numerous times for these coatings, and similar results were obtained for a range of WC-12%Co coating structures and qualities from quite porous (~20%) coatings produced from agglomerated and sintered powders to much denser (~5% porosity) coatings produced from cast and crushed powder feed material.

4. Conclusions

The present results show the importance of ensuring adequate polishing time for the true representation of porosity in carbide composite coatings. This result also applies to the relatively more ductile metallic coatings and more brittle ceramic coatings deposited by various thermal spray methods because the potential for smearing over (or filling in of) pores remains. Insufficient polishing can result in considerable underestimation of porosity, thus providing incorrect representation of coating structure.

This should be a major consideration for the thermal spray industry, from the commercial coater wishing to ensure the quality of a product to the coating researcher who requires reliable and reproducible metallographic methods for controlled experimentation.

Acknowledgments

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References

1. H.E. Exner, Physical and Chemical Nature of Cemented Carbides, *Int. Met. Rev.*, No. 4, 1979, p 149
2. S.D. Washko and G. Aggen, Wrought Stainless Steels, *ASM Metals Handbook*, Vol 1, 10th ed., ASM International, 1990, p 841
3. S.D. Glancy, Preserving the Microstructure of Thermal Spray Coatings, *Adv. Mater. Process.*, Vol 7, 1995, p 37
4. M.F. Smith, D.T. McGuffin, J.A. Henfling, and W.J. Lenling, A Comparison of Techniques for the Metallographic Preparation of Thermal Sprayed Samples, *J. Therm. Spray Technol.*, Vol 2 (No. 3), Sept. 1993, p 287
5. L.E. Samuels, *Metallographic Polishing by Mechanical Methods*, 3rd ed., American Society for Metals, 1982, p 23-36
6. S.D. Glancy, Universal Metallographic Procedure for Thermal Spray Coatings, *Struers J. Materialography*, Vol 29, 1996, p 12
7. S.D. Glancy, How Metallographic Preparation Affects the Microstructure of WC/Co Thermal Spray Coatings, *Thermal Spray: Practical Solutions for Engineering Problems*, C.C. Berndt, Ed., ASM International, 1994, p 771-777