

# The metallographic characterization of thermal spray coating microstructures

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Thermal spray coatings from several different coating families have been metallographically prepared using traditional and modern metallographic techniques. The different recipes used were intended to demonstrate the effect of abrasive on coating appearance. Traditional metallographic recipes, which rely heavily on the use of silicon carbide (SiC) abrasive papers, were found to produce a notably different appearance than those prepared using modern recipes. Modern recipes, which incorporate extended diamond grinding and polishing steps, were found to produce what appears to be a more representative coating structure. Other variables, including mounting media and use of vacuum impregnation, were also found to influence coating appearance.

## 1 Introduction

Within the past twenty years, there have been a number of significant advances in the area of metallographic preparation techniques. Traditional preparation methods have been replaced by new methods which better utilize diamond as the abrasive for both grinding and polishing (see Table 1). As a result, microstructural features which were previously lost or masked during preparation are now readily visible [1].

**Table 1.** Traditional (top) and modern (bottom) metallographic preparation recipes.

Paper/Cloth	Abrasive	Time
SiC Paper	120-1200 grit	60 seconds/step
Low-nap Cloth	1µm diamond	2-4 minutes

Paper/Cloth	Abrasive	Time
SiC Paper	120-240 grit	Until Planar
Diamond Grinding Disc	9µm diamond	6 minutes
Woven Cloth	3µm diamond	4-5 minutes
Low-nap Cloth	1µm diamond	2-4 minutes
or		
Chem Cloth	Colloidal Silica	45 seconds

Unfortunately, the slow adoption of these new techniques has led to confusion and mis-interpretation within the aerospace industry. Due to the sensitive nature of thermal spray coatings to metallographic preparation, different operators using different metallographic procedures can end up with varying results. As a result, decisions affecting spray booth parameters are frequently made based on an inaccurate evaluation. This problem is compounded by the fact that the aerospace industry has long used photostandards for plasma spray coating analysis. Unfortunately, in many cases these photostandards were developed by engine manufacturers prior to the development of modern metallographic techniques.

In this presentation, plasma spray coatings including WCCo, NiAl, Molybdenum, and TBC (yttria-stabilized zirconia) will be examined to demonstrate the effects of metallographic preparation on coating features. Vacuum impregnation of low-viscosity epoxies contain-

ing fluorescent dyes will be used to highlight features inherent to each coating [2]. Scanning electron microscopy (SEM) will allow for analysis of cryogenic fracture surfaces (see Figure 2) from which voids and other microstructural features can be qualitatively observed [3]. Particular attention will be paid to the bonding of individual splat particles and the formation of linear detachments (voids) or oxides at these interfaces [4].

Due to the relationship between microstructural analysis and spray booth operation, metallographers must ensure that the laboratory conveys accurate results to the spray booth operator. This accuracy is directly dependent on improvements in specimen preparation for metallographic examination. Modern metallographic methods and automated sample preparation play a major role in this area.

## 2 Coating Evaluation

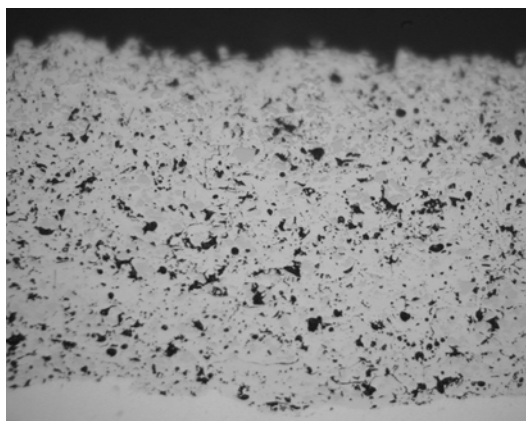
Coupons of the previously mentioned coatings were mounted by three different methods: hot mounted, cold mounting without vacuum impregnation, and cold mounting with vacuum impregnation. Each of the samples was then metallographically prepared using traditional and modern preparation recipes.

Figure 1 shows optical micrographs of a WCCo plasma spray coating. In this case, the different recipes produce significant variations in apparent porosity. This is attributed to the rapid breakdown of SiC papers, and subsequent smearing of the cobalt phase over inherent pores.

The speed at which SiC papers break down is a function of the coating hardness and mounting material (the overall hardness of the coupon and mount system). Hot mounted hardcoats will cause papers to break down relatively quickly, while cold mounted metallic coatings will break down papers relatively slowly. The amount of metal that is present in a mount will also lead to faster break down of SiC papers.

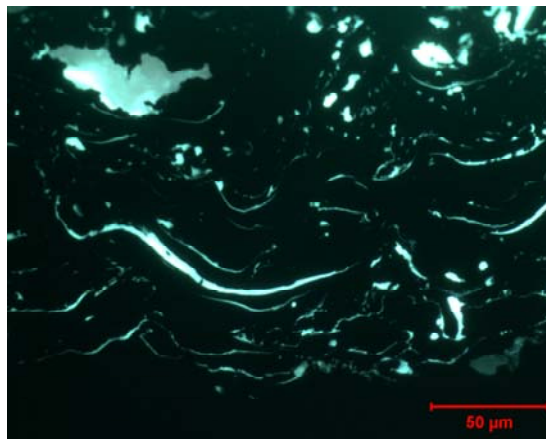
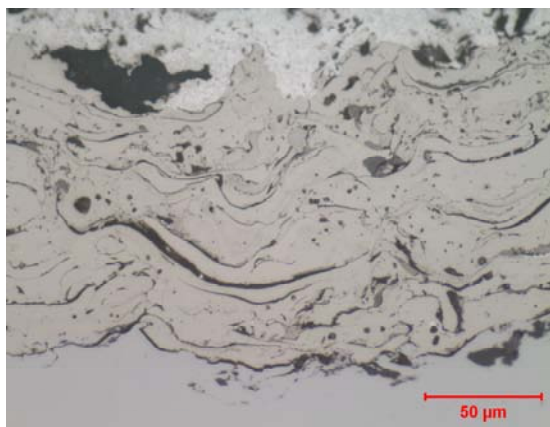
To verify penetration of the cold mounting epoxy, and also to identify inherent porosity, fluorescent dye can be added to the epoxy. Features induced during metallographic preparation will not fill with epoxy, and

consequently will not luminesce under a UV light-source.



**Fig.1** Plasma spray WCCo coating prepared using traditional methods (top) and modern methods (bottom). Variations in porosity result from the preparation method.

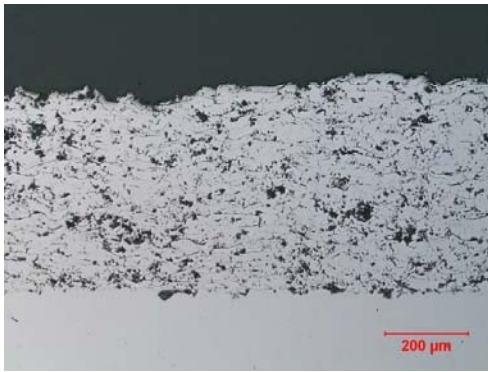
Figure 2 shows a NiAl plasma spray coating using both brightfield and UV lumination. Using a UV light-source, the porosity present in this coating is readily visible. This technique not only reveals inherent porosity within this coating, but also shows that the linear features commonly referred to as oxides are actually pores (or splat lines). Comparing the two images, it can be seen that porosity visible using brightfield lighting is also visible using UV lighting. This indicates that no porosity has been introduced into the coating.



**Fig. 2** Plasma spray NiAl coating impregnated with a cold mount epoxy containing fluorescent dye. The use of a fluorescent dye enables the metallographer to differentiate between inherent porosity and mechanical damage.

Epoxy penetration into a given coating was found to be very important. TBCs have shown to be insensitive to the metallographic preparation recipe used, as long as the coating has been impregnated with epoxy. Other coatings, such as molybdenum, while different in terms of coating ductility also rely upon epoxy impregnation. Figure 3 shows a molybdenum coating which has been hot mounted, cold mounted without vacuum impregnation, and cold mounted with vacuum impregnation. The lack of epoxy impregnation in the hot mount sample enables pores to be smeared shut, therefore producing a coating with artificially low porosity. When epoxy penetrates only part way into the coating, that part which has been impregnated shows the appropriate amount of porosity. The correct coating structure is achieved when the coating is fully impregnated with epoxy.





**Fig. 3** Plasma spray molybdenum coating hot mounted (top), cold mounted with no vacuum impregnation (middle), and cold mounted with full vacuum impregnation (bottom).

### 3 Summary

By varying the mounting method and metallographic recipe, notable changes in the appearance of a given thermal spray coating can be achieved. Understanding the influence of the numerous variables involved with the metallographic preparation of thermal spray coatings is critical to producing truly representative coating microstructures. Furthermore, an accurate evaluation of these coatings can only be achieved after the sample has been prepared correctly.

### 4 References

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