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Discussion Topics and Threads on Thermal Spray

Compiled and edited by Dr. R.S. Lima, National Research Council of Canada (NRC). These questions and answers were extracted from the discussion group of the Thermal Spray Society of ASM International. The content has been edited for form and content. Note that the comments have not been reviewed. To sign up to the discussion group visit www.asminternational.org. Go to Affiliate Societies, Thermal Spray Society, and under Technical resources sign up for e-mail discussion list—or simply send e-mail to join-tss@maillists.com.

Question 1

Heating the substrate before thermal spraying. You all know that humidity may affect coating adhesion to the substrate during any spraying operation. Do you think it is a must to use a heat source, i.e., gas torch to heat the blasted surfaces before metallizing?

Answer 1.1: Standard practice is to limit time between grit blasting and spraying operations (say, to 2 h maximum). Preheating is difficult to control for large areas (besides, flame torch generates water).

Question 2

Spray forming. We are interested in finding out whether a monolithic substrate made by vacuum plasma spraying (VPS) or low-pressure plasma spraying (LPPS), or controlled-atmosphere plasma spraying, might be a good way for producing bulk material rather than for coating applications. We are very interested in obtaining a very thick (~10 mm) vacuum plasma sprayed coating on a steel substrate.

Answer 2.1: We have vacuum plasma spray (VPS) formed materials such as tungsten and other refractory/oxidation sensitive metals to thickness in the range you describe. Samples can be removed from the substrate for annealing, hot isostatic pressing, vacuum hot pressing, etc.

Answer 2.2: If you are looking for spray forming rather than VPS in particular, cold spraying is a very good method of spray forming. High deposition rates, oxide-free and low-porosity coating are characteristics of cold spray coatings.

Answer 2.3: To what extent does the ductility of the feedstock play a role? Can brittle metals be deposited by cold spray?

Answer 2.4: It is easier to deposit ductile metals by cold spray. However, with the proper nozzle design, powder size plus morphology and optimizing cold spray conditions it is possible to deposit high melting point metals. I have seen examples of metals such as tungsten, tantalum, MCrAlY, Inconel, and titanium being cold sprayed. The available literature so far shows that it is possible to deposit brittle materials such as ceramics and cermets if they are cosprayed with a ductile matrix (metal). The advantage of cold spray over VPS or LPPS would be (for spray forming):

- Narrower spray pattern width (about 5 mm or less depending upon design of nozzle and available powder feed stock)
- Rapid powder deposition and high deposition efficiency
- Possibility of depositing nanocrystalline coating

Moreover, although VPS or LPPS produce dense coatings with low oxide, the issues you may have include problems with leaks in the system, which would lower the quality of your coating. In addition to that, during VPS or LPPS spraying there is a high heat input to the part/coating. The quality of cold sprayed coating is easily controlled as no vacuum chamber is involved.

Answer 2.5: It is important to define what final microstructure is specified. The as-sprayed cold spray structure is basically blocky, with nonmolten particles impacted upon each other. The VPS or LPPS as-sprayed structures can be controlled from the blocky nonmolten particles to splats to more recently equiaxed recrystallized. I would also assume that the deposit efficiency in cold spray will go down as one starts spraying materials such as tungsten or as one changes the substrate materials to those with higher hardness. I would also suggest that the flow rates of gases are higher in cold spray, this can be a problem in production when using helium. In spray forming, depending on what is being formed, sometimes it is advantageous to have a wide spray pattern. I recall about 18 years ago we also used to use words such as rapid deposition rates etc. without understanding that this differs from application to application and material to material in VPS and LPPS. Although cold spray will possibly have a significant niche in thermal spray applications in the future, it is also important to note its limitations.

Answer 2.6: Cold sprayed coatings are formed after significant deformation of the sprayed material and the microstructure reflects the cold working that goes into cold sprayed material. Thus, describing cold spray microstructure as blocky unmelted material is not entirely correct as you would for a plasma sprayed or HVOF coating with nonmolten particles, where the nonmolten particles are due to insufficient heating during spraying. The nonmolten in this case are a source of weakness in the coating. I work with VPS/LPPS systems, and there are significant issues with part cleaning, which affects adhesion. The transfer arc cleaning cleans and preheats the part before applying the coating. Any leak in the system is detrimental to coating quality as it results in improper part cleaning leading to poor adhesion as well as higher oxides in the coating. Additionally, if the process is not properly controlled, damage or melting of the component might take place due to excessive heat input. The advantage of the VPS/LPPS systems is that they can be used to spray a wide variety of materials, and the technology is also relatively better known than cold spraying. There is direct microstructural as well as indirect (x-ray diffraction) evidence of nanograined structure in cold sprayed copper coatings as well as modeling studies that indicate localized heating (possibly melting). These effects result from deformation during impact. Just to emphasize again, cold sprayed coatings are more than blocky nonmolten material impacted upon each other. The deposition efficiency of cold spraying copper is around 90%; it might be lower for spraying materials such as tungsten, etc. I would be happy to receive data on the same if someone has them. The Achilles heel of cold spraying remains the high cost of helium, but again most materials can be adequately sprayed using nitrogen, or, with time helium recovery systems may become available.

Question 3

Coatings that resist sodium hydrochloride. Can anyone recommend a coating that will resist the effects of sodium hydrochloride?

Answer 3.1: Is this for a seawater environment? What temperature are you running at? Are you looking to have the coating acting as a sacrificial anode? If positive, sacrificial anode made of an aluminum coating might be good. Please be aware that these coatings have temperature and pH limits. If you need a barrier coating, Hastelloy C-276 coatings sprayed by a high-velocity process are highly resistant. I would not recommend 300 series stainless steel.

Question 4

Differences between acetylene and propane flame spray. We want to spray steel wire with 0.25%C with propane flame spray. Can anybody throw light on the differences in coating properties in comparison with oxyacetylene flame?

Answer 4.1: In general, carbon steel coatings sprayed with propane tend to have a little more oxides then acetylene sprayed coatings but overall performance is the same. Mechanical coating properties are considered equal, but not much data exist beyond typical hardness and tensile bond strength. Propane gas does require about two times more oxygen to produce an equivalent flame temperature compared to acetylene.

Answer 4.2: I sprayed different metallic wires (including 0.25% C steel) using liquefied petroleum gas (LPG) as fuel. LPG that I used was a mixture of butane and propane. The coatings tended to have more oxides as compared to coatings deposited using acetylene as fuel. In order to get an equivalent temperature with propane you will require two times more oxygen, thus increasing the possibilities of oxide formation in the coating.

Question 5

Spraying onto castings. I have a potential job that requires the application of Metco 136F or equivalent to be applied to a ductile iron casting. I have experienced bonding issues with thermally spraying coating on cast materials. This job requires machining of the applied material. Can anyone provide some insight on this particular application and spraying to cast material in general?

Answer 5.1: Spraying onto castings can be deceivingly difficult. First, you need to ensure there is no oil in the pores of the casting. This is best accomplished by initially heating the part in a low-temperature oven at ~400 °F to "burn out" all oil, followed by cool down and thorough degreasing. Grit blast is best accomplished with chilled iron grit, though others have been used successfully. During spraying, the part temperature must be controlled to prevent overheating. This is very important for maintaining coating adhesion. The use of a good pyrometer such as an infrared type is very helpful. 136F coatings need to be finished by grinding using either silicon carbide or diamond wheels or the thickness for flat surfaces or outside diameters should not exceed 0.025 in.

Answer 5.2: Cast iron tends to erode away instead of taking on a blast profile. No need to keep blasting, a 3 to 5 mils profile just will not happen. We would use a bond coating when applying a ceramic topcoat. Only other thing is to stress the degreasing. Whatever you would normally do, do it twice.

Question 6

Bond coat under WC-Co. This question is in reference to using a bond coat under tungsten carbide (WC) high-velocity oxyfuel (HVOF). Sometimes if a part is more than 0.020 in. undersize we spray an undercoat of bond coat, using either powder or wire. I have had better luck with wire, although it goes on rougher so you have to be careful to knock off any heights and really keep up with your size so you do not break through as you are finishing. For some reason, and I have wondered if it had to do with the impact force of the HVOF process, when I use the power bond material, the finished part will have spider-web-type cracks. I treat all parts the same as far as an initial 150 °F preheat and as-spraying temperature to 300 °F then cool down to 200 °F and keep repeating if I have to. Some parts can be sprayed all the way up without exceeding the 300 °F point. Are my methods the norm? Is there a good limit to how much bond coat I can use under the top coat?

Answer 6.1: We have experienced the same exactly problem. Indeed our colleagues who have gas fuel HVOF systems find this thickness limitation to be a big challenge. We have tried many different approaches to solving the problem:

- Controlling the temperature
- Limiting temperature to below 200 °C
- HVOF buildup, Arc spray buildup
- Arc spray buildup, machine to 0.010 in. (250 μm under final size), blast on arc spray, and then HVOF top coat
- Thick and thin buildup layers

We actually get the best results (as observed after grinding, and in the field trials) from a HVOF buildup of SM 4008NS, followed by the top coat of WC.

Answer 6.2: You are adding extra work wire spraying and knocking off the high spots. How thick do you apply the WC per pass? If it is 0.004 in. on the diameter, that is pushing it. Your temperature range looks good.

Answer 6.3: The problem is thermal expansion mismatches between the coating and the substrate. Fine "spider-web"-type cracking indicates that the substrate was too "cold." During cooling after spraying, the WC coating shrank more than the substrate, generating perpendicular cracks. A solution sounds simple: preheat the substrate more or do not cool too much the surface between the passes (avoid forced cooling—this would be the best).

Answer 6.4: Standard substrate temperature ranges in HVOF WC-Co coating are 150 to 350 °F for steel and Ti and 150 to 250 °F for Al components. If the component surface is peened before applying coating, the top limit is reduced by 50 °F. The top limit is to prevent softening and microstructural changes of the component, the bottom limit is to prevent moisture condensation (from flame) and offset tensile stresses building up in the coating during cooling. Substrate component is to be preheated to 150 °F across, so that some soaking time may be needed during preheating, prior to spraying. Also, quoted temperatures set a guideline only; real substrates can be heavy or light, flat or curved, and coatings can be thick or thin, sprayed at high or low spray rate with high or low traverse speeds, from hotter or cooler flames, and using longer or shorter standoffs. Coating is the main path of heat entering into the substrate component during spraying; thus, monitoring an average coating temperature and its evolution over the entire substrate component during spraying time is a good way to estimate the upper temperature of the substrate. That is because it is reasonable to expect the substrate to be slightly cooler than the coating during spraying. Temperatures will even out during natural (nonforced), convective cooling after spraying, but the initial 150 °F substrate preheat combined with the operation within the prescribed temperature range would, still, ensure compensation of the tensile stresses in the coating on cooling. Elevated preheat and running temperatures could build up excessive compressive stresses in the coating during cooling, hence, flaking. Of course, the thermal alteration of the substrate taking place in this case is another matter. On the other hand, too low preheat and running temperatures, especially cooling the substrate from the "back side" during coating application would lead to high tensile stresses in the coating and the subsequent coating cracking during cooling. The back side substrate cooling during coating should be avoided or minimized because during the subsequent, natural (nonforced) convective cooling after spraying, only coating shrinks, not substrate, i.e., coating cracks. An effective and safe forced cooling (gas jets) during coating operation should be applied from the "front side," so that both the coating and substrate have a chance to shrink in the subsequent, natural cooling after spraying. In a sense, the entire thermal spray coating operation is a gentle balancing act between the coating cracking (for too cold substrates during spraying) and the flaking (for too hot substrates during spraying). The optimum substrate temperature is a function of thermal expansion/contraction coefficients between the coating and substrate, solidification shrinkage of the coating material, and a whole range of additional factors. Bond coat complicates the matters further if it acts as a thermal barrier between the substrate and the top coating, display thermal expansion coefficient much different than the substrate, and/or contains high residual stresses. It is quite possible that wire arc sprayed bond coats are more similar to the metal substrate material than HVOF sprayed bond coats by being coarse grained and less oxidized. This would allow the wire arc sprayed bond coats to expand, contract, and transfer heat more like the substrate with the obvious consequence of producing better top coats whenever the process requires substrate preheating. One more note on metallurgy: the front side cooling or coating surface cooling during spraying makes metallurgical sense when the impinging gas jets do not oxidize the coating material which, in the case of WC-Co can lead to the loss of hardness, mainly due to the conversion of WC to W_2C, and in the case of bond coat would form a layered metal-oxide composite that is undesired with WC-Co top coats for the reasons listed above. Thus, the front side cooling of all metallic and cermet/carbide coatings should be done with inert gas; air is chemically compatible with ceramic oxide coatings.

Question 7

Measuring bond strength via scratch testing. I am trying to measure the adhesion of thermal spray coatings by scratch testing. The best I have learned so far is that scratch testing can be used as a comparative test for different types of coatings. The way we do it is that we prepare a sample for metallography observation and then scratch it across the coating using different loads using a diamond stylus. Above a certain load, a crack is initiated parallel to the coating/substrate interface, the length of crack formed is used as a comparative measure of adhesion for different coatings. I want to find out adhesion strength in MPa by scratch testing; is it possible?

Answer 7.1: Take a look at the following references. They may help you:

- C. Godoy and J.C.A. Batista, Adhesion Evaluation of Plasma Sprayed Coatings Using Piecewise Linear Regression Analysis, *J. Therm. Spray Technol.,* 1999, **8**(4), p 531-536
- M. Bartsch, I. Mircea, J. Suffner, and B. Baufeld, Interfacial Fracture Toughness Measurement of Thick Ceramic Coatings by Indentation, *Key Eng. Mater.,* 2005, **290**, p 183-190
- P. Araujo, D. Chicot, M. Staia, and J. Lesage, Residual Stresses and Adhesion of Thermal Spray Coatings, *Surf. Eng.,* 2005, **21**(1), p 35-40
- D. Chicot, P. Araujo, N. Horny, A. Tricoteaux, and J. Lesage, Application of the Interfacial Indentation Test for Adhesion Toughness Determination, *Surf. Coat. Technol.,* 2005, **200,** p 174-177

Instead of using scratch testing (diamond stylus) for obtaining a scratch parallel to the coating/substrate interface to measure coating adhesion, these authors employed a diamond indenter (Vickers) to indent the coating/substrate interface. By the size of the crack generated, they were able to evaluate the bond strength in terms of toughness units, that is, $MPa \cdot m^{1/2}$. It is a simple and fast alternative to bond strength measurements. These papers do not have exactly what you want, but they may guide you to achieve your objective.

Question 8

Thickness gages and coating temperature effects. During thermal spraying of aluminum we are measuring much higher dry film thickness (DFT) values when coated steel is hot just after Al deposition with arc spray. When this steel gets cold, the same areas are found to be lower in thickness compared to the initial measurements. I know it sounds strange although we are using calibrated DFT gages with National Institute of Standards and Technology (NIST) certificated foils that are used for in-process calibration. Could you please share your opinions and experiences about this issue?

Answer 8.1: Do your gages compensate for thermal expansion of the substrate and coating?

Answer 8.2: I find the same thing happens when we use our magnetic induction meters. I am aware that magnets lose their "power" when hot. Since the meter is measuring magnet strength and then comparing it on a scale, I think it reasonable to assume that reduction of strength equals a thicker coating as far as the meter is concerned. I advise my people not to accept readings on a hot part and to assume them to be high until room temperature. You may be able to get acceptable readings by heating the substrate and doing your calibration on a hot part.

Answer 8.3: I assume you run your thickness gages in "magnetic induction" mode. Magnetic permeability of metals decreases with temperature. Some phases in steel (derivative from austenite) have very low Curie point (temperature when metal loses its magnetism). It is specifically true if your substrate material has little chromium, nickel, or manganese in it. If the substrate material has phases with Curie point just slightly above ambient temperature, then its magnetic permeability will noticeably decrease when heated during spraying, resulting in higher aluminum coating thickness readings by your gages. The remedy is simple: calibration of the gages and thickness readings must be done at the same temperature. I would stick to ambient temperature, unless you would run the test to find out at what temperature thickness readings "stabilize" and perform measurements below this critical point only.

Question 9

Measuring particle temperature and velocity in VPS systems. Has anyone been able to measure powder particle speed for VPS spraying? I have tried high-speed photography through a view window but could not get any results even at 18000 frames per second. Has anyone tried laser Doppler anemometry with this process?

Answer 9.1: We once did some work with Control Vision, Inc. (Idaho Falls, ID) using an orthogonal laser sheet illumination system on VPS and got some believable numbers. Depending on the filters and exposure time used, straight high-speed photography can be a big hit or miss owing to things such as persistence of luminosity from the plasma. The current systems like SprayWatch, Thermaviz, DPV 2000, and so on use CCD detectors and fast shutters together with multiple exposures to capture either streaks or multiple images of particles from which particle speed can be determined. Some systems can measure particle temperature too using two-color pyrometry. Sometimes it is necessary to illuminate the particles traveling in a bright plasma jet using something that momentarily "outshines" the plasma and reflects back to the detector, and this is done using. for example, pulsed lasers with narrow-band filters etc. These systems tend to favor the hotter/brighter/larger particles, which are more readily seen by the detectors. Jim Fincke at the Idaho National Engineering and Environmental Laboratory (INEEL) has also worked extensively in this area and published a number of papers in the *Journal of Thermal Spray Technology* and the various thermal spray conference proceedings. In Oct 2004, ASM Thermal Spray Society (TSS) organized a two-day workshop meeting at NRC in Boucherville, Canada on "Sensors & Controls," which included presentations on then current sensors and systems.

Answer 9.2: The DPV 2000 has been used to carry out particle temperature and velocity measurements in different VPS systems at LERMPS (France), EMPA (Switzerland), Sulzer-Wohlen (Switzerland), Osaka National Research Institute (Japan), etc. Some results were reported in ITSC Proceedings and other journals.