

**The Use of Metallographic Standards
in Calibration of the Polishing Process**

**J.P. Sauer
Metcut Research Inc., Cincinnati, Ohio**

Abstract

The metallographic process for evaluating thermally sprayed coatings is sometimes viewed as a variable process in the scope of coating evaluation. There is always a question as to whether the failure of a coating is polishing related or an actual change in the spray production process. The use of metallographic standards similar to hardness calibration can be implemented to provide assurance of a repeatable metallographic process. Development and use of the standards will be discussed and examples given of the standards principle.

THE USE OF STANDARDS IN THE TESTING process is a concept which has been a permanent part of evaluation procedures for a very long time. Employing standards provides confidence that the testing process will produce the same test sensitivity every time a part is evaluated. How many times have situations occurred in which a standard was not checked and parts were inspected with an invalid test? The absence or incorrect use of a penetrate/magnetic particle block would be an excellent example of this occurrence. Parts with very fine cracks could easily pass through inspection if the test machine or process is not calibrated correctly. An everyday example in many thermal spray shops is the calibration of hardness testers for R_{15N} hardness testing. A normal practice is to calibrate the machine at the beginning of every

day or shift, dependent upon the amount of usage. If the tester is not functioning properly, the machine must be checked and all parts processed since the last acceptable calibration must be considered for review. The spray process itself is not exempt from standards verification. Daily calibration of mass and gas flow rates, voltage/amperage meters, part spray temperatures, etc., are required to assure that the spray process will be in control and capable over time. Extending the use of standards for metallographic preparation is obvious, especially when metallography is used to verify process stability.

Why Do We Need Metallographic Preparation Standards?

Testing is a very critical aspect of the total thermal spray process. Confidence in the metallographic process is necessary to permit decisions to be made whether the variation in microstructural results is a result of testing or spraying. In microstructural evaluation, photo-standards or pictures are sometimes used to compare the current process results to a process standard. However, there is no assurance that the polishing process itself is the same as yesterday or the day before because the test specimen is from a different spray run every time. If the preparation process is out of control, good parts may be rejected because the process can introduce too many voids and not reveal the "true" structure. If

metallographic standards or reference samples are introduced that are polished in the same rack as other new or daily samples, confidence begins to grow in the assessment of variability within the process. If polishing is undertaken and the results of the standard are similar to the last time, the process can be considered repeatable and the data reflects a “true characterization” of the spray process. If results are not the same, review of the preparation process is in order to ascertain if there has been some change in the work steps to cause this difference. This concept is very practical and reflects the use of everyday practices employed in all other testing arenas.

How to Establish Metallographic Standards

Establishing metallographic standards must be well planned and executed. The standards must reflect the quality level that is part of production processing at the facility in question. The issue of “immune” vs. “sensitive” coatings must be addressed with respect to quality level. If standards are established using coatings that are not sensitive or are “immune” to polishing variability, there is no verification of the polishing process. The standard that is developed must be “sensitive” show a variation in polishing response if processed with different polishing techniques. This is the same concept used in NM standards for penetrant or magnetic particle, as mentioned earlier. In that case, there may be different sizes for the defect in each standard. The length or “tightness” of the crack may also be important in determining how “sensitive” the process will be to identifying whether a defect is present and, if so, whether it is acceptable or rejectable. If the NDT process is variable, the use of the standard must reflect this sensitivity or the standard is not useful for this purpose.

In the evaluation of thermal spray coatings, many characteristics are evaluated. Some examples are:

porosity/voids	phase (type) or distribution thereof
oxides	unmelted particles
interface	foreign
contamination	particles

The standard must reflect a typical distribution/frequency of the features that are encountered in normal daily processing. There essentially will be a need to establish these standards for most of the coatings sprayed in a particular shop. If the coating is sensitive to pullout during polishing, the standard should then be somewhat sensitive to induced or polishing-induced porosity. If a material is sensitive to oxide pullout during preparation, the standard should then also show some sensitivity when the mount is polished with different polishing parameters.

With all this in consideration, how should metallographic standards be established? The first order of business is to assess production over a range of coating quality that represents the expected variability of the production process. These materials must then be polished to establish whether the metallographic process can differentiate among various production conditions. When this differentiation is found, the quality level which reflects the target processing conditions should then be selected as our metallographic standard.

There must also be consideration of how the selected sample reflects the industry as a whole. Is the processing and standard typical of what other shops in the business produce and polish? This can be established by use of Round Robin (RR) principles. This has been successfully established in the Central Coatings Lab (CCL) Program as referenced in earlier NTSC proceedings⁽¹⁾⁽²⁾. Sample sprayed all at one time have been polished and a “typical” polishing response established. These samples are consistently used at the Metcut/CCL facilities to validate and verify procedures if questions arise or changes are made.

However, a round robin does not need to consist of 30+ companies as the CCL RR did to validate standards. This may be performed inter-company if many spray sites/labs are involved; or, verification with established testing houses also would be an option. Regardless of how verification or validation is established, it is strongly suggested that standards not be established without some collaboration with another laboratory source. If verification with another source is not performed, a laboratory may have a repeatable process that is consistently not in calibration with the rest of industry as a whole. It is important to consult peers and obtain feedback from many different sources.

How to Use The Standards That Are Established

It is very **logical** to use **metallographic** standards for validation of the process on a daily basis or to **ascertain** if the **preparation procedure** varies over a **period** of time. **However**, there are **other** very important **uses** that can be identified for **metallographic** standards. These are:

a) **Procedural Changes: Metallographic** suppliers sometimes contact laboratories about totally **new** systems for preparation of mounts. The **new process may be acceptable but it is difficult to** determine if the new process **will be** the same as the old process. How **can** the **results** be **verified** as **similar**? If standards existed, the known samples **can be run with the new system and compared to results from** established **procedures**. If a similarity exists, then it **would** be acceptable to **change** processes. If not, **further** investigation must be done to establish the best practice.

b) **Consumables: Consumables** are a very **important** part of the metallographic process. It is **very** critical to **define specific** consumables when dictating the **metallographic** process. **Unfortunately**, all **consumables** are not created **equal**. A no nap cloth **from** company X **will not necessarily** perform the same as a cloth **from** company Y. Also, **do not** assume that materials **from a specific vendor purchased over a period of** time will be consistent. **Consumables are manufactured** or formulated **differently** under the **same** headings of 6 micron diamond **suspension** or **colloidal** silica and sold as the same **product**. The materials will not provide the same result **on** sensitive materials **such as coatings**. It is, **unfortunately**, up to the metallographic **consumables** customer to find **out** if the new supplier of **consumables** has a product comparable to his present **brand**. **In** many cases the **change** is initiated by a **reduction in cost** of the **consumable** (**papers, polishing compounds, etc.**). This cost difference may be **reasonable** in some cases but in the case of diamond, the reduced price could **mean** a reduction in diamond particle concentration, **which effectively** reduces the polishing ability of the solution. This change could then require more solution to polish and ultimately cost more;

furthermore, decreased efficiency can result in longer polishing times. This situation **would** be an excellent application for **metallographic** standards.

There **can** be **many** other applications for **metallographic** standards in the laboratory such as comparison of old and improved spray **parameters**. It is a very **useful** concept that must be given serious **consideration** in the evaluation of thermal spray coatings by **metallographic** polishing and **evaluation**.

Summary

The concept of metallographic standards in the **preparation** of metallographic samples for evaluation of **thermal spray coatings** is a useful tool. Standards can be used to evaluate areas such as daily **process** variation, change in **procedural** parameters over time, possible changes in preparation **procedure**, changes in consumables, and many other **factors**. With a small investment of time and effort, reliable **metallographic standards can be produced** that will provide **confidence** in the **metallographic** process and produce consistent and reliable **laboratory** results.

References

- 1) Sauer, J.P., **Proceedings of NTSC**, pg. 777-783 (1996)
- 2) Sauer, J.P., **Proceedings of NTSC**, pg. 773-776 (1996)