The Preparation and Evaluation of Thermal Spray Coatings: Mounting

Douglas G. Puerta
Director of Metallurgical Engineering
IMR Test Labs, Lansing, New York

Abstract

This article is the second in a series of articles dealing with the metallographic preparation and evaluation of thermal spray coatings. Previously, critical parameters and best practices for the sectioning of coated components have been discussed. In this article, different mounting methods and their applicability to various coating families will be explored.

Hot & Cold Mounting

Most modern metallographic laboratories are equipped with the ability to create both hot mounts (compression mounts) and cold mounts (typically using a two part epoxy resin). Unfortunately, in many cases a mounting system is chosen which leads to either difficulties in evaluation or possibly damage to the coating. Often times, these decisions are motivated by the need to support high-paced production environments.

Hot mounting involves heating epoxies and/or phenolic powders above 300°F while maintaining a constant pressure up to 4,500 psi. Cycle times are relatively short (10-15 minutes), which makes this method very attractive for labs subject to short turn-around requirements. However, due to the heat, pressure, and flow limitations associated with this process, hot mounting is a poor choice for most coating families. Figure 1 shows a nickel graphite sample which was hot mounted and metallographically prepared. The minimal amount of epoxy penetration into the porous abradable coating can be seen. This represents the main drawback to hot mounting for porous thermal spray coatings. When compared to cold mounting with a low-viscosity epoxy, hot mounting media does not penetrate porous coatings effectively. During grinding and polishing, areas of the coating not impregnated with epoxy are more susceptible to mechanical damage.

Looking at the expanded views of Figure 1, two very different coating structures are evident. The top of the coating has been at least partially impregnated by the hot mounting media. The resulting coating structure is representative of a nickel graphite abradable coating. In contrast, the lower region of the coating has not been impregnated. Mechanical damage during grinding and polishing has resulted in the graphite phase being “pulled-out”, leaving behind only porosity and nickel. While an accurate coating evaluation can be performed on the top portion of the coating, this is not the case for the bottom portion.

In general, hot mounting has only been successfully used for a limited number of coating families. Hardcoat coatings (HVOF and thermal spray) are one group where hot mounting is widely used. In general, hardcoats are fairly dense and relatively hard (generally > 800 HV). This combination makes the coating less susceptible to mechanical damage, even with the lack of epoxy impregnation. Another coating family where hot mounting is frequently used is metallic coatings. Despite exhibiting moderate porosity levels, coatings such as CuNiIn also appear to remain sufficiently intact during metallography.

Remaining common coating families such as ceramic, dimensional restoration, and thermal barrier are poor candidates for hot mounting. Coatings from these families rely on the support of epoxy impregnation during grinding and polishing. In addition, the heat and pressure associated with hot mounting can induce damage into delicate coatings. Therefore, the negatives of hot mounting outweigh the positive benefits such as fast cycle time and excellent edge retention.

Cold mounting is the general term used to describe multi-component systems such as epoxies, acrylics, and polyesters which are mixed together and cast at (or near) room temperature. There are a large number of choices within this group, as evidenced by the catalogs of most metallographic supply companies. Despite the selection, very few of the available products in this area are generally recommended for mounting thermal spray coatings.

The important considerations for cold mount material selection are cure time, hardness, viscosity (the ability of the material to fill inherent porosity & voids), and shrinkage. Based on these requirements, several candidates can be quickly eliminated. Despite their short cure time (< 30 minutes), acrylics and polyesters generally exhibit relatively high viscosity, high shrinkage, and low hardness when compared to epoxies. Shrinkage and low hardness lead to poor edge retention and subsequently hinder coating evaluations. High viscosity limits coating impregnation, even with the assistance of a vacuum chamber.

Epoxies are the most widely used system for mounting thermal spray coatings. With few exceptions, epoxies maintain a very consistent relationship between cure time, viscosity, hardness, and shrinkage. This relationship highlights the difficulties in balancing turnaround requirements with good metallographic practices. While a number of options exist for "fast cure" epoxies which are purported to cure in less than 1 hour, this is typically accomplished at the expense of reduced hardness, increased shrinkage, and elevated viscosity. Elevated viscosity makes impregnation difficult, and can lead to difficulty in distinguishing porosity from mechanical damage. At the other end of the epoxy spectrum, slow cure epoxies (12-24 hours) offer minimal shrinkage, low viscosity, and high hardness. However, the long cure time makes this option unattractive for production facilities.

Vacuum impregnation is accomplished by placing the sample, covered by liquid epoxy, in a vacuum approaching 1 atmosphere (29.9 inches of mercury or ~760 torr). As air is removed from the sample, the epoxy impregnates cracks and pores within the coating. For porous samples such as nickel-graphite, epoxy can generally penetrate to a depth of at least 0.1". However, the ease of penetration and the depth to which the epoxy can penetrate is a function of its viscosity. Viscous, fast-cure epoxies (or hot mount epoxy) will not effectively impregnate even porous samples. In contrast, slow cure epoxy can readily impregnate coatings, even those with low porosity levels. It is worth mentioning that when placing a sample into a vacuum chamber, it is best to only fill the mounting cup half way with epoxy. Filling the cup all the way will result in epoxy spilling over the edge as a vacuum is pulled and the epoxy bubbles. After pulling a vacuum, the cup can be topped-off with epoxy. It is not necessary to place the mount back into a vacuum at this point as the plane of view should be well below the unimpregnated region.

Figure 2 shows a TBC coating, mounted using low-viscosity epoxy *without* vacuum impregnation. From this image, it is observed that despite not using a vacuum system, some epoxy penetration into the sample still occurs. However, a difference in the apparent porosity level exists between the top of the coating (impregnated) and bottom of the coating (not impregnated). It is difficult to determine if the porosity levels throughout the coating are inherent or if they are exaggerated from mechanical damage (i.e., artifact from the preparation method).

A new family of epoxies has come onto the market within the last several years which offer very low viscosity, excellent hardness, minimal shrinkage, and short cure time (< 2 hours @ 70-80°C). These epoxies have quickly become very popular across the thermal spray industry due to their great combination of properties. Even at room temperature, heat curing systems such as these will vacuum impregnate with great success.

All epoxies have the advantage of being at least nearly transparent. By adding fluorescent or colored dyes into the epoxy, an accurate rating of the coating can be performed and induced features can be distinguished from inherent features. Figure 3 shows brightfield and UV illuminated views of the same coating. From these images, it can be concluded that all porosity is inherent (has been filled by epoxy) and, therefore, the grinding and polishing steps did not induce mechanical damage. This method also reinforces the importance of fully impregnating the sample in question with epoxy.

As a final note before leaving the topic of mounting, a clean and dry sample prior to mounting is extremely important. As-sectioned samples should be cleaned with water, rinsed with alcohol (preferably ethanol), and dried using an oven or other heat source. Due to the delicate nature of thermal spray coatings, ultrasonic cleaners should not be used. Dirt or moisture left within a coating can result in outgassing from the sample during curing or failure to cure.

Conclusions

Given the choice between hot and cold mounting and the large number of products available for each method, an inexperienced operator may have difficulty selecting the best process for mounting thermal spray samples. Time permitting, slow cure, two-part epoxy resins have been shown to provide excellent hardness, impregnation, and minimal shrinkage. While hot mounting is fast, it has only been demonstrated to be effective for a few coating families. Any coating can be cold mounted following proper procedures without risk to the integrity of the coating.

Table 1 summarizes several coating families along with recommended mounting practices for each. Please note that while two families list hot mounting as a viable option, this process should be qualified within the operator's lab by hot and cold mounting samples and then comparing results. If identical polishing procedures are used and the microstructure of the two samples is equivalent, it can be concluded that the mounting method did not have a detrimental effect on the resultant structure. Cracking or other mechanical damage within the hot mounted specimen, but not the cold mounted specimen, indicates that hot mounting should not be used for that particular coating.

Coating Family	Common Coatings	Hot Mount	Cold Mount Notes
Abradables	Ni-Graphite, XPT-268	No	Vacuum impregnation critical to preserve structure.
Anti-Fretting	CuNiIn, Al-Bronze	Yes	Moderate impregnation is adequate to preserve structure.
Ceramics	Alumina, Titania	No	Vacuum impregnation critical to preserve structure.
Dimensional / Bondcoats	MCrAlY, NiAl	No	Vacuum impregnation critical to preserve structure.
Hardcoats	WCCo, CrC-NiCr	Yes	Moderate impregnation is adequate to preserve structure.
Thermal Barrier	YSZ	No	Vacuum impregnation critical to preserve structure.

Table 1. Recommended mounting systems for common coating families.

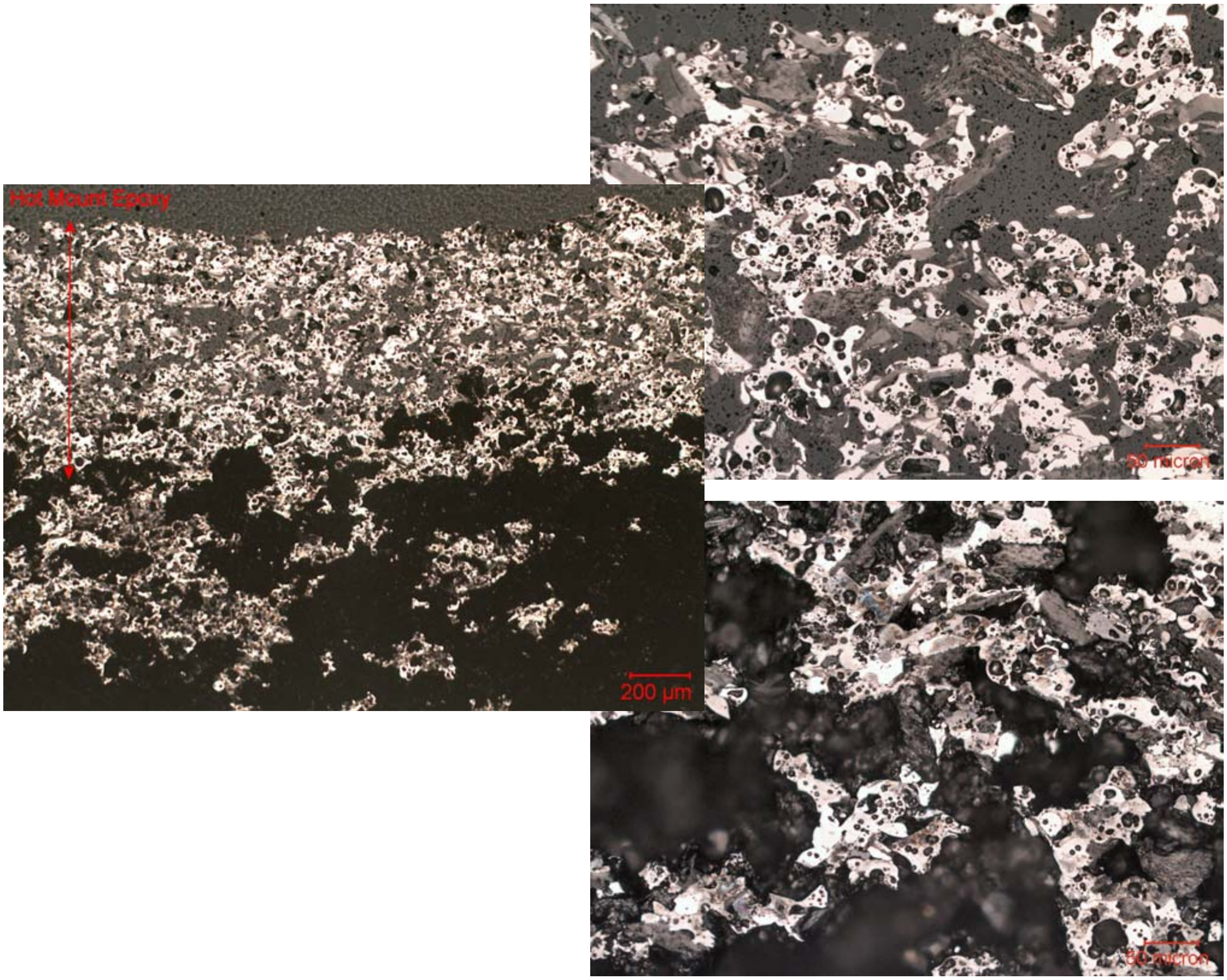

Figure 1: Nickel-graphite coating mounted in hot mount epoxy. The depth of epoxy penetration (red arrow) into the coating results in dramatically different structures between the impregnated region (top) and non-impregnated region (bottom).

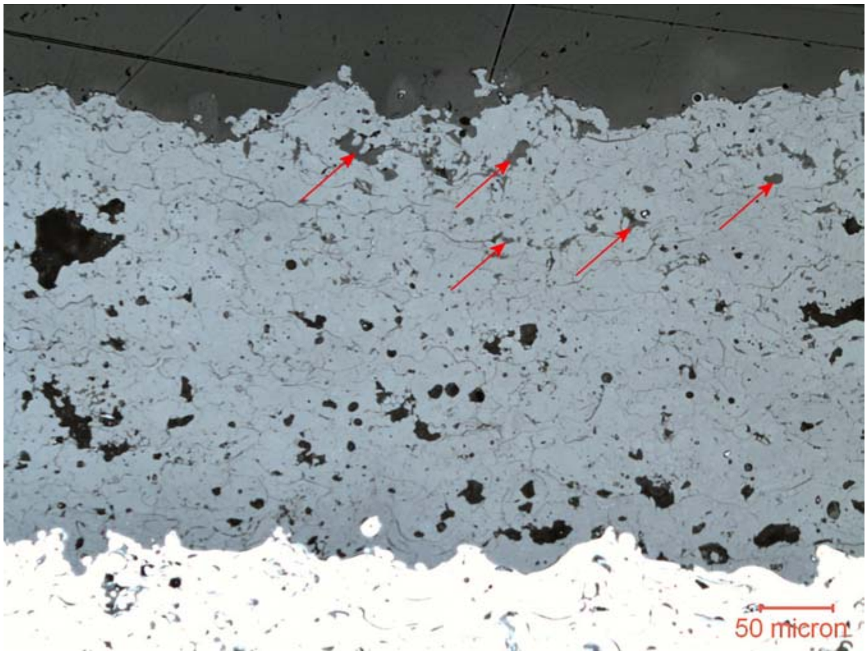

Figure 2: Thermal barrier coating (TBC) sample mounted in slow-cure epoxy and metallographically prepared. The lack of epoxy penetration into the coating is due to a lack of vacuum impregnation used during mounting. The arrows indicate locations where epoxy is present. Please note the elevated porosity concentration below the epoxy-impregnated region, as evidenced by the empty (dark colored) pores.

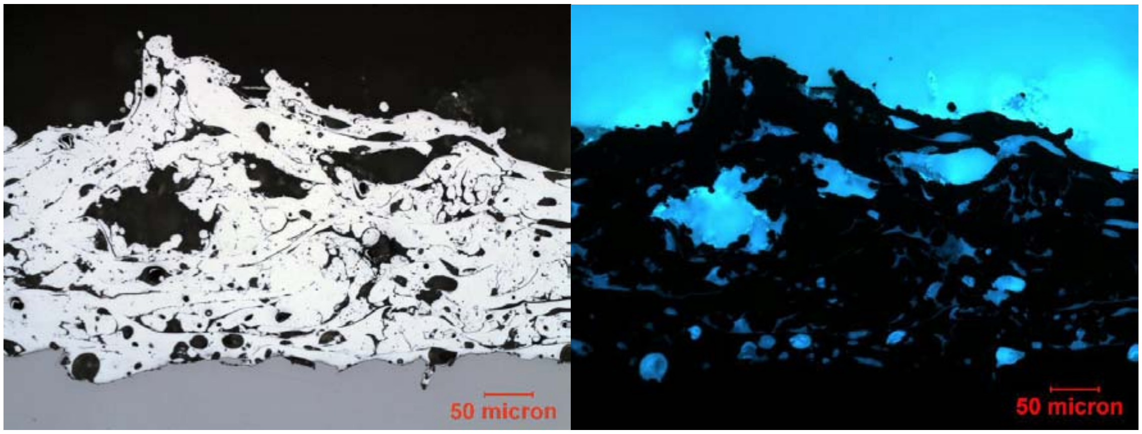

Figure 3: Brightfield (left) and UV illuminated (right) views of an aluminum thermal spray coating. Low-viscosity cold mount epoxy was used which contained a fluorescent dye. Epoxy penetration into all of the visible voids indicates that the porosity present within this coating is inherent to the coating and not induced during grinding and polishing.