# Introduction to Coating Design and Processing

SEVERAL KEY PROCESSING STEPS are required to produce optimized thermal spray coatings. For example, to ensure adequate bonding of a coating, it is critical that the substrate be properly prepared. The substrate surface must be clean and usually must be roughened after cleaning by grit blasting or some other means. Masking and preheating of the substrate are most commonly carried out prior to the application of thermal spray coatings; for more detailed information, see the section "Processing Prior to Coating" in this article as well as the article "Precoating Operations" in the "Pre- and Postcoating Processing" Division of this Volume. Coating quality also depends on spray process variables and parameters such as part temperature control, gun and substrate motion, spray pattern, and deposition efficiency and deposit rate.

Postcoating operations further enhance the quality of thermal spray coatings. As discussed in the section "Postcoating Processing" in this article and in greater detail in the article "Postcoating Operations" of the "Pre- and Postcoating Processing" Division of this Volume, these operations include finishing treatments (such as grinding and polishing), densification treatments (fusion, hot isostatic pressing, and heat treating), sealing, and testing and inspection.

In addition to these processing steps and parameters, it is important to understand how to design coatings for optimal performance. Thermal spray coatings are fabricated by combining equipment (spray guns, process controls, manipulators, etc.), feedstock materials (powders, wires, or rods), and engineering and process know-how (experience). Only when all components of the spray process are used correctly does a well-designed coating result.

This introductory article reviews the advantages and disadvantages associated with thermal spray processing. Good processing practice allows the advantages of thermal spray coatings to be realized, while minimizing some of the disadvantages.

# Advantages and Disadvantages of Thermal Spraying

Thermal spray coatings exhibit a unique microstructural architecture. Coatings are formed as melted and partially melted particles of different sizesimpact substrates at a rate of perhaps one million particles per second and build up one upon the other, as shown in Fig. 1. Figure 2 shows the typical lamellar (layered) thermal spray coating microstructure that results from particulate deposition. This unique microstructure imparts several advantages and disadvantages.

## Advantages

Many of the defects identified in Fig. 2 are controllable through appropriate equipment

Solid or powder feedstock

and feedstock selection, but by far the greatest influence on coating structure comes from the actual processing step. Properties of a sprayed coating stemming from the lamellar shape of the splats and residual porosity can be overcome by postdeposition treatments. Porosity can sometimes be a benefit, as in the cases for oil retention on bearing surfaces, for chemically active structures such as batteries, for bone attachment in orthopedic implants, and so on. Rapid particle cooling rates and the lamellar splat shapes are two features that distinguish thermal spray coatings from other coatings. Brittleness, hardness, anisotropic properties, and residual stresses are the result of very rapidly cooled and flattened particles. Properly applied thermal spray coatings have many uses and offer several advantages.





Fig. 1 Schematic of the thermal spray process



Fig. 2 Thermal spray coating. Buildup of a thermal spray coating is a chaotic process. Molten particles spread out and deform (splat) as they strike the substrate, at first keying onto asperities on the substrate surface, then interlocking to one another. Voids can occur if the growing deposit traps air. Particles overheated in the spray jet can become oxidized. Unmelted particles may simply be embedded in the accumulating deposit.

A wide range of materials can be deposited as coatings, including metals, metal alloys, oxide and nonoxide ceramics, plastics, cermets, and composite structures comprised of metals, ceramics, and plastics, can be applied using thermal spray. Competitive coating processes do not afford this versatility.

Low Processing Costs. Rapid deposition rates on the order of 1 to 45 kg/h (2 to 100 lb/h) or more can be achieved. Typically, 2 to 7 kg/h (5 to 15 lb/h) is normal practice. Rapid spray rates and high deposit efficiencies result in relatively low processing costs.

Wide Range of Coating Thicknesses. Coating thicknesses from  $25 \mu m$  to  $6.5 \mu m$  (0.001 to 0.250 in.) are used today (2012). Thicker coatings are possible using electric arc spray, cold spray, and vacuum plasma spray (VPS). Coating capabilities on the basis of thickness are:

- Wide application range. Thermal spray coatings function effectively for a broad range of surface modifications.
- Wear resistance—abrasive, adhesive, galling, antifretting, cavitation, and erosion
- Multilayered thermal barrier coatings comprising metallic bond coats and oxide ceramic topcoats
- Abradable and abrasive coatings for gasturbine engines
- Atmospheric and aqueous corrosion control
- High-temperature oxidation resistance and corrosion control
- Electrical resistance and conductivity
- Net and near-net shape component manufacturing<br>Metal- and
- ceramic-matrix composite structures

Equipment Costs and Portability. Basic thermal spray equipment is relatively low in cost compared to competitive coating processes and can, to some extent, be made portable. Exceptions to this are VPS and fully integrated thermal spray systems.

Minimal Thermal Degradation of the Substrate. With proper control, there is little risk of thermally degrading the substrate during spraying. In terms of substrate interaction, thermal spraying is a relatively "cold" process, and substrates are usually kept below 65  $\degree$ C (150  $\degree$ F). Higher temperatures are often used to produce enhanced bonding or unique coating characteristics.

#### **Disadvantages**

As with all coating technologies, thermal spray has some limitations. Understanding these limitations is important so that engineers can design effective solutions to the challenges of surface modification. Failures have occurred because coating and substrate limitations were not considered or were poorly understood.

Porosity. Thermal spray coatings generally exhibit some porosity, allowing the passage of gases and/or liquids through to the coating/substrate interface. Porosity is controllable down to

levels below approximately 0.1% with most processes. Postdeposition treatments such as fusion, sintering, or hot isostatic pressing can decrease porosity and close up splat boundaries. Surface and interconnected porosity can be closed off using a variety of sealing methods, including anaerobic sealers, epoxies, phenolic sealers, and so on. Connected porosity is minimized by using high-velocity oxyfuel, VPS, and cold spray processes.

Anisotropic Properties. Thermal spray coatings are inherently anisotropic in the as-sprayed condition. That issue is discussed elsewhere in this Volume.

Line-of-Sight Process. Thermal spray deposition of coatings on simple geometrical configurations is relatively straightforward. Complex geometries, such as gas-turbine airfoils, require automated, robotically-controlled manipulation of the gun and/or substrate to properly address the coating surface. Deviation from spraying normal to the surface can compromise coating properties (Fig. 3a). As a rule of thumb, low-velocity spray processes<br>need to stay within  $+15^{\circ}$  of normal (Fig. 3b). need to stay within  $\pm 15^{\circ}$  of normal (Fig. 3b).<br>Higher-velocity processes can tolerate off-axis spraying up to  $+45^{\circ}$  (Fig. 3b). Porosity tends to increase and coating cohesion tends to decrease, along with deposit efficiency, beyond these limits.

## Processing Prior to Coating

Processing of thermal spray coatings may involve some or all of the steps outlined in this article. Each step represents an essential part of the planning process that must be considered before and during the coating process. For instance, a first-time repair application will require some attention to all of the steps shown, whereas some of the steps can be omitted for an original-equipment-manufactured component. More information on processing prior to coating can be found in the article "Precoating Operations" in the "Pre- and Postcoating Processing" Division of this Volume.

Thermal spray begins with proper surface preparation, including cleaning, roughening, and deburring, chamfering, or radiusing edges, and so on. Repairing worn parts often requires undercutting to remove damaged surface.

After cleaning, masking protects selected component areas, adjacent to the area being coated, from unwanted abrasive and/or spray particle impact. Common masking methods include metal shadow masks, high-temperature tapes, and paint-on compounds. Some tapes offer protection from both abrasive grit and sprayed particles without damage.

Preheating, the next step before spraying, is used to drive off moisture and present a warm, dry surface to the first impacting particles. Aluminum, copper, and titanium substrates normally are not preheated. Excessive preheating in either time or temperature should be avoided, because it may oxidize the surface.

#### Substrate Metallurgy and Thermal History

Substrate material type, hardness, and prior heat treatment must be evaluated before proceeding. Understanding metal alloy type and heat treatment helps determine the correct surface preparation and preheat temperatures to use during spraying. Thermal spray coatings can be applied to virtually any substrate, including metals, metal alloys, wood, plastic, glass, paper, and so on.

Substrates with hardnesses less than 45 HRC can be cleaned, roughened, and sprayed using most standard techniques. When hardness exceeds 40 to 45 HRC, surface roughening with common abrasive grits and techniques is difficult to achieve. Procedures for handling hardened substrates are covered later. There will be little change to heat treated substrates if preheat temperatures are kept below 65 $\degree$ C  $(150 \text{ °F}).$ 

Care should be taken when preheating materials that oxidize easily, such as copper, titanium, aluminum, zirconium, uranium, and so on, to minimize oxidation prior to coating deposition. When spraying reactive materials, limit preheat temperatures to 65  $^{\circ}$ C (150  $^{\circ}$ F) and spray immediately.



Fig. 3 Thermal spray stream positions. (a) Good. (b) Acceptable. (c) Minimum acceptable. HVOF, high-velocity oxyfuel

## 78 / Thermal Spray Processes and Coatings

Castings or powder metallurgical (P/M) components may require special cleaning, because of inherent porosity. This often includes baking out trapped moisture and processing fluidlike cutting oils.

## Undercutting for Repair and Dimensional Restoration

When parts come in for repair, the surface is usually damaged due to wear and/or corrosion. Repair procedures call for removal of the damaged surface material by undercutting into the substrate. Undercutting removes some good material as well as the damaged surface in order to provide a uniform flat surface to accept the new coating. It is important to remember that undercutting reduces the cross-sectional area and correspondingly reduces the tensile and ultimate strength of the part being restored. Sprayed coatings will not restore the original tensile strength, even though the cross-sectional area will have been restored. The undercut surface area can be prepared by grit blasting, macroroughening, or bond coating, depending on the depth of undercut and the coating material chosen for spraying. It is important to reiterate that macroroughening can reduce fatigue life as well as the strength of the part.

## Substrate Preparation, Part Configuration, and Coating Location

In addition to the following discussion of substrate preparation and part configuration and coating location considerations, more information can be found in the "Pre- and Postcoating Processing" Division of this Volume.

The substrate should be inspected for surface defects such as cracks, scratches, dents, porosity, organic contamination, and so on. Superficial flaws will be replicated in the sprayed coating, more so with thinner coatings. Conventional coatings do not add strength to the substrate and cannot be used to repair damage that may affect structural integrity.

Engineering drawings and job routers should be examined for coating location, coating thickness, and other coating attributes prior to cleaning, surface preparation, and coating. Understanding the part geometry is important to fixturing and tooling in order to maintain the proper spray angle. The following acceptable practices and design considerations should be considered for compliance with coating requirements (Fig. 4):

- When the substrate target area is undercut to accept a coating (Fig. 4a), always chamfer or radius the corners before spraying. One rule of thumb is that the radius should equal or exceed the sprayed coating thickness.
- Likewise, the substrate edges as shown in Fig. 4(b) should be chamfered or radiused. Again, a 45° chamfer or radius equal to the coating thickness is suggested or

recommended. Edge effects common to all coating techniques cause unacceptable stresses at the edges, which in turn will lead to cracking and coating separation.

 Figure 4(c) shows the acceptable practices for depositing a coating onto a substrate and against a shoulder or boss. Using procedures similar to undercutting, corners must be radiused or chamfered. Sharp corners against a shoulder are likely to result in porosity and voids.

Substrate surface preparation prior to thermal spraying is absolutely essential. Steps must be



Fig. 4 Design of substrate geometry for thermal spray coating processes

undertaken correctly for the coating to perform to design expectations. For a one-of-a-kind part to be coated or repaired, surface preparation, masking, and setup may account for 75% of the total time required to complete the job. Considering labor and overhead expenses, the task should be done correctly the first time.

Cleaning. The first step in preparing a substrate surface for spraying is to remove organic and inorganic contaminants such as oil, grease, paint, rust, scale, and moisture. Contaminants remaining on the substrate will affect adhesion.

Vapor degreasing is commonly used to remove oils, greases, and other solvent-soluble organic compounds. It is fast, economical, and efficient. Hot vapor degreasing is a variation that is even faster and more efficient. Porous materials such as castings, P/M parts, or cast irons should be soaked for 15 to 30 min to dissolve hidden contaminants that may have penetrated the substrate. Baking may be required to remove contaminants from these porous structures. Large parts can be steam cleaned or submerged in solvents. Manual cleaning with solvents is often all that is necessary to remove surface greases and oils from machined components. Common solvents include:

- Alcohol
- Acetone
- Aqueous washer solutions containing acetic acid

To avoid the possibility of corrosive cracking, chlorinated solvents must be avoided when cleaning titanium and titanium alloys.

Baking. When solvent soaking does not work for porous materials such as castings or P/M parts, baking at 232 °C (450 °F), actual part temperature, for 1 h will vaporize and remove most deeply penetrated oils. If not removed, residual oils will "bleed" out of the substrate onto the surface during preheating, coating, and/or after spraying.

Ultrasonic cleaning can also be used to dislodge some contaminants in confined or hidden areas. Commercial equipment is available, together with the manufacturer's recommended cleaning solutions. Check to be sure that the solution chosen is compatible with the substrate material. Ultrasonic cleaning can also be used to remove residual entrapped grit after grit blasting. The substrate must be oriented, however, so that dislodged grit can fall free of the substrate.

Dry abrasive blasting rigorously removes surface contaminants. This process is used to remove old sprayed coatings, mill scale, paint, burrs, corrosion products, and oxides. Never use the same abrasive equipment for both cleaning and roughening, because contaminants from cleaning could contaminate the surface.

Roughening. Second to cleaning, roughening is the most important step in preparing the surface to accept the sprayed coating. Common methods used to roughen surfaces include:

- Grit blasting
- Machining or macroroughening
- Application of a bond coat, where grit blasting is not possible

Combinations of these methods are often used, for example, bond coats applied over grit-blasted surfaces to provide the highest bond strength, or grit blasting over macroroughened surfaces. Properly cleaned and roughened surfaces provide the critical interface upon which the first layer of coating particles impact. A properly prepared surface has the following attributes:

- Cleanliness provides white metal-to-metal contact for interatomic and metallurgical interactions between the substrate and sprayed particles.
- Roughening provides significantly increased surface area for particle-to-substrate contact and increased atomic and metallurgical interaction.

Dry abrasive grit blasting is the most commonly used surface-roughening technique. Dry abrasive particles are propelled toward the substrate at relatively high speeds. On impact with the substrate, the sharp, angular particles act like chisels, cutting small irregularities into the surface. Note that masking of the part may be necessary to confine the grit blasting to the area to be coated. Masking is discussed subsequently.

The amount of plastic deformation of the surface is a function of the angularity, size, density, and hardness of the particles, in addition to the speed and angle at which the particles are directed toward the substrate. The result of grit blasting places the outer layer of the substrate in residual compressive stress. With materials prone to fatigue, such as titanium, substrates are sometimes shot peened before dry abrasive grit blasting to induce a deep compressive stress.

Grit-Blasting Variables. Variables that determine the size and geometry of the surface irregularities include the size, shape, hardness, specific gravity, and chemistry of the abrasive particles, as well as their speed and angle of impingement. Hardness of the base material and uniform coverage of the substrate are other important variables.

Abrasive Grain Size. Because the mass of an abrasive grain varies as the cube of its diameter, the force of the impact of a 20-mesh  $(840 \mu m)$ abrasive particle will, for example, be eight times that of a 40-mesh  $(420 \mu m)$  particle (assuming the particles are equiaxed). Consequently, the larger particle will cut much deeper into the surface, all else being equal. Following ANSI B74.12, Tables 2 and 3, common grit sizes for thermal spray are 16, 24, 36, 60, 80, 120, and 240. Grit selection is based on roughness requirements and substrate tolerance to the grit.

Surface roughness values are generally expressed in microinch  $(\mu$ in.) or micrometer (um) units. The American National Standards Institute's standard for surface roughness measurements calls for  $R_a$ , which the arithmetic average deviation from the mean (ANSI B46.1-1978, "Surface Texture," American Society of Mechanical Engineers). Other definitions of roughness, such as root mean square, are falling into disuse. There are many other significant attributes of roughness that are equally, if not more, important in defining roughness. Those parameters help to define the population density of irregularities and surface area. See Fig. 5 for a guide to surface roughness expected from alumina of various grit sizes on typical substrates.

Particle Shape (or Morphology). The angularity of the abrasive particles profoundly affects the depth and shape of the pits cut into the substrate surface. Rounded shot will make smooth, rounded indents in the surface, producing roughness with little added surface area. Angular grit crushed from chilled iron shot provides a greater roughness and surface area. In general, abrasive particulate with sharp, angular cutting edges is required.

Particle Hardness. Blasting particles should be hard enough to cut efficiently but not so brittle as to shatter and embed excessively. A certain amount of toughness (ductility) is desirable to avoid rapid breakdown.

Specific Gravity (Density) of Particles. Angular chilled iron grit has nearly twice the density of the heaviest nonmetallic abrasive and so will have nearly twice the impact energy at the same velocity, for equal-sized particles. This will result in deeper pitting.

Grit Velocity. With any type of abrasive, the force of the impact varies directly with the impact speed. Hence, higher pressures result in deeper pitting. With nonmetallic abrasives,



Fig. 5 Typical surface finish obtained using aluminum oxide abrasive. Curves illustrate the range of results obtainable by pressure and abrasive size variations. The exact results will be influenced by material differences, nozzle selection, and other variables. RMS, root mean square

high pressures result in rapid shattering of grit particles, and for this reason, blast pressures are reduced to save on grit. As a rule, blast machines of the suction-feed type provide only one-third to one-half the speed possible with the pressure-pot type of blast machines. It has been demonstrated and reported that increasing grit size is more effective in increasing roughness than increasing air pressure.

Angle of Impact. It is generally considered that the angle of impingement of the blasting be similar to that of the spraying, maintained as close to  $90^\circ$  to the surface as possible for most materials, but may be varied to lower depending on the substrate materials and grit type.

Substrate Material Hardness. It is difficult to obtain sufficient roughness for a strong bond to hard metals or ceramics through grit blasting. This is largely true because it is difficult to cut the hardened surface to the required depth to create roughness. Hard materials lack ductility, and the surface cannot be displaced in a manner that provides rough asperities. On hardened materials, the use of larger grit sizes with harder grit will effectively fracture, creating roughness. This approach has substrate limitations. However, when coupled with effective preheating, it can produce good bonding.

Uniformity of the Grit Blasting. Grit blasting should be accomplished in one uniform traverse of the surface being blasted. This is best accomplished with automation but can be done by experienced operators. The intent is to maximize roughness while minimizing embedment of the grit and avoiding work hardening of the substrate.

Abrasive Grit-Blasting Equipment. There are three basic types of grit-blasting equipment:

- Pressure machines or pressure pots
- Suction-type nozzles
- Centrifugal or airless blasting machines

Pressure machines provide higher abrasive speeds and use air more efficiently than suction-feed types (Fig. 6a). For work on large pieces or for continuous production blasting, suction-type systems are used. Properly engineered systems provide a means for removing dust and ultrafine particles from the abrasive to maintain proper grit sizing.

Grit velocity and blasting efficiency, with pressure-type machines, depend primarily on nozzle diameter and air pressure. Table 1 shows the consumption of air, sand, and chilled iron grit for various nozzle sizes and air pressures, and the horsepower needed to drive them.

Suction-type nozzles (Fig. 6b) do not provide as high an abrasive speed as pressure blasters, and they are less efficient with respect to use of air; however, they are very convenient for small work, on-site/field work, and continuous blasting operations. The abrasive is recycled so that periodic reloading is not required.

Centrifugal blasting machines are well suited for production blasting, particularly for large parts with thick cross sections. With this equipment, grit is thrown in a fan-shaped pattern against the substrate by a rapidly revolving paddle wheel. In some cases, the work is also rotated to expose all surfaces to the blast. Other machines may use two or more wheels that can be positioned to suit the substrate geometry. This kind of equipment is much more efficient than air-type blast generators in terms of power used.

Blasting abrasives may include the following types.

Angular chilled iron grit is practical for use with centrifugal blasting equipment in preparing surfaces for flame spraying. Chilled iron grit has hardnesses up to 60 to 62 HRC and can be used on substrate materials as hard as 40 to 45 HRC. Chilled iron is often called steel grit, but steel grit has a hardness of only 43 to 48 HRC and is too soft for most blasting. Steel grit becomes dull and does not produce the sharply defined surface details needed for proper bonding of sprayed coatings. Chilled iron grit stays sharp longer, and some fracturing occurs during use to produce new, sharp cutting edges.

Maintaining the right grit-size distribution requires that blasting equipment is capable of screening and dust collection during blasting. Grit size should be the smallest that will produce the desired surface characteristics.

Alumina  $(Al<sub>2</sub>O<sub>3</sub>)$  grit works well in blasting cabinets of both designs. The specific gravity of alumina is just over half that of chilled iron, so that this type of abrasive is readily picked up by the suction feed and is effectively accelerated through the blasting nozzle. The rate of

breakdown is not excessive when used at moderate air pressures.

Brown, fused alumina is the more commonly used grit medium and is extremely hard  $(>9$  on the Mohs scale); when properly crushed, it has sharp cutting edges. Alumina grains of 240 and 150-mesh size are shown in Fig. 7.

Alumina grit is also used for hard surfaces (>45 HRC) and on nonferrous materials where embedded steel grit could result in corrosion problems.

Crushed flint for blasting is available in all mesh sizes. Some users feel that crushed flint is superior to ordinary silica sand. It is very clean, and the particles are extremely angular and sharp. Flint is, of course, a form of silica, and proper precautions against silicosis must be taken.

Crushed garnet is available in several areas. The composition of garnet varies widely, and some grades may contain free silica and thus mandate respiratory protection. As may be expected, the breakdown rate also varies with garnets from different sources. Some grades are almost dust-free when blasting, while others produce nearly as much dust as silica sand.

Silicon carbide (SiC) is also available in many mesh sizes. However, for coating applications where high temperatures are encountered, it is not recommended because of its reactivity with various substrates and coating materials.

Crushed slag is a black, glassy grit made from the slag produced by certain types of furnaces.



Fig. 6 (a) Pressure-type and (b) suction-type grit-blasting machines. Adapted from Ref 1





Ordinarily, it contains no free silica, but this should be verified with the supplier. Crushed slag is usually angular and well graded.

Machining and Macroroughening. Macroroughening is usually accomplished by machining grooves or threads into the surface to be sprayed (Fig. 8). Typically, rough-machined surfaces are also grit blasted prior to spraying. Surfaces roughened to this magnitude are often used for thick coatings to restrict shrinkage stresses and to disrupt the lamellar pattern of particle deposition in order to break up the shear stresses parallel to the substrate surface (Fig. 9). Caution should be used with this technique. Threading reduces cross section and can affect load capability and increase fatigue.

When to Start Spraying. It is always good practice to begin thermal spraying of a component as soon as possible after completion of surface preparation. Freshly exposed clean metal is metallurgically active and readily susceptible to contamination and oxidation. Fingerprints or contact with foreign materials may severely affect coating adhesion. If a freshly prepared surface must be stored before spraying, it should be kept covered and stored in a clean, dry area with the temperature maintained above the dewpoint, but preferably within two hours depending on substrate materials.

Application of a Bond Coat. Using a bond coat is a common method of obtaining a roughened, textured surface and to mitigate coefficient of thermal expansion (CTE)-induced differential expansion between the coating and substrate.

The following guidelines can be applied to determine when to use bond coats:

- A minimum of surface preparation is possible. This is particularly useful on thin substrates where blasting may cause warpage or distortion.
- Increased reliability when combining grit blasting with bond coating
- Use for hard substrates that cannot be effectively roughened by grit blasting
- Use for corrosion protection of the substrate
- Use to help accommodate differences in CTE between topcoats and substrates
- When the topcoat has a thickness limitation, bond coatings may be applied to build up extra thickness for repair work.

Bond coat thicknesses are typically between 75 and  $125 \mu m$  (0.003 and 0.005 in.).

Bond Coat Materials. Bond coats can be applied by wire- and powder-flame spray, plasma arc spray, high-velocity oxyfuel (HVOF), and electric arc spray. Most materials are available in the form of powders or wires. Some typical materials, their chemistry, and service temperatures are listed in Table 2.

#### Masking

Various masking techniques have been developed to protect components next to the coating area from impact by overspray particles. These methods include:

- Shadow masks
- High-temperature tapes, most commonly two-sided, silicone tape to resist blasting and spray, 3M stencil tape for blasting and spray resistance, and glass-filled tape for both blasting and spraying
- Paint-on masking compounds

Metal shadow masks are placed approximately two to three times the total coating thickness away from the part to be sprayed and in front of the spray torch, shielding selected areas from the direct spray stream (Fig. 10). The targeted spray area may be stationary or rotating. The mask is typically stationary and securely affixed. As the spray stream is traversed across the target area and onto the mask, the unwanted spray collects on the mask and is prevented from reaching the substrate. The coating will end in a narrow feathered band rather than as a sharply defined line (Fig. 10b). In contrast to the feathered edge, note that a contact mask (Fig. 10a), similar to the tape that is discussed in this section, leaves a sharp edge, which may act as a stress raiser that

often leads to disbonding of the coating. Metal masks can be reused, subject to the amount of coating buildup and/or distortion due to heat from the spray process.

Sheet metal masks are preferred for HVOF spraying, because tapes do not stand up to high-velocity particle impact and the high thermal output of the gun.

Tape masks are applied by hand wrapping areas that do not require a coating. After spraying, the tape is easily removed, along with accumulated overspray, leaving the protected areas clean. Many different tapes are available commercially, some for resistance to grit blasting and others for thermal spraying. The best tapes for the job have sufficient toughness to resist both grit blasting and heat resistance to withstand hot process gases and particle impact during spraying. The coating line when the tape is removed after the application of thin coatings leaves a neat and clean edge, but if the coating is thick, chipping is likely to result where bridging over the tape has occurred.

Masking Holes and Complex Geometries. Further masking techniques are shown in





**Fig.** 7 Aluminum oxide  $(A|_{2}O_{3})$  particles. Original magnification:  $130 \times$ . (a) 240 mesh. (b) 150 mesh

Fig. 11. Radiused corners are protected using curved masks to keep unmelted particles and other debris from contaminating the coating. Dowel and screw holes can be masked using simple tapered silicone rubber plugs whose top surfaces are above the surface being coated and a slightly larger diameter than the hole, so tapered, low-stress edges will result.



 $(a)$ 

 $(a)$ 





Fig. 8 Examples of surface preparation by threading or grooving. Adapted from Ref 2





Fig. 9 Comparison of thermal spray coatings deposited on macroroughened and smooth surfaces. (a) Sprayed metal over grooves; shrinkage constrained by grooves. (b) Sprayed metal on smooth surface; effect of shear stress on bond due to shrinkage. Adapted from Ref 3

Masking compounds are useful for providing short-term protection from overspray but are not useful for protection from grit blasting. Commercially available compounds comprise refractory particles, such as graphite, carried in water- or solvent-soluble binders. These can be applied by brushing or spraying. When dry, the film prevents overspray from sticking, while the refractory particles provide short-term heat protection. Excessive exposure to the spray stream, however, will cause these compounds to break down, after which spray particles may stick. Residual compound is easily removed by rinsing with water or solvents, depending on the carrier solubility. Masking compounds are easy and convenient to use in spite of their shortcomings. Epoxy/puttylike compounds are also suitable for plasma and HVOF masking needs.

#### **Preheating**

Preheating of substrates prior to thermal spraying is a normal and acceptable practice. A no-powder preheat pass using the flame or plasma is often used to drive off adsorbed moisture from the surface of the part. Highertemperature preheats can be used to offset the CTE mismatches between the coating and substrate. Coating at an elevated substrate temperature can place the coating in compression after cooldown. This approach is not practical for all applications but can be useful for moderate operating temperatures.

Preheating Temperatures. As a general rule, a single preheat pass is sufficient to drive off adsorbed moisture. Larger parts may require more preheat passes or supplemental heating. Preheating temperatures are part, coating, and application dependent and typically range from 100 to 150 °C (210 to 300 °F). As soon as the preheat temperature is reached, spraying must begin.

Although preheating is an accepted and common practice, preheating is not recommended for some materials. Carbon- and glass-fiberreinforced composites have matrices that are temperature-sensitive. To remove moisture from sensitive substrates, an alcohol rinse prior to spraying is used.

Excessive preheating will cause surface oxidation. Materials such as copper, aluminum, titanium, uranium, beryllium, and zirconium oxidize readily in air, producing a layer that

Table 2 Common bond coat materials

Coating	Wire	Powder	Service temperature	
			$^{\circ}C$	°F
Molybdenum	Yes	Yes	315	600
80Ni-20A1	.	Yes	845	1550
90Ni-5Mo-5Al	.	Yes	.	
95Ni-5Al	<b>Yes</b>	Yes	845	1550
95NiCr-5Al	Yes	Yes	980	1800
95FeNi-5Al	Yes	Yes	1200	1650
90FeNi-5Mo-5Al		Yes	650	1200

## Introduction to Coating Design and Processing / 83



Fig. 10 Coatings resulting from (a) contact masks versus (b) shadow masks



Fig. 11 Examples of how to use masking techniques for special configurations

inhibits good bonding. Preheat and coating temperatures should be kept lower, below  $100^{\circ}$ C (212 $\degree$ F), for high-expansion alloys such as the 300-series stainless steels, superalloys, and nickel-base alloys. Lower-expansion coatings such as carbides and cermets will fail due to CTE mismatches when applied to these alloys at higher temperatures. However, careful selection of coating material relative to the substrate composition and preheating temperatures can result in the formation of compounds at the interface that produce extremely strong bonds.

Atmospheres. Spraying done under inert cover, either by inert gas shrouding or by VPS, can take advantage of very high preheating because the deleterious effects of oxygen are minimized. For example, gas turbine blades that are to be plasma sprayed with MCrAlY coatings in low-pressure inert gas chambers are often preheated to 815 °C (1500 °F).

## Coating Deposition

## Spray Stream

The spray stream is fundamental to understanding thermal spray coatings. Spray processes involve the acceleration of particulate matter in a cold to molten or semimolten state toward a substrate to form a coating. Particle trajectory, temperature, and velocity at any moment in time in the gas stream constitute the spray stream or pattern.

To understand what is happening in a hot jet stream, refer to Fig. 12, which shows particle trajectories in a plasma jet. While the figure shows particles entering the gas stream radially, they could just as well be introduced along the centerline of the jet or generated by atomization, as in wire flame spray or electric arc spray. Some particles travel along the jet centerline, others take an intermediate position, and still others ride along the periphery of the jet. Fine particles riding the periphery may, in fact, never enter the main jet, and coarse particles may pass through the jet and take an outside position on the opposite side to their injection point. On impact with the substrate, particles impacting virtually simultaneously make up the spray pattern.

Figure 13 depicts the spray pattern in cross section. In a planar view, parallel to the substrate, the spray pattern is circular or oval in shape. The deposit at an instant in time is essentially Gaussian in cross section, where, at the center, the highest degree of melting has occurred. Moving radially out from the center, temperatures and velocities are lower, resulting in slightly different microstructures. Each particle will have a unique time/temperature/ velocity profile as it passes through the plume or jet. By maintaining tight control of the feedstock and parameters, the stream can produce a statistically significant number of properly melted and accelerated particles.

The spray-pattern phenomena described in this section and shown in Fig. 12 and 13 apply



Fig. 12 Particle trajectories in a typical plasma spray stream

to all thermal spray processes. Variables common to each process, such as total gas flow, energy levels, particle size distribution, specific gravity, and carrier gas flow, combine to affect time, temperature, and mass. These in turn affect porosity, unmelted particles, and oxides quantitatively. The spray pattern varies between processes more as a function of gas stream velocity.

#### Coating Buildup

As the spray stream is manipulated over the part, overlapping occurs. After several passes, a coating such as that shown in Fig. 14 results. A typical coating may include some porosity, unmelted or resolidified particles, and oxides. Depending on hood and dust collector design, masking and tooling, and airflow around the part, fumes from vaporized feedstock may be trapped in the layers between passes.

Effect of Part Geometry on Coating Buildup. Figure 15 illustrates two basic concepts related to part geometry. First is that inclusion of unmelts and other debris is reduced when spraying small target areas. This effect is illustrated in Fig. 15(a). The shaft diameter is smaller than the spray pattern diameter; consequently, one-half of the spray stream actually misses the substrate. The larger target area shown in Fig. 15(b), however, captures the entire spray pattern on the substrate when traversed across and/or up and down. To state the obvious, process efficiency is poor when coating small parts, because of the low target efficiency.

The second concept is that small, round parts do not behave as flat surfaces, due to the angle of impingement. As a rule of thumb, curved surfaces begin to approximate flat surfaces at radii above 40 mm (1.5 in.).

Substrate Cooling Devices. Supplemental air cooling has two distinct advantages: to control part temperature and to remove overspray and fume debris from the coating surface. Clean, dry air is a must. Condensation and compressor oils are detrimental to coatings. To spray large target areas, an ancillary air-cooling device can be attached to the spray gun or the manipulator following the gun, such that a stream of compressed air blows dust and debris from the substrate surface in advance of, and following, the traversing spray stream. Figure 16 shows an air device positioned for spraying a large shaft. Here, debris is blown off the surface before it can be embedded into the coating. When spraying flat surfaces, as shown in Fig. 15(b), air coolers should be positioned on both sides of the spray stream to help remove dust and debris. Air coolers are usually adjusted to precede and follow the spray torch along the traverse direction. However, air cooling can be placed anywhere on the part as long as it does not interfere with the spray stream. The type of air jet, placement, and air volume are determined by the temperature required and the potential to create and embed debris.

Cooling with air may be the most common type or source of cooling; however, other types may be used, including liquid  $CO<sub>2</sub>$ , which is very efficient, or Ar or  $N_2$  gases in specialized applications.

Relationship between Particle Melting and Coating Structure. In addition to the effects of particles in the periphery of the spray stream, resulting from poorly classified materials or nonoptimized process parameters, consideration should be given to particles in the spray stream and how variations in the degree of melting and velocity affect coating structure. Particles in the center of the spray pattern may include one or all of the following states on impact:

- Fully molten, or just above the material melting temperature
- Superheated, well above the melting temperature and perhaps close to the vaporization point



Fig. 13 The spray pattern, illustrating particle deposition and the effect of size and debris on thickness and porosity in cross section

- Semimolten, with the outside liquid but the core still solid
- Molten then resolidified while in flight before impact
- Under- and overaccelerated particles

Fully melted particles at or just above the material melting point arrive at the substrate, impact, flow, and flatten (Fig. 17). Particulate material spreads and cools rapidly as heat is conducted into the substrate. The classic lamellar splat morphology is thus achieved (Fig. 17b). Lamellar particle thickness depends on particle velocity, particle temperature, substrate temperature, and other minor factors. Superheated particles may splash on impact, sending out fine droplets radially that can end up in the coating as debris (Fig. 17c). Debris formed from splatter differs from debris produced in the jet, in that splatter lodges in the coating, building up at the first ridge that stops its radial travel. Air blasting does not remove splatter debris. Superheating is a condition that generally should be avoided, in that it produces fumes, oxidation, and lowered deposition efficiency. Particles that resolidify in flight after having been melted may either not deposit or they may appear as embedded unmelted particles, usually with clearly discernible oxide layers on the particle surfaces (Fig. 17a).

During development and as a quality-control measure, sprayed coatings should be examined metallographically in cross section to see these effects. Additional information on spray processing and microstructures can be found in the articles "Coating Structures, Properties, and Materials" and "Metallography and Image Analysis" in this Volume.

#### Process Variations

The controllable parameters for each of the major thermal process types are different, but all share some common fundamentals. Equipment and theory are covered extensively in the article "Thermal Spray Processes" in





Fig. 14 Typical coating microstructure, containing unmelted particles, oxides, porosity, and debris. (a) Unmelted particles. (b) Oxides. (c) Debris. (d) Fine particles. (e) Porosity

this Volume. It is important that the reader is familiar with various equipment theory and parameter variations in order to understand the cause-and-effect relationship between input parameters and sprayed coatings, as discussed in this section.

Converting feedstock material (whether powders, wires, or rods) into a coating requires both thermal energy for melting and gas flow for atomization and/or particle acceleration to propel the particles toward the substrate. The combination of available energy and jet velocity acting on the mass of the feedstock directly affects particle temperature, velocity, and trajectory. Table 3 shows a simple overview comparing thermal energy and particle velocity for each process. In general, powder- and wireflame spray and electric-arc-sprayed coatings are generated from high heat input and low particle velocities. These coatings tend to exhibit higher oxide contents and higher porosity. Electric arc has a stronger potential to create fumes, where powder flame has a stronger tendency to produce unmelted particles in the coating. As a result of the higher particle velocity, particles present in plasma-sprayed coatings are flattened more than in flame-sprayed coatings. The resulting coatings are also denser, with finer porosity. High-kinetic-energy processes such as detonation gun, HVOF, and cold spray produce very dense coatings with less embedded debris. In addition to the input spray parameters that control the spray pattern, particle heating and velocity, substrate temperature control, and torch-to-substrate relative motion are also critical for producing desired coatings.

#### Process Temperature Control

During spraying, it is necessary to control the temperature of both the sprayed coating and the substrate to avoid substrate degradation, oxidation, and coating failure due to differential expansion.

Substrate Degradation. As with preheating, mentioned previously, there are limitations to substrate temperature during spraying. Lowertemperature-substrate materials or matrix materials may be damaged by allowing the part to overheat during the spray process.

Oxidation. Coating layers deposited and allowed to reach high temperatures during spraying will oxidize and appear to darken. Surface oxides so formed leave a plane of weakness in the coating, between passes, and could lead to delamination under applied stress during service. The critical oxidizing temperature for each material varies.

Shrinkage/Expansion. Controlling the CTE mismatch between the coating and substrate follows the same guidelines as mentioned under the preheating section. Along with the substrate, sprayed coatings shrink as they cool. The CTE differences can exert considerable shear stress along the interface and lead to possible separation or distortion (Fig. 18). Substrate materials having a high CTE that are allowed to expand because of high temperatures during the spray process will experience catastrophic disbonding at the substrate/coating interface.

### Manipulation

Thermal spraying is a line-of-sight process. As mentioned previously, the substrate coating area should preferably be normal (at  $90^\circ$ ) to the spray stream. The fundamental objective of torch and substrate motion during spraying is to present the target area to the spray stream in a steady, consistent, and repeatable manner, always maintaining the same torch-to-substrate spray angle, standoff (spray distance), and pitch or increment. Manipulation is commonly done by moving the gun with an X-Y manipulator or robot. The part is often stationary, as with flat surfaces, or is rotated on a turntable or lathe, as in the case of cylinders. More complex



 $(b)$ 

Fig. 15 How part geometry affects coating deposition. (a) A small target has peripheral debris missing the substrate, while  $(b)$  a larger target collects all the peripheral debris and oxides.

components may require the use of coordinated motion between the robot and external axis of motion.

Manipulation Variables. Controlled relative movement of the gun with respect to the part is critical for continuous and even deposition of the coating. Critical parameters are standoff distance, surface speed, angle of impingement, and pitch or increment.

Standoff distance is the distance between the face of the gun and the part or workpiece. This distance is critical because the optimal particle temperature and velocity occur in one plane along the length of the spray stream.



Air shroud cooling

Fig. 16 Positioning of an air-cooling device to blow debris off the surface before it rotates into the center spray region and is incorporated into the coating



Fig. 17 Effect of the degree of particle melting at or just before impact on shape and coating structure. (a) Particle is heat softened or beginning to resolidify. At impact, it does not flow out and begins to lift at the edges. (b) Properly melted particle impacts and flows to form a well-bonded classical lamellar splat shape. (c) Superheated particle impacts and splashes, creating debris, satellites, and dusting.

Tolerance for standoff is a function of particle velocity. Higher-velocity processes are generally more tolerant of variation than the lowervelocity processes.

Angle of impingement has been mentioned several times in this article. Again, highervelocity processes such as HVOF are more tolerant of off-axis spraying than low-velocity processes such as conventional flame spraying. Pitch or increment, that is, the overlap

between subsequent strokes of the gun/spray stream, is important to coating uniformity.

Pitch is the term used for rotating elements such as rolls. As the roll turns, the gun must travel across the surface such that the pitch (thread) does not create a "barber pole" effect. The same holds true for planar surfaces, where the increment must be set and controlled to prevent striping. As a rule, lower-velocity processes that have wider spray patterns also have wider pitch/increment values. Conversely, high-velocity processes have narrower pitch/ increment values. Using rules-of-thumb, pitch/ increment values for HVOF and high-velocity plasma may range from 3 to 6 mm (0.13 to 0.25 in.), while flame and electric arc may be 6 to 12 mm (0.25 to 0.50 in.).

One final consideration would be the deposition rate in terms of unit thickness per pass buildup, that is,  $\mu$ m/pass or mils/pass. This parameter is controlled by the speed of the spray stream across the surface of the part, that is, surface speed. Generally, the material feed rate is held constant and manipulation is sped up or slowed down to produce the required mm/pass or mils/pass. Another rule-of-thumb is that for higher-density coatings, thinner  $\mu$ m/ pass or mils/pass layers are better; for example, plasma-sprayed alumina may be 13  $\mu$ m/pass or 0.5 mil/pass. The common baseline surface speed seems to be approximately 500 mm/s (20 in./s) but varies widely about that point. Consider the spray pattern shown in Fig. 13 and then refer to Fig. 19.

## Process Efficiency

Process efficiency (PE) is the product of target efficiency and deposit efficiency. Target efficiency (TE) is the ratio of the spray pattern envelope to the target area on the substrate. Deposition efficiency (DE) is a ratio of the spray material that impacts the substrate to the spray material that actually adheres to the substrate. TE  $\times$  DE = PE. For example, if the spray stream impinges the prepared surface of the part half of the time, the TE would be 50%. If only half of the sprayed material that impinges the prepared area adheres to the prepared surface, then the DE is also 50%. The PE is therefore  $0.50 \times 0.50 = 0.25$ , or 25%.

Measurement of TE is a geometric calculation, based on the physical dimensions of the prepared coating area and the total working envelope of the robotic/manipulator spray pattern. Deposition efficiency, on the other hand, is a measurement made for a specific gun/parameters set, with a fixed feedstock material, a given set of manipulation variables, and controlled, known substrate temperature.

Deposit efficiency can be measured easily. One simple way to measure DE is to use a flat plate with a known width, for example, a 150 mm (6 in.) square that is 3 mm (0.013 in.) thick. After properly preparing the surface, set the manipulation for the appropriate standoff, increment, surface speed, and normal angle of impingent. Stay within the vertical





(a) Particle speed ranges from a high of approximately 1000 m/s (3000 ft/s) to a low of 25 m/s (80 ft/s). Further variations within each process depend on the particle size, material type, and gas velocity.



Fig. 18 Effect of coating shrinkage on interfacial shear stresses. The sprayed metal cools, creating a tensile stress in the coating and a compressive stress in the underlying substrate material. These stresses may deform the substrate or weaken the bond between the sprayed coating and the substrate, leading to debonding of the coating. The bottom illustration shows deformation of a thin-section substrate and debonding of the coating due to stresses generated by cooling of the sprayed metal. Adapted from Ref 2

limits of the plate, but program the manipulation to extend beyond the horizontal limits of the plate by 100 mm (4 in.). Use air cooling to keep the plate temperature below  $65^{\circ}$ C  $(150 \ ^{\circ} \text{F}).$ 

With a known surface speed, width of the plate, weight of the plate, number of strokes across the plate, and material feed rate, one can calculate the mass buildup on the plate per unit time impinging the plate, against the feed rate.

To carry the example a step further:

- 150 mm plate width <br>• 500 mm/s traverse rs
- 500 mm/s traverse rate<br>• 100 strokes across and
- 100 strokes across and off the plate
- 60 g/min feed rate
- 500 g initial plate weight
- 520 g final plate weight

 $(150 \text{ mm} \times 100 \text{ strokes})/500 \text{ mm/s} = 0.5 \text{ min on part}$ 



 $(520 \text{ g final} - 500 \text{ g initial})/30 \text{ g} = 0.667$ , or 67% DE

Note that the substrate temperature is critical. The DE is directly related to temperature. Allowing the temperature to rise will produce false-high DEs.



 $x =$  diameter of spray pattern



Fig. 19 How to obtain an even coating layer by indexing the torch after each pass. See text for details.

## Statistical Design Techniques

As noted earlier in this article, many variables/parameters require study before a coating can be optimized. Statistical design techniques reduce the number of experimental runs or parameter iterations needed for coating optimization. The results obtained are often unambiguous and at a minimum cost. Statistical design methods encourage the systematic study of the many variables that confront coatings engineers. Furthermore, these studies provide data on how variables interact and how they influence coating properties, as well as a plan

## 88 / Thermal Spray Processes and Coatings



for constructively changing input parameters to affect the most desirable properties.

Designs of experiments (Ref 3) and Taguchi factorial experiments (Ref 4) are two commonly used statistical techniques. The fundamental approach for each is the same: A matrix of selected variables versus a series of experimental runs is established. Table 4 is a Taguchi L-8 fractional factorial matrix taken from Ref 5. Nerz et al. (Ref 5) chose six variables represented by high and low values. The number of variables chosen determines the matrix size and number of runs. Typically, a fractional factorial matrix is constructed with variables deemed most likely to influence coating results, rather than a complete factorial of all variables.

The matrix shown in Table 4 includes a maximum of six variables and eight experimental runs. Selected coating properties are measured for each run. To understand the magnitude of variable influence on coating properties, an analysis of variance (ANOVA) is performed. An ANOVA is a Taguchi analysis of results showing how each variable, both high and low, influences results. Another method for evaluating factorial experiments is to use analysis of means, developed by Ott using the Null hypothesis (Ref 6). This method is a statistical approach employing graphics and statistical theory.

## Postcoating Processing

Most thermal spray coatings require additional processing after the coating is deposited. These include sealing, heat treatment, and finishing. Postcoating processing is described in detail in the article "Postcoating Operations" in the "Pre- and Postcoating Processing" Division of this Volume.

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