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The Effects of Thermosetting and Castable Encapsulation Methods on the Metallographic Preparation of Ceramic Thermally Sprayed Coatings—A Technical Note

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Accurate microstructural analysis of thermally sprayed coatings is possible only if specimen preparation produces a surface that clearly reveals the true microstructure. Comparison of similar specimens prepared by different laboratories, or different personnel in the same laboratory, often shows significant differences or variations in the microstructures, producing conflicting information that lowers the confidence level of microstructural analysis. A study was conducted on an alumina coating and an 8 wt% yttria-stabilized zirconia coating, both sprayed on metal substrates, to evaluate the effects of two different encapsulating procedures.

1. Introduction

VARIOUS methods are used to evaluate the characteristics of thermally sprayed coatings, but metallographic examination provides the most information about the many different coating characteristics (Ref 1). If improper methods are used during any one of the preparation stages, the end result will produce questionable information about the coating integrity (Ref 2). Basically, thermally sprayed coatings can be categorized as friable or nonfriable. Nonfriable coatings are not susceptible to excessive damage during each stage of metallographic preparation. Friable coatings, such as the two ceramic coatings in this report, are brittle and tend to readily crumble or fracture during standard metallographic preparation. The potential to incorrectly prepare ceramic coatings is a possibility, thus causing the rejection of coatings that actually meet the required specifications. The results reported in this paper will enable laboratory personnel to select the proper sectioning, mounting, and metallographic preparation techniques that will consistently produce reliable results.

Key Words: ceramic coatings, microstructure, polishing procedures, preparation artifacts, specimen preparation

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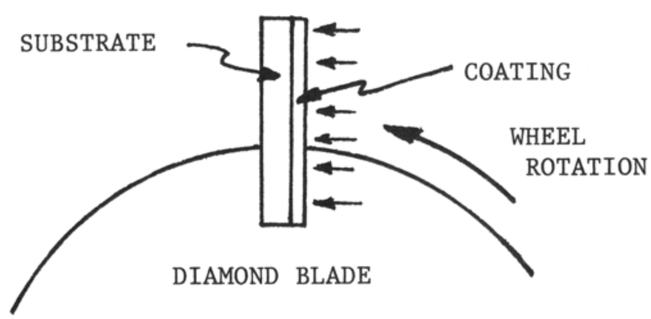


Fig. 1 Recommended wheel rotation and sample position for sectioning

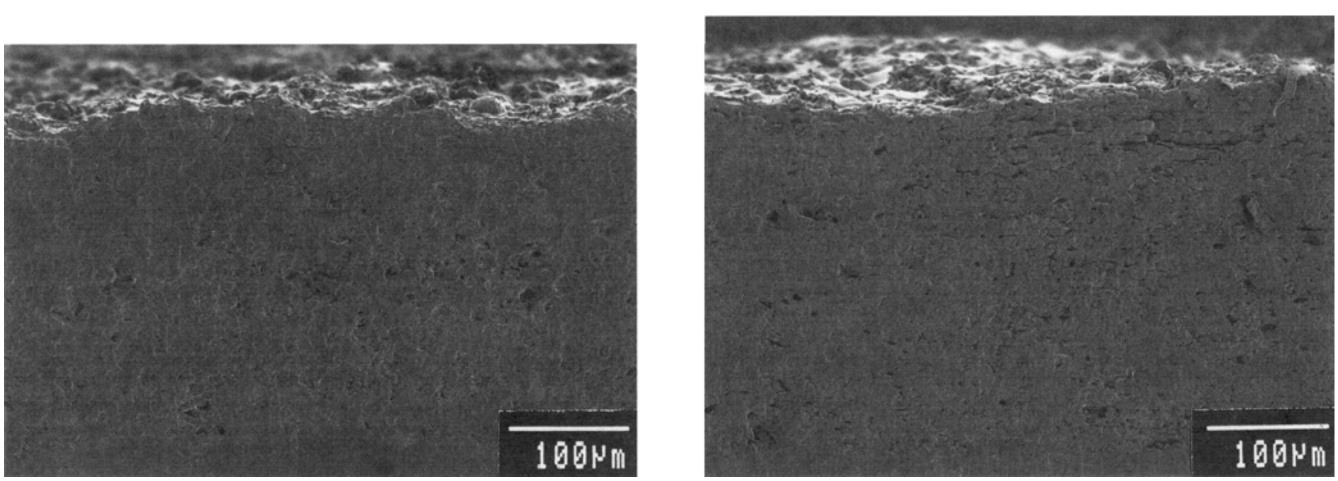
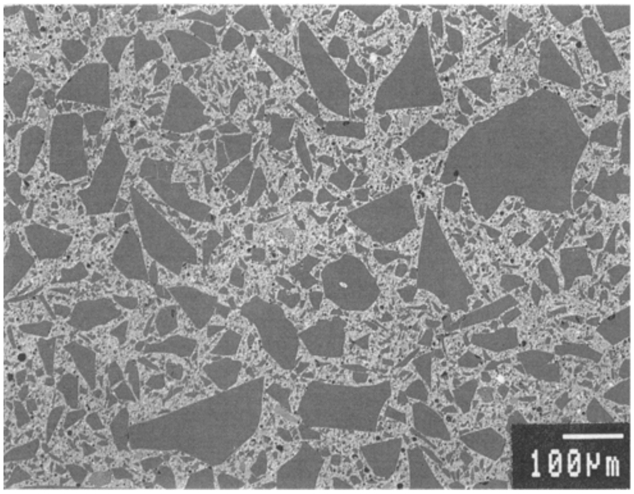


Fig. 2 Sectioned surface of the alumina coating (as-cut). (a) Fully dense. (b) Less dense



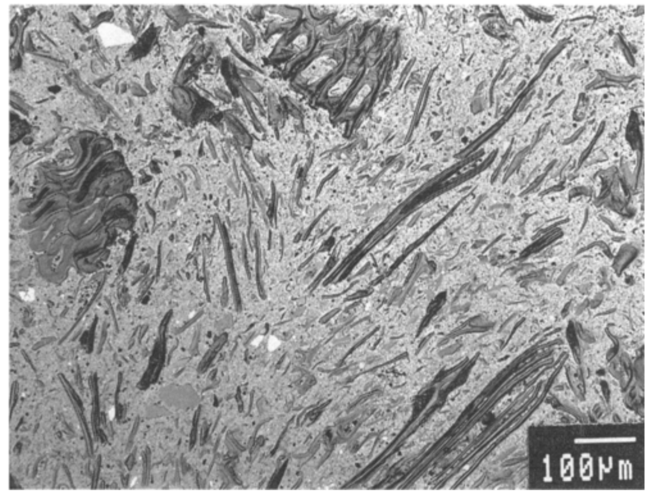
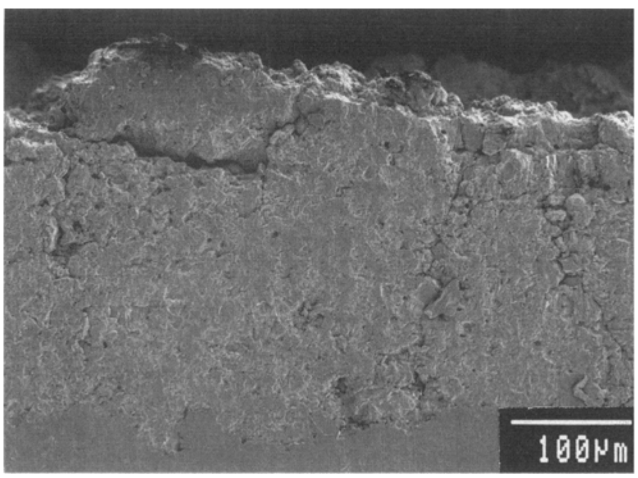


Fig. 3 Sectioned surface of the 8% YSZ coating (as-cut). (a) Fully dense. (b) Less dense



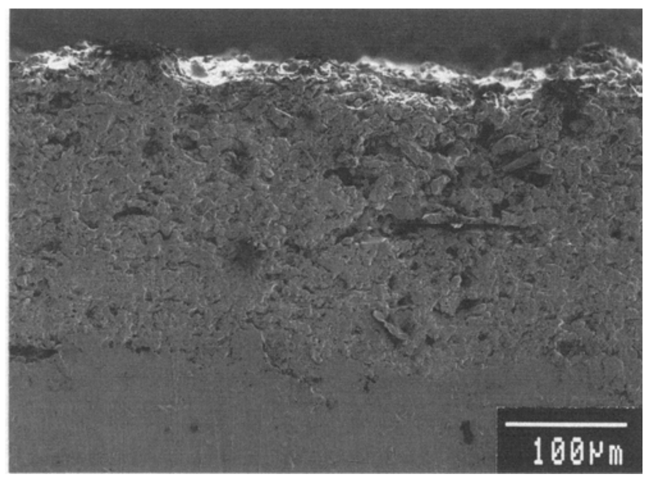


Fig. 4 Thermosetting mounting powders. (a) Silica-filled. (b) Mineral-filled

2. Sectioning

Each sample was sectioned perpendicular to its axis using a precision diamond saw with a metal-bonded diamond wafering blade. The specimen was clamped in the vise and positioned so that the diamond blade entered the coating and exited the substrate. This practice places the coating in compression and substantially reduces coating damage. Figure 1 is a diagram showing the recommended wheel rotation and sample position. Figures 2 and 3 are scanning electron micrographs showing the characteristics of the sectioned surface of the alumina coating and the 8 wt% yttria-stabilized zirconia (8% YSZ) coating. Figures 2(a) and 3(a) are fully dense alumina and 8% YSZ coatings, respectively, and show low levels of sectioning damage. Less dense alumina and 8% YSZ coatings show more sectioning damage, as shown in Fig. 2(b) and 3(b) in the form of pullout and cracks. In both cases the sectioning damage can be removed during the initial grinding step that is part of proper sectioning technique. The cutting parameters used for these coatings are:

- *Wheel type*: Diamond
- *Wheel speed*: 2500 rpm
- *Applied load*: 400 g
- *Lubricant*: Water soluble

3. Mounting

The purpose of mounting is to provide a safe, convenient means of holding the coating samples during preparation. It also protects the coating from destructive attack of the abrasive materials during the grinding and polishing steps. The materials used for metallographic encapsulation fall into two main categories, thermosetting or castable epoxy. The selection of the proper mounting medium is critical when dealing with ceramic coatings. In this study the alumina and 8% YSZ coatings were mounted in both thermosetting and castable mounting materials to determine how each method affects the coating.

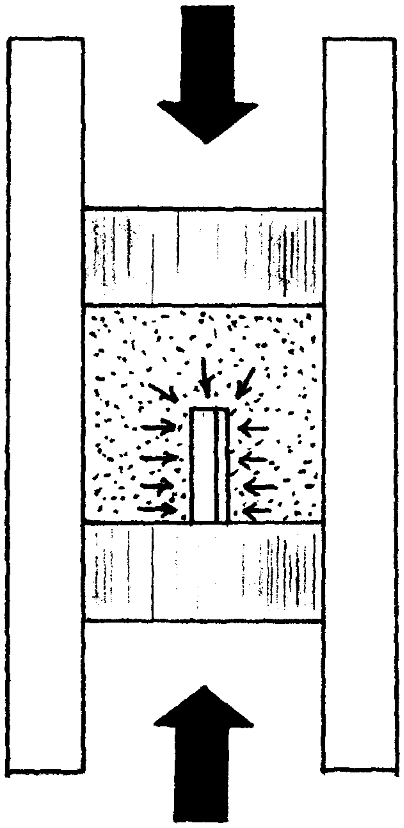


Fig. 5 Double-ram method used to apply consolidation pressure for thermosetting encapsulation

3.1 *Thermosetting Materials*

Silica-filled (Fig. 4a) and mineralfilled (Fig. 4b) mounting powders were selected for thermosetting encapsulation. The silica-filled powder required a consolidation pressure of 21 MPa (3000 psi) at 150 °C and the mineral-filled powder required a pressure of 29 MPa (4200 psi) at 150 °C. Both powders were cooled under pressure to 60 °C after the heating cycle was completed. The total cycle time was 7 min for the silica-filled powder and 10 min for the mineral-filled epoxy. Figure 5 shows that a double-ram method was used to apply the pressure and that the pressure distribution on the thermally sprayed sample was approximately isostatic during the pressure cycle.

3.2 *Castable Epoxy*

Clear castable epoxy with a cure time of 45 min was selected for the nonpressure encapsulation. Coating samples were encapsulated under either ambient pressure or under a vacuum of 30 in. Hg. Four samples were placed in a plastic sample cup and coated with the mixed epoxy. A vacuum of 30 in. Hg was held for 5 min to allow maximum penetration of the epoxy into the open pores in the coating. After the vacuum cycle was completed, each sample cup was filled to the 3/4 level and allowed to cure. All of the mounted coating samples were placed in specimen holders and metallographically prepared automatically.

4. Metallographic Preparation

Although the preparation parameters were the same for all of the samples, the silica-filled, mineral-filled, and castable epoxy samples were prepared separately. In order to provide a consistent, repeatable preparation procedure for the two coatings in various mounting media, a procedure was used that will be incorporated in a new ASTM thermal spray metallography standard presently being developed (Table 1). This procedure has proven to be very reliable and produces accurate coating characteristics. Micrographs were taken documenting the results of the preparation procedure and the effects of the three types of encapsulation.

5. Observations

Sectioning the coatings on a precision diamond saw using a metal-bonded diamond wafering blade substantially reduced

Table 1 Preparation procedure

Surface	Lubricant	Abrasive type/ size/carrier	Time, s	Force per sample, lb	Speed setting, rpm	Relative rotations
Rough grinding						
Paper	Water	180-grit SiC	30	5	240	Comp.
Fine grinding						
Paper	Water	240-grit SiC	25	5	240	Comp.
Paper	Water	320-grit SiC	25	5	240	Comp.
Paper	Water	400-grit SiC	25	5	240	Comp.
Paper	Water	600-grit SiC	25	5	240	Comp.
Paper	Water	800-grit SiC	25	5	240	Comp.
Rough polishing						
Nonwoven textile	Lapping oil	3 μm diamond	120	6	120	Comp.
Final polishing						
Synthetic suede		0.05 μm Al_2O_3, 0.04 to 0.06 μm SiO_2	120	7	120	Contra

(a) Comp., rotations in the same direction; Contra, rotations in the opposite direction

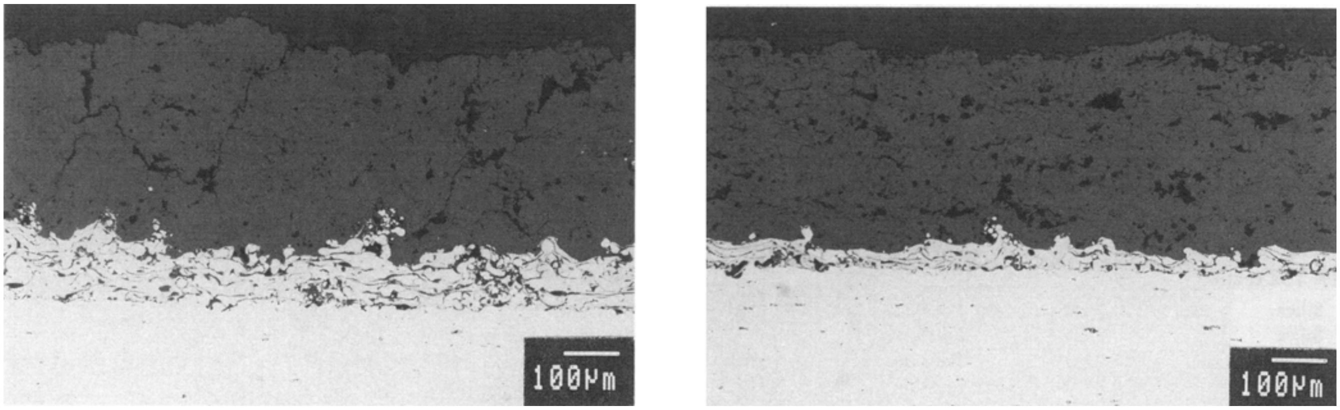


Fig. 6 Alumina coating sample mounted in silica-filled powder. (a) Fully dense. (b) Less dense

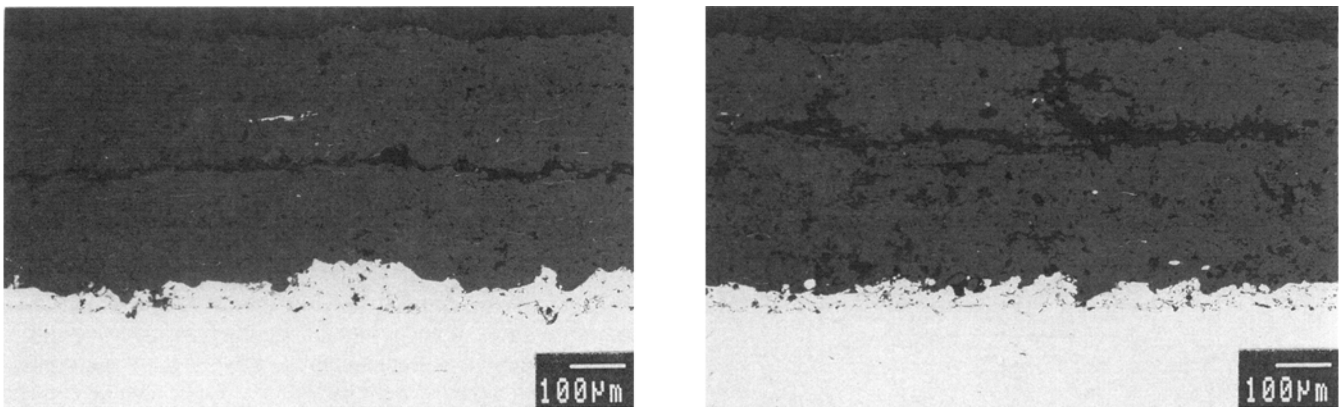


Fig. 7 8% YSZ coating sample mounted in silica-filled powder. (a) Fully dense. (b) Less dense

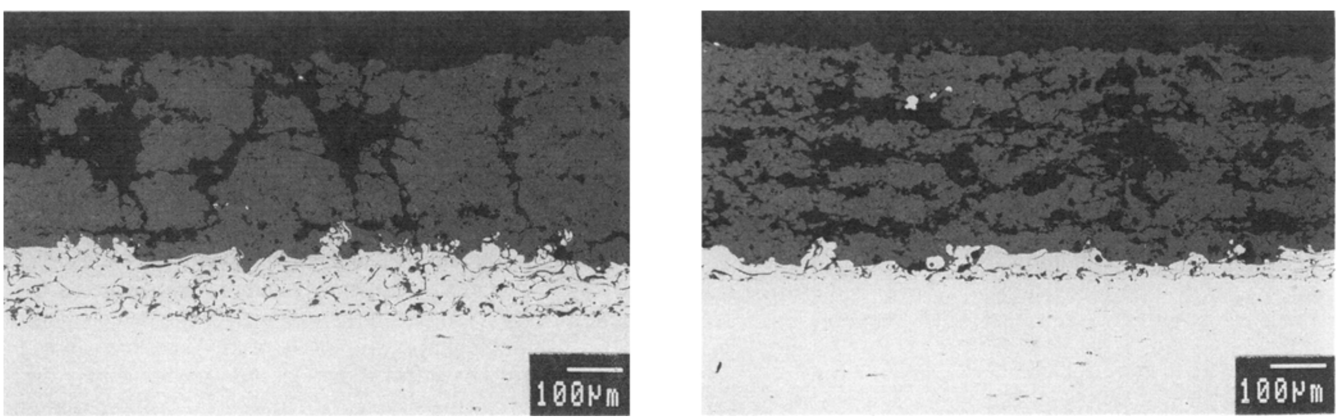


Fig. 8 Alumina coating sample mounted in mineral-filled powder. (a) Fully dense. (b) Less dense

the coating damage when the coating was placed in compression. The 8% YSZ coating exhibited more damage than the alumina coating, regardless of the coating density.

5.1 *Thermosetting Process*

Silica-filled powder produced very good edge retention because the silica filler increased the abrasion resistance of the mount. Although the mounting pressure was lower than that used with the mineral-filled epoxy, the damage to both the alumina and 8% YSZ coatings was greater. This excessive coating damage can possibly be attributed to the presence of the hard silica particles. Figures 6 and 7 show the level of damage caused by this mounting material. These results are not acceptable and produce incorrect coating characteristics.

Mineral-filled powder did not produce the edge retention of the silica-filled powder, but neither did it create the same level of coating damage. The fully dense alumina coating showed no

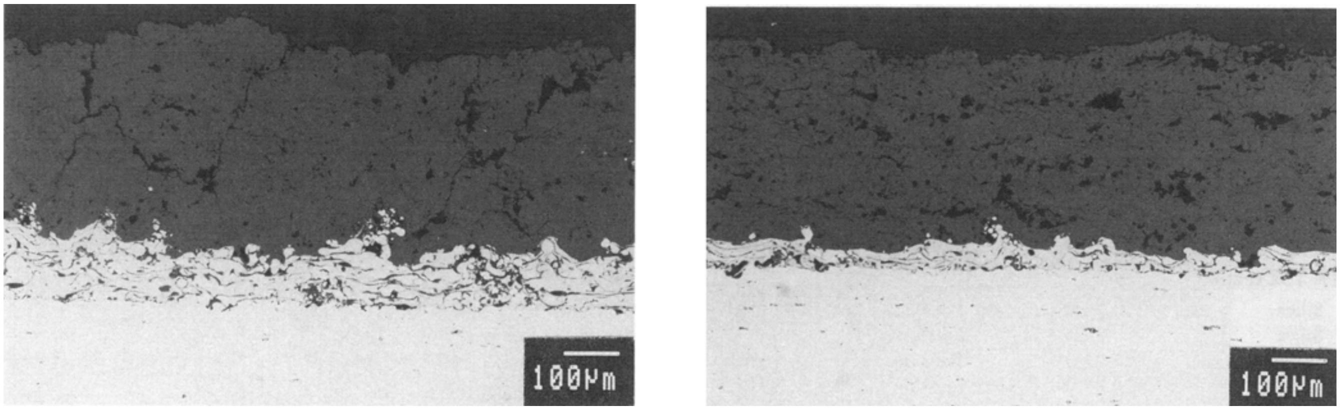


Fig. 9 8% YSZ coating sample mounted in mineral-filled powder. (a) Fully dense. (b) Less dense

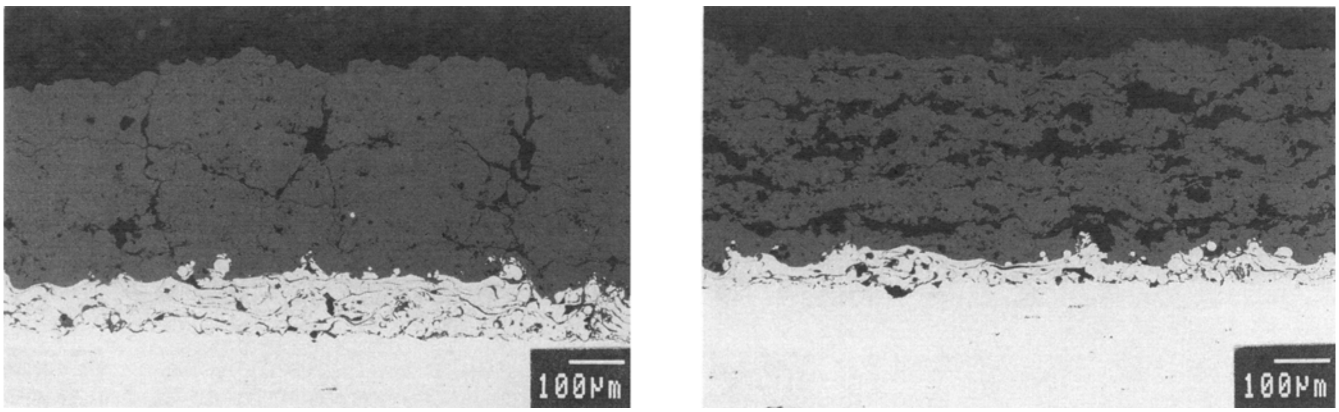


Fig. 10 Alumina coating sample mounted in castable epoxy under ambient pressure. (a) Fully dense. (b) Less dense

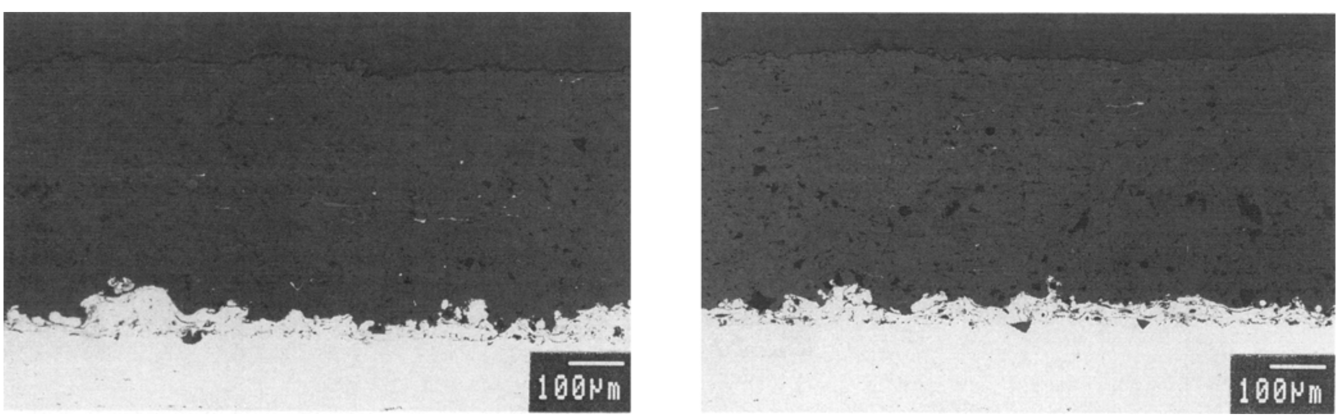


Fig. 11 8% YSZ coating sample mounted in castable epoxy under ambient pressure. (a) Fully dense. (b) Less dense

damage, but the fully dense 8% YSZ and the less dense alumina and 8% YSZ coatings did show damage. This damage can be attributed to the high pressure (29 MPa, or 4200 psi) applied during the mounting procedure. These results are not acceptable and will also produce incorrect coating characteristics. Figures 8 and 9 show the level of damage caused by this mounting material.

5.2 *Castable Epoxy*

Specimens prepared under ambient conditions produced acceptable edge retention on both coatings. Due to the absence of high pressure during encapsulation, the coating damage was substantially less than in the thermosetting process. The absence

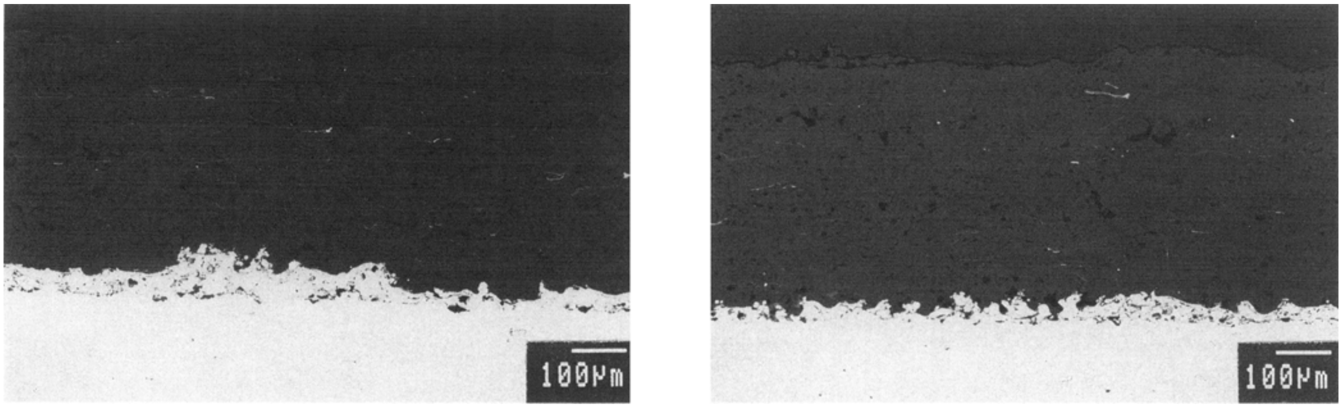


Fig. 12 Alumina coating sample mounted in castable epoxy under vacuum

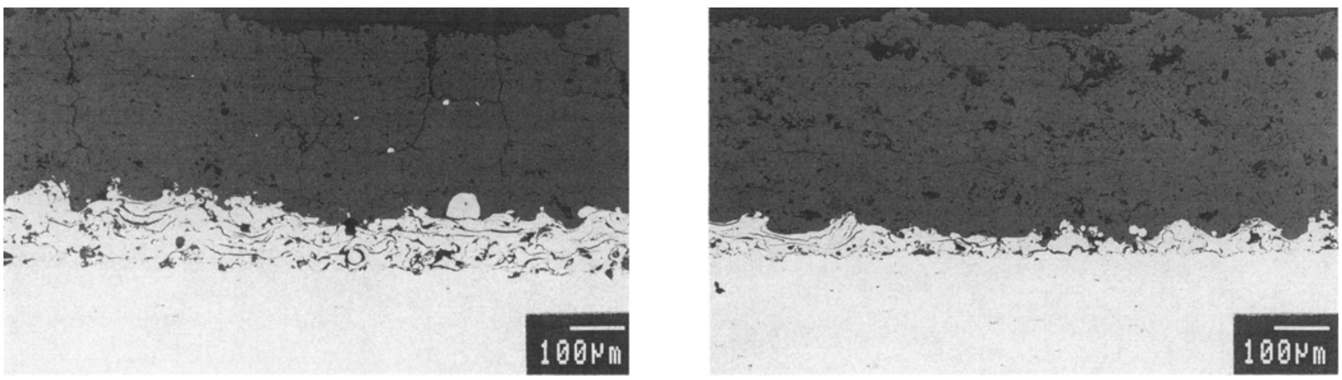


Fig. 13 8% YSZ coating sample mounted in castable epoxy under vacuum pressure. (a) Fully dense. (b) Less dense

Table 2 Summary of test results

Mounting media	Coating density	Environment	Damage
Alumina coating			
Silica	FD	Pressure	Y
Silica	LD	Pressure	Y^+
Mineral	FD	Pressure	N
Mineral	LD	Pressure	Y^+
Castable epoxy	FD	Ambient	Y^-
Castable epoxy	LD	Ambient	Y^-
Castable epoxy	FD	Vacuum	(N)
Castable epoxy	LD	Vacuum	(N)
8% YSZ coating			
Silica	FD	Pressure	Y
Silica	LD	Pressure	Y^+
Mineral	FD	Pressure	Y^-
Mineral	LD	Pressure	Y^+
Castable epoxy	FD	Ambient	Y^-
Castable epoxy	LD	Ambient	Y^-
Castable epoxy	FD	Vacuum	(N)
Castable epoxy	LD	Vacuum	(N)

FD, fully dense; LD, less dense; +, major damage; –, minor damage; ( ), acceptable results; N, no; Y, yes

of vacuum infiltration caused an increase in pullout and cracks. Figures 10 and 11 show the absence of coating encapsulation damage and reveal the presence of pullout due to the lack of epoxy infiltration.

Vacuum preparation also produced acceptable edge retention on both coatings. The absence of high pressure and the use of vacuum infiltration procedures during encapsulation produced results that substantially reduced the damage to the coating during the metallographic preparation procedures and revealed the true characteristics of the coating. Figures 12 and 13 show coatings that are flat, scratch-free, and free of encapsulation damage and pullout.

Table 2 is a summary of the results obtained from the tests performed on the two coatings. It can be used as a reference when a reliable metallographic preparation method is required for an alumina or 8% YSZ coating.

6. Conclusion

- The use of high pressure (21 to 29 MPa) during the encapsulation of alumina and 8% YSZ coatings causes damage that cannot be eliminated during the subsequent grinding and polishing steps.
- Silica-filled powder creates greater damage to the coatings than mineral-filled powder.
- The higher the coating density, the more resistant the coating is to damage during high-pressure encapsulation; however, silica-filled powder caused damage at all density levels.

- The absence of vacuum infiltration of castable epoxy allows for pullout and enlarging of porosity during metallographic preparation.
- Vacuum infiltration of castable epoxy enables the epoxy to infiltrate cracks and open pores and maintain the structural integrity of the coating during metallographic preparation.

Acknowledgments

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