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Materials Production for Thermal Spray Processes

A lesson from THERMAL SPRAY TECHNOLOGY

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Course 51
Lesson, Test 4

MATERIALS ENGINEERING INSTITUTE

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The aim of this lesson is to describe the common materials that are used in thermal spray processes. The manufacture and processing of materials prior to the thermal spraying operation will be covered. An understanding of the powder manufacture will enable the technological limits of the process to be defined.

Thermally sprayed coatings have a large range of applications: to combat corrosive environments, to prevent high-temperature oxidation, to minimize adverse wear, or to confer specific electrical insulation and conduction characteristics on a bulk material. The definition of ***ceramic*** will be taken in its most broad context and may include classical ceramics such as alumina, high-tech materials such as zirconia alloys, and the refractory materials based on carbides.

The coating applications are a direct consequence of their ability to retain some intrinsic characteristics of the feedstock material. For example, aluminum, zinc and aluminum-zinc alloys are used for corrosion protection, and tungsten carbide and cobalt mixtures are used for wear-resistance applications. It must be emphasized though that the thermal spray coating need not necessarily retain the intrinsic characteristics of the feedstock since this process may modify the attributes of the material. Thus, the structure of a coating is quite dissimilar to that of a material which is produced by a bulk fabrication process. The material properties of the coating that may limit its utility include the mechanical, physical, thermal and/or electrical properties. An additional property of great interest is the adhesion property of the coating to the substrate since it is crucial for the coating to remain attached throughout its service life.

It can be seen that the properties of the coating rely on the starting material used in the thermal spraying process. Materials from different manufacturers which have the same nominal chemical composition and physical shape (whether it be in powder, rod wire or composite form) may exhibit quite different material and physical properties and therefore have different service performances, even when deposited under identical conditions.

The differences in the feedstock materials mentioned above may be factors such as the production route, the particle size distribution or the particle morphology. These characteristics influence not only the thermal spray parameters but also the coating performance. The range of industries that presently take advantage of thermal spray coatings includes the aerospace, automotive and biomedical industries, the military sector, powder production, steel making, off-shore engineering, nuclear and power-generation industries, other heavy industries (papermaking, mining, textiles, printing), and engineering maintenance.

The present focus is to describe the manufacturing methods of feedstock materials for thermal spraying. The prime area of interest will be on powdered materials since these have a wide diversity and utility. Feedstock materials in the form of wire or rod will not be mentioned since the feedstock morphology is not highly variable and the production route is not unique to thermal spray technology. An important aspect of the discussion is to highlight how different powder characteristics can be controlled in order to retain some specific material property. Thus, to a certain degree, the powder processor has the ability to "tailor" the feedstock materials to produce a desired coating property. Testing procedures that enable determination of the feedstock particle size are essential for this discussion since these measurements often determine the classifications and utility of the initial process material.

Terminology

The generic term for the starting material in a thermal spraying process is ***feedstock.*** The processing of raw materials into feedstock powders which may be suitable for thermal spraying involves a large range of manufacturing and classification procedures. The technical terms which are used in the powder processing industry are listed in the AWS *Thermal Spraying* Glossary (this is included with your course materials). The Glossary summarizes the prime terms used in this lesson. It should be noted that there may be some slight differences between North American and European terminology, especially with respect to the thermal spray jargon.

It is important that the reader of this lesson becomes familiar with what may be new terms. Those who are familiar with this area are also advised to reinforce their knowledge by going through the Glossary since these definitions will be used in the following text. For example, the term "alloy" will be used to describe combinations of metallic materials or combinations of ceramic materials.

Upon completion of this lesson, the student will be able to:

- Understand the basic processes by which powders for thermal spraying are produced;
- Learn that the various production routes for powders may have attributes and deficiencies that are, in turn, related to individual powder chemistries and thermal spray processes;
- Appreciate that quality control and specifications for powders are essential;
- Comprehend that the term *particle size* should always be related to the technique by which the measurement was performed; and
- Understand that the powder feeding problem is the root-cause for coating variability and poor material properties.

The Powder Feeding Problem

A fundamental requirement of any thermal spraying operation is to transport the feedstock to the heat source and then further direct it towards the substrate. Special attention is now drawn to one aspect of thermal spraying terminology that is commonly known as the powder feeding problem. This is a technological aspect of thermal spraying that is incorporated within the phrase "the sprayability of the feedstock." Powder feeding often refers only to the initial stage of powder transportation, i.e., from the powder hopper to the thermal spraying device. However, it should be emphasized that the manifestation of this technical difficulty may be coatings of poor quality, regardless of the fact that powder seems to be flowing into the heat source. This aspect of thermal spray technology is the root-cause for examining the production and physical aspects of powder preparation. Therefore it is worthwhile to examine, briefly, the types of powder feed delivery systems and requirements which constitute good powder feeding. Thus, a specific powder may not have identical feeding characteristics with different powder feeding devices.

Powder Feeding Systems

Figure 1 illustrates the six primary types of powder feeders. They are classified under the following headings:

(a) A gravity-based device whereby the powder falls into the thermal source. Some vibratory action may be supplied to shake the powder into the gas stream and prevent any blockages from occurring.

(b) A rotating wheel which delivers precise parcels of powder to the powder feed delivery tube. The wheel can be mounted in either the vertical or horizontal directions.

(c) A fluidized bed-container system whereby the feedstock is gas-suspended within the hopper. The powder feed rate is determined by the venturi effect that is created when the carrier gas passes across an aperture within a "powder port" that is placed in the hopper.

(d) A vibrating powder-feed device. The powder mass is vibrated and passes up a spiral channel that ends with the powder moving into the feed gas stream.

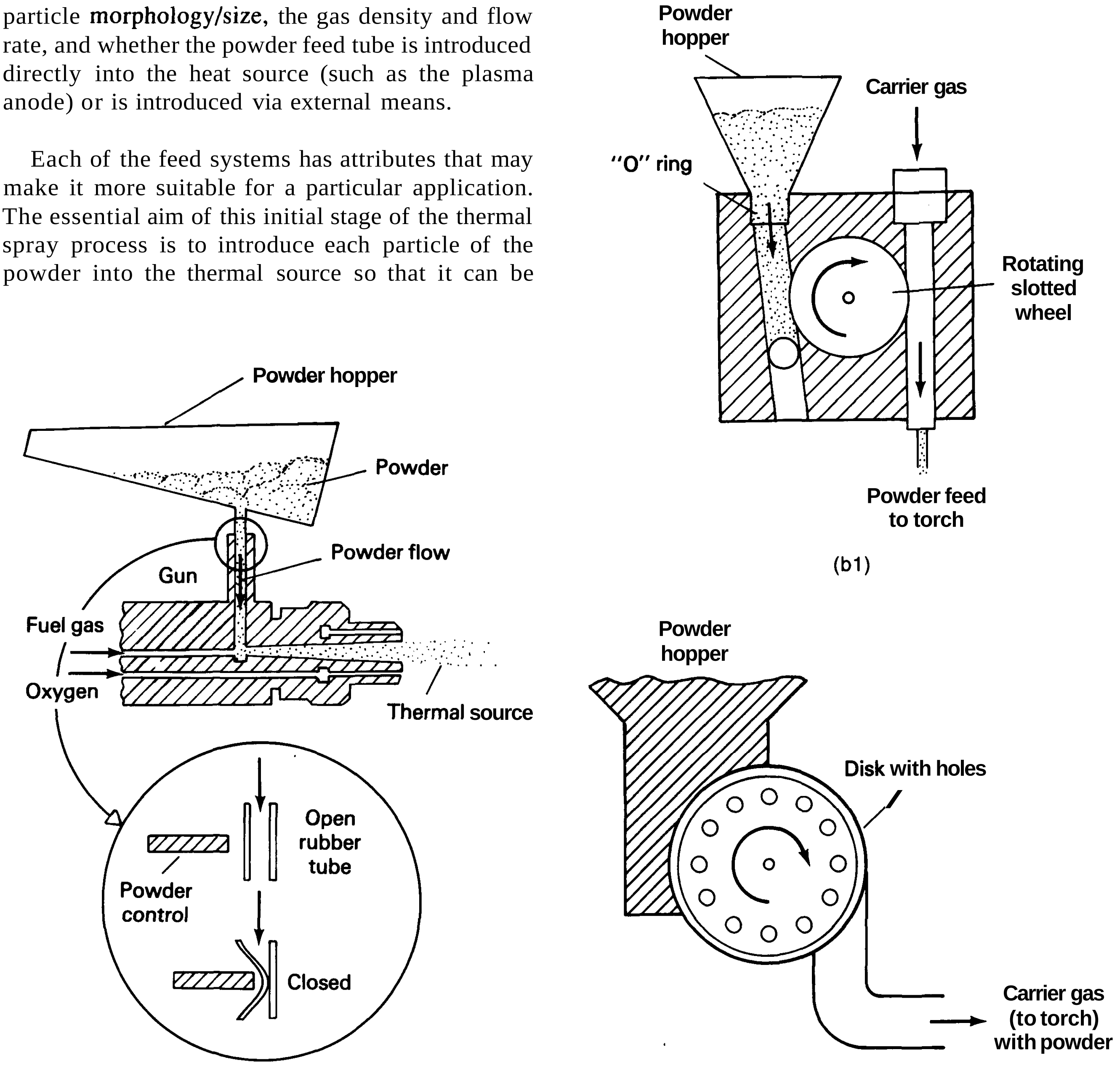
(e) A delivery device based on an Archimedes spiral arrangement which shifts the powder into the gas transport stream.

(f) Powder may be encapsulated in well-defined mass lots (or shots) within a role of paper tape.

The tape is fed through the spray torch so that the contents of each powder capsule are punched into the thermal source.

The net effect of the powder feed device should be to carry feedstock at a consistent, smooth, non-pulsating and reproducible rate, into a gas stream which, in turn, transports the powder to the thermal source. The prime variables that influence the quality of powder feeding are the powder density, the particle morphology/size, the gas density and flow rate, and whether the powder feed tube is introduced directly into the heat source (such as the plasma anode) or is introduced via external means.

Each of the feed systems has attributes that may make it more suitable for a particular application. The essential aim of this initial stage of the thermal spray process is to introduce each particle of the powder into the thermal source so that it can be heated up and then accelerated towards the substrate under sufficient thermo-kinetic conditions that enable spreading of the particle on impact against the substrate. The possible particle trajectories within the thermal source are diagramed in Figure 2. The essence of the powder feeding problem can now be described in terms of each particle following the correct path.



(a)

(b1)

(b2)

Figure 1. Powder feeding systems: (a) gravity- and vibration based-system, (b) rotating-wheel system and rotating-disc system, (c) fluidized-bed feeder, (d) vibrating spiral feeder, (e) Archimedes spiral feeder, (f) encapsulated shots. Continued on the following page.

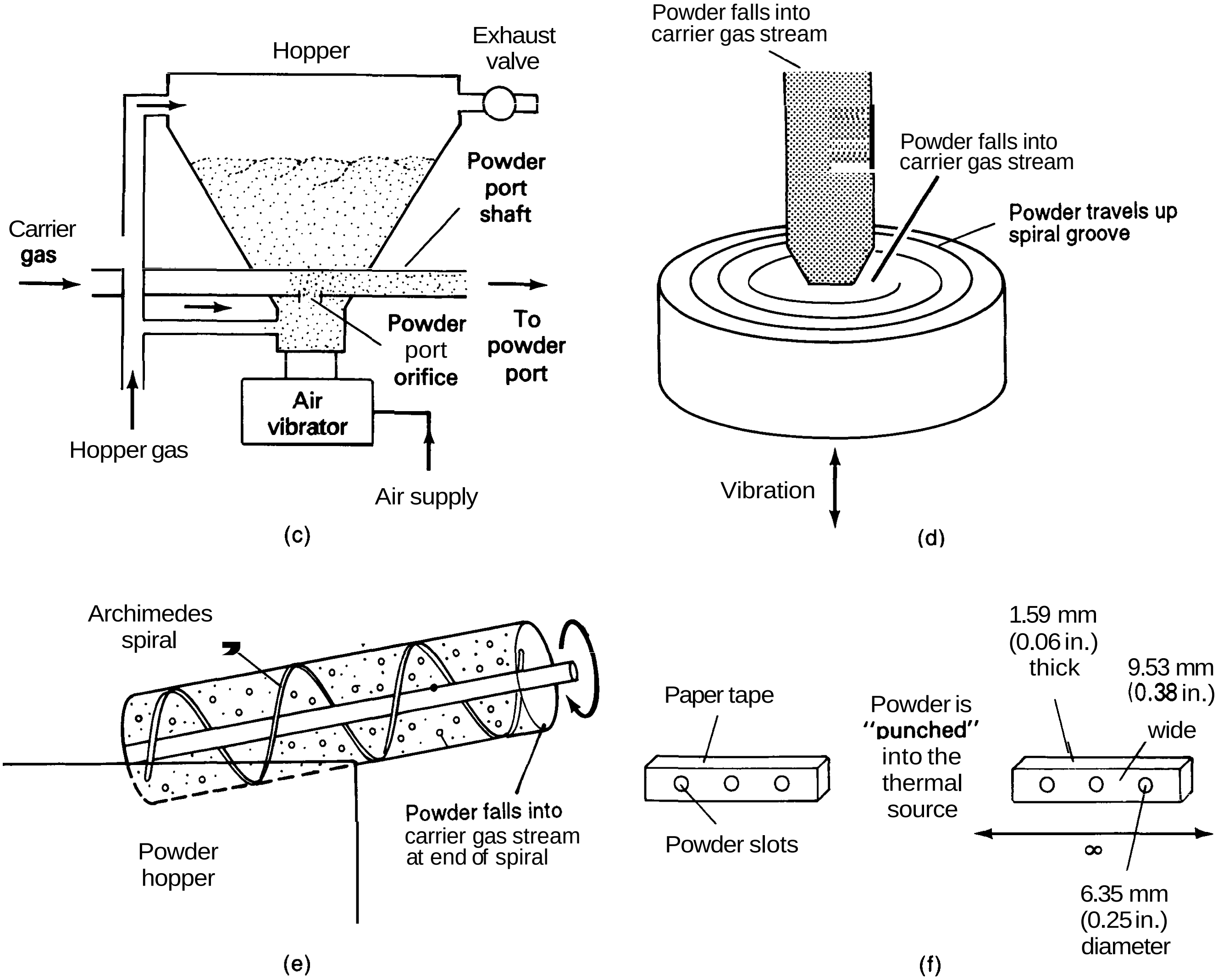


Figure 1. (continued) Powder feeding systems: (c) fluidized-bed feeder, (d) vibrating spiral feeder, (e) Archimedes spiral feeder, (f) encapsulated shots.

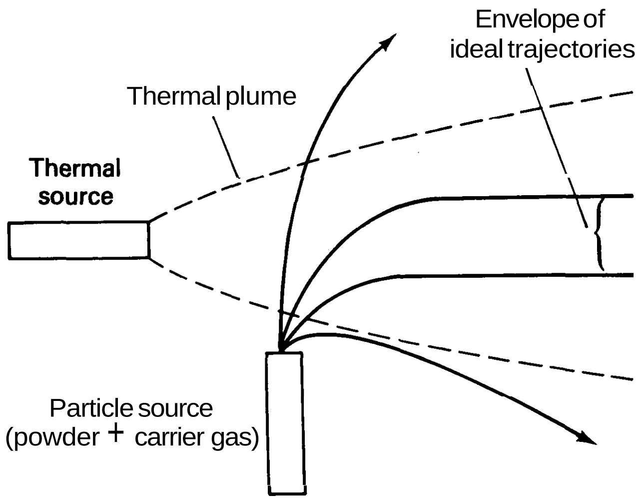


Figure 2. Particle trajectories within the thermal source.

The impact of poor powder feeding cannot be underestimated since it limits the utility, productivity and efficiency of the thermal spray process. Observations such as irregular or pulsating feed rates, powders which do not flow within their container, agglomerates of melted powder at the anode throat, and low deposition efficiencies all indicate process parameter difficulties that may be traced back to the feedstock. The ideal powder is thought to be a mono-sized and spherical particle that follows an ideal path through the thermal source. Each particle becomes fully melted and in the plastic condition so that it flows on the substrate surface and forms a coherent and dense deposit. Such powders are not commercially available for all materials.

Material Categories for Thermally Sprayed Coatings

This section establishes an understanding for choosing particular feedstock materials. Figure **3** is a diagrammatic representation of relationships between the material chemistry, material morphology, **size** distribution, thermal spraying process and the intended application. It is intended to demonstrate that the initial chemistry and form of the feedstock material is of utmost importance with regard to the engineering application, as indicated by the dashed line on Figure 3 which shows the link between these two aspects of thermal spraying. The following discussion also indicates that powder processing and powder quality can be considered from many viewpoints.

Classification According to Thermal Spray Process

- Oxyacetylene gas flame spraying (flame spraying, combustion spraying)
- Atmospheric plasma spraying (APS)
- Low-pressure plasma spraying (LPPS, VPS)
- Wire-plasma arc
- Detonation gun spraying (D-Gun)
- Ceramic rod process (Rokide process)
- High-velocity oxyfuel process (HVOF, Jet Kote, DJ Gun, and others)
- Fuel-air repetitive explosion process (FARE **gun**)
- Wire explosion process
- Arc metallization
- Electromagnetic coalesce process (EMC)
- Wire spraying
- Liquid-stabilized plasma torch.

The nature of the particular thermal spray process is directly related to the types of materials that can be sprayed by that process. For example, arc metallization requires that the feedstock be in the form of electrically conducting wires. It is clear that the material cannot be an insulating ceramic or polymer; however, it may not be as obvious that **non-conducting** materials can be inserted into conducting tubes so that arc metallization procedures can be utilized. The

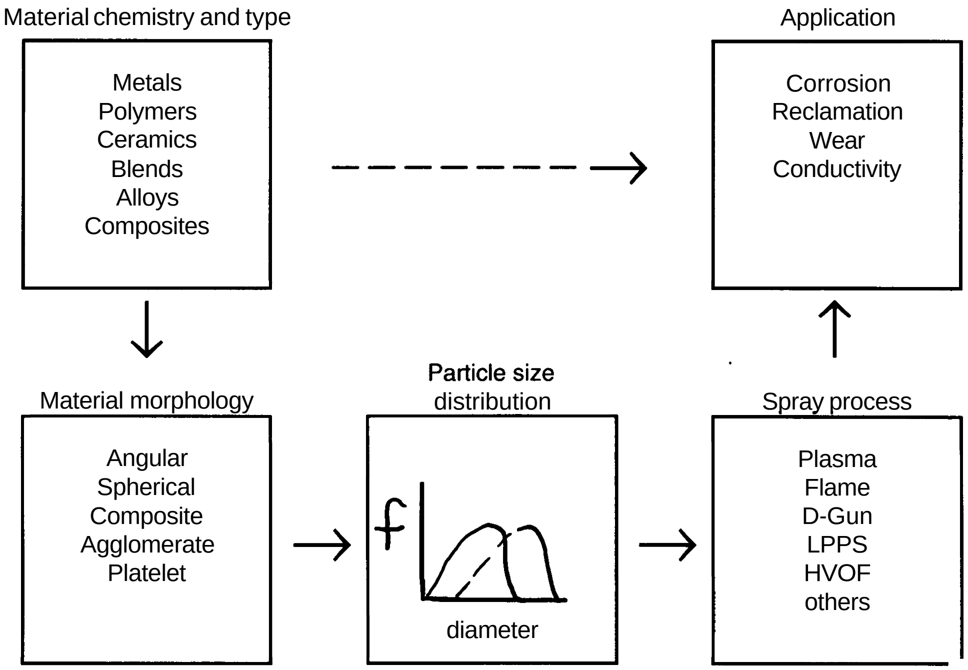


Figure 3. Relationships between material chemistry and the intended application.

powder feed can also be controlled by incorporating the powder into discrete lots that are individually added into the thermal spray stream, as indicated in Figure 1(f).

The temperature and velocity of the process are matched to the feedstock characteristics. Processes such as flame spraying have relatively low temperatures and velocities and therefore may not melt high-melting-point ceramics. One way of overcoming this type of problem would be to decrease the average particle size; however, this creates another range of difficulties that are associated with reliable feeding of material. This conflict in melting ability of the feedstock with respect to process conditions can be resolved by changing to a thermal spray technique that employs a more intense heat source, such as plasma spraying, high-velocity oxygen fuel or the detonation gun. Thus, a measure of the characteristics of a certain feedstock can be gaged from examining its spraying performance under certain processes.

One other process of unique importance is low-pressure plasma spraying (LPPS) since it allows the processing of reactive metals such as titanium and nickel alloys in an inert environment. These metal alloy materials would oxidize under ambient spraying conditions and the oxide boundaries formed result in poor high-temperature performance of such coatings. The LPPS process has, on the other hand, no particular advantage for the spraying of ceramic materials since they do not chemically degrade during spraying. Thus, the point being made is that certain behavior of a material during thermal spraying can be controlled by altering the process. Thermal interactions of the superheated feedstock particles with the environment can be controlled by flooding the working area with an inert gas such as argon or nitrogen. This is called a ***shrouded gas environment.***

The topic of thermal spray process also includes a set of variables that are collectively known as the thermal spraying parameters, or the ***spray parameters*** in thermal spray jargon. A summary of thermal spray parameters is shown below:

- Powder specifications — chemistry, size distribution, morphology, and density;
- Powder feed rate;
- Thermal spray gas pressures and consumption rates;
- Deposition efficiency;
- Torch-to-substrate distance (stand-off distance);
- Equipment specification;
- Substrate surface preparation; and
- Coating thickness(es).

It is essential to point out that even with similar thermal spraying processes, these parameters are likely to change from process to process and among different equipment. Powders which are presumably identical may also have different spray parameters for each set of equipment.

Classification According to Material Morphology

The morphologies of the feedstock materials are depicted in Figure 4. The material morphology refers to the shape of the individual particles, and this characteristic is commonly determined via optical or scanning electron microscopy (SEM). The morphology is directly related to the thermal spraying process, as indicated in the figure, and usually the prime process variables are the thermal spray parameters.

The particle shape is controlled by the production process. It is incorrect to assume that the powder surface is smooth or that each particle is chemically homogeneous and 100% dense. The particle shape determines the material transport from the powder hopper to the plasma torch. Thus, this characteristic is an essential quality to be determined. The particle size distribution is also an important parameter to determine since this is optimized with regard to the coating quality. It is generally agreed that a closely sized distribution is required to take advantage of the few optimum particle trajectories through the plasma flame in order to manufacture a dense coating. The ideal morphology is spherical, although needles and platelets are of research interest since they may confer special properties to the coating. The section on Particle Morphology and Porosity will show powder morphologies of some powders.

Classification According to Chemical Nature of the Material

The broad classes of materials used are:

- Metals and alloys: aluminum, aluminum-zinc, copper, molybdenum, nickel-aluminum alloys, nickel-chromium alloys (e.g., NiCrAlY, Ni-Al);
- Ceramics: aluminum, chromium carbides, chromium oxide (chromia), hydroxyapatite, mullite, spinel, titanium oxide (titania, rutile), tungsten carbide-cobalt, yttria-stabilized zirconia, zirconium oxide (zirconia);
- Composites: aluminum/silicon/polyester composites, molybdenum-nickel-chromium-boron-silicon, nickel-graphite, bentonite-NiCrAl; and
- Polymers: nylon, polyesters, polyamides, polyethylenes.

This classification is most commonly used by thermal spray applicators since the material chemistry is related to the coating application. The knowledge of this chemistry allows close matching to specific properties of the substrate. For example, reclamation coatings may require feedstock of similar chemistry so that the initial component characteristics are retained. On the other hand, protective coatings can be manufactured which consist of a material dissimilar to the base material. Thus, materials are often classified as ceramics when an abrasive or thermal barrier coating is required, or as a polymer for chemical resistance or low friction.

The classification of composite has a special meaning in thermal spray technology since it refers to powders that consist of 2 or more distinct chemi-

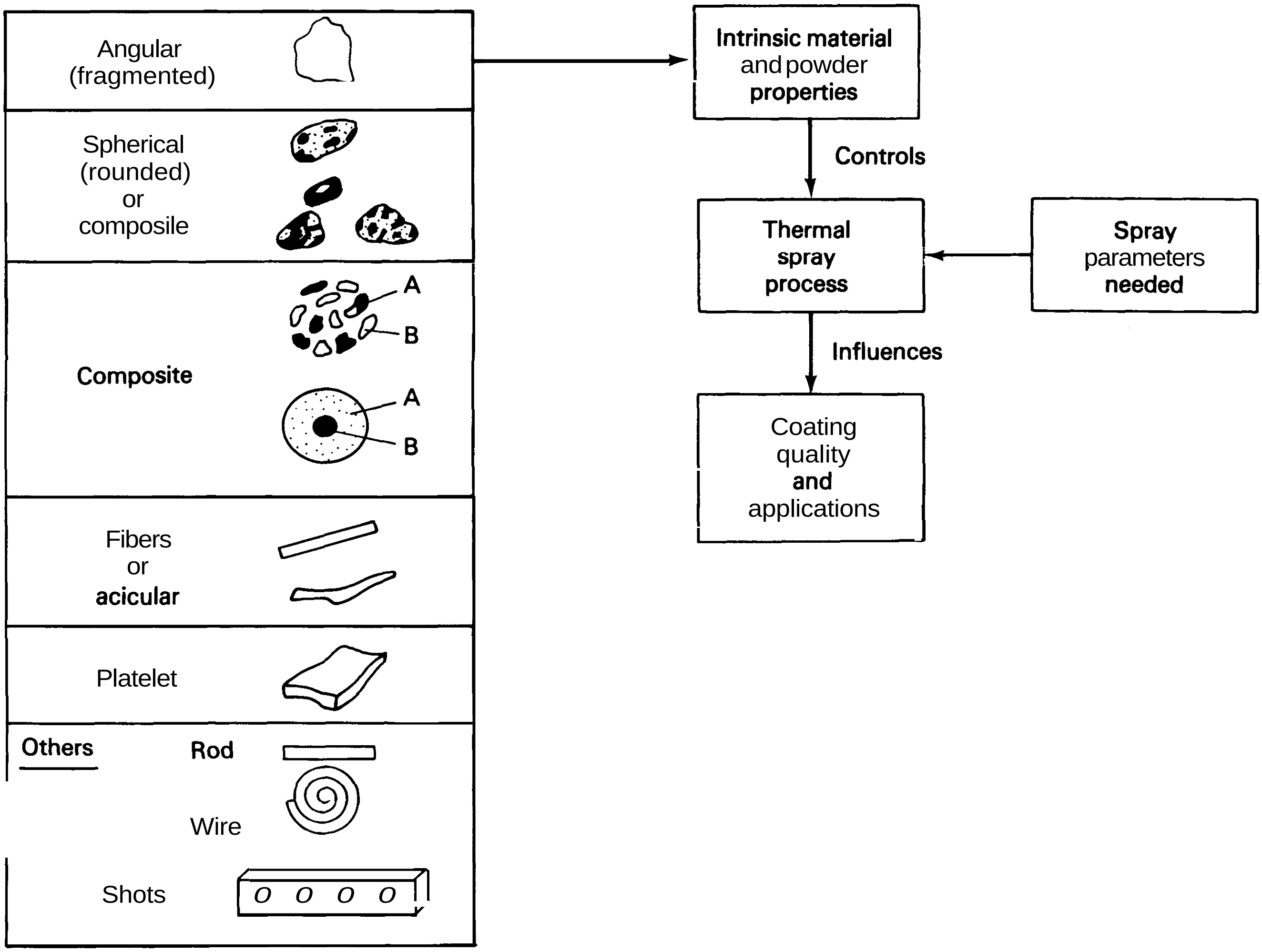


Figure 4. Feedstock morphology.

cal species. The composite structure can be in the form of an aggregate or of two (or more) distinctly dual phase regions. Every particle of the material exhibits this inhomogeneous structure. The mechanical combination of different powders is referred to as a *blended* material. The terms *composite structure* or *composite coating* are reserved for coatings that consist of two or more distinct morphological features, such as spherical particles in a fully melted coating or needle-shaped artifacts in a coating. *Graded coatings* are also often described as being composites, although this description is not entirely correct. These coatings consist of two (or more) different materials that have been blended together in a graduated fashion so that the composition changes from a majority of one species at the substrate to a majority of the other species at the top part of the coating. The material structure is probably more correctly described as being a cermet, since the two prime blending constituents are usually a metal and a ceramic.

Classification According to Applications

The preceding sections of the lesson infer that coating technology often incorporates a coating system which consists of a number of separate constituents. For example, a bond coat is deposited prior to the ceramic overlay. The utility of many materials could be quite limited without this coating design and the classification according to applications assumes that an appropriate coating system has been developed. It is often only the outermost layer that confers the intrinsic material property of interest to the component. Several application classifications are listed below.

- Hard surfaces: sizing punches, extrusion dies, pump seals, hot crushing rolls, hot forming guides;

- Control of abrasive grain wear: buffing and polishing machines, polishing rod liners;

- Adhesive wear: piston guides, thrust bearing shoes, bronze and babbitt bearings;

- Hard bearings: fuel pump rotors, impeller shafts, piston rings, armature shaft journals;

- Fretting: cylinder liners, automotive valves, rocker arms, lathe and grinder dead centers;

- Cavitation: water turbine buckets, wear rings for hydraulic turbines, impeller pump housings;

- Particle erosion: exhaust fans, cyclone dust collectors, exhaust valve seats;

- Heat and oxidation: tuyeres for liquid metal industries resistance, continuous casting molds, heat-treating fixtures and brazing jigs, exhaust mufflers;

- Atmospheric and immersion corrosion: electrical conduits, bridges, transformer cases, steam cleaning equipment, ship superstructures, ship holds and tanks, storage tanks for oils, fuels and solvents, power line hardware, heat exchangers;

- Electrical conductivity and resistivity: ground connectors, lightning arresters, solder iron tips, ground coating for locomotive axles; and

- Machine element clearance control: air seals (to replace silver coatings), compressor seals (to replace rubber), for aircraft engine components.

Material Production Processes

Thermal spray powders have evolved principally from powder metallurgy procedures. The most recent developments have arisen from ceramics and composites processing techniques. The production of polymeric powders which are suitable for thermal spraying is a relatively new engineering field. The powder production technique has a marked influence on the nature of the powder that is produced. The technology and principles of the processes that relate to thermal spraying will be described. The generic terms which describe the manufacture of powders by mechanical processes are *comminution* and *attrition.*

Mechanical Techniques

Machining. This is an expensive process that is employed for relatively difficult-to-work materials, e.g., magnesium, copper and aluminum alloys, and noble materials such as gold, silver and platinum. A rod of the cast material is typically lathe cut and the machine fragments collected. Often the process is performed under an inert atmosphere so that any spontaneous combustion of the product (e.g., magnesium) is kept to a minimum. There is little call for this method to produce thermal spray powders.

Crushing and Milling. The purpose of crushing is to break up large particles into a smaller size fraction by using mechanical energy. These processes are used mainly for ceramics since metals would be plastically deformed, not broken up into smaller particles. High-purity stock can be made by melting the raw materials in an electric furnace.

Ceramics and some metals can be reduced in size by mechanical impact and deformation in processes that are termed *attrition.* Crushing processes employ equipment such as hammer mills, stamping mills, jaw crushers and gyratory crushers. The crushing stage is often followed by a more refined technique that enables control of the particle size, i.e., milling. Milling involves the disintegration of brittle, friable materials (some metals and most ceramics), or the pulverization of malleable metals. The particles so-formed often have an irregular shape and are of variable particle size which may be less than 5 microns. All of these powders are classified so that any fines can be post-processed by agglomeration to form a suitable thermal spray powder.

Milling machines include rod mills and ball mills. Care must be taken to ensure that the grinding media does not unduly contaminate the material that is being reduced in size. The milling media is usually of high specific gravity. For example, alumina, steel, zirconia and mullite are common although cemented carbides are sometimes used where contamination must be kept to a minimum. The use of higher-density grinding media gives rise to a higher grinding rate since the impact during tumbling is greater. The mill liners are generally vulcanized rubber, polyurethane, high-density alumina, porcelain, tungsten carbide (for laboratory mills), or stainless steel.

Rod mills grind large particles (greater than 15 microns) more efficiently than ball mills. Ball mills grind all sized particles to the same degree and hence a larger variability in size distribution occurs. Ball mills are filled to about 50% of the total volume with the grinding media. The powder charge constitutes 25% of the total mill volume for dry ball milling and is generally 30 to 40% of the total volume for wet ball milling operations. A small amount (about 1 wt%) of grinding aid such as stearic acid or foleic acid is added to prevent caking of the powder charge. In wet ball milling the charge is suspended in an inert liquid such as alcohol, acetone or water. High-viscosity suspensions usually give rise to low grinding rates while the other extreme of low solids content increases the wear rate on the mill lining and grinding media.

The speed of the rod or ball mill is quite critical with regards to the life of the equipment and the efficiency of the process (see Figure 5). It is necessary for the balls (or rods) to drop from the top of the mill onto the material that is being ground (see Figure 5b). If the mill speed is too fast then the media will either not fall at all due to centrifugal forces or it will fall directly onto the media near the bottom of the mill and accelerate the media wear due to chip-

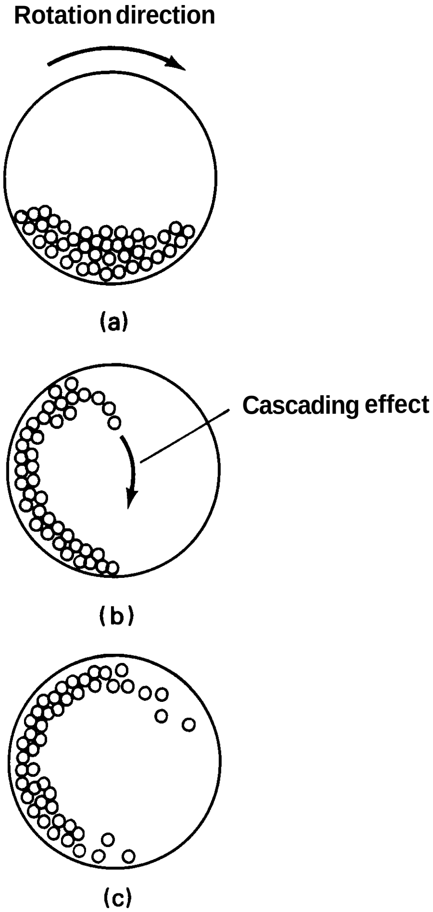


Figure 5. Comminution variables that control particle grinding and efficiency: (a)low speed,(b)optimum speed, and (c)high speed. The balls or rods within the mill are indicated.

ping (Figure 5c). At low speeds the media does not drop at all, whereas at the optimum speed the media continuously "cascades" onto the material that is being crushed.

The newly formed surfaces of pulverized material are highly active and therefore the particles may tend to cluster into agglomerates. This decreases the efficiency of the attrition process. Organic additives such as alcohols are added in amounts of less than 0.1 wt% to reduce agglomeration and thereby improve the grinding efficiency. The active surfaces of the newly formed particles may also cause enhanced reactions with the environment or activate any post-processing operation such as sintering.

The problems of mill wear have been partially overcome with fluid energy and shear mills. The operating principal of these devices is approximately the same. The material is incorporated into either a gas or a fluid stream and then these two streams of the fluidized material are forced to be coincident (Figure 6). The individual particles collide and abrade each other. The grinding efficiency of these mills is quite high and there is minimum contamination since there is little high-velocity contact with the walls of the mill. The major problem with this type of mill is that it is necessary to separate a fine particle from the fluidizing gas or liquid. The required filtering system adds to the complexity of the milling operation.

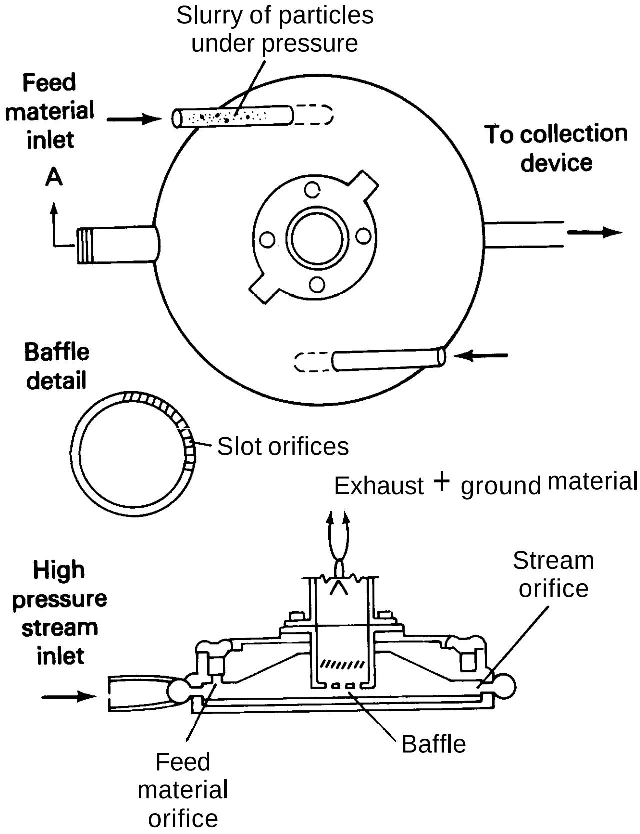


Figure 6. Fluid energy mill.

The purposes of milling are many: to reduce the size of particles or agglomerates, to eliminate particle segregation or preferred orientation in single-phase particles or, in multi-component powder systems, to homogeneously disperse the many components. Three basic stages are followed during the milling process: an initial rapid reduction in the size of aggregates, fracture of individual particles and, eventually, reagglomeration of fine particles after extended milling. The initial aggregates may be of size about 10 to 15 microns and these will be reduced to fines of the order of 0.1 micron.

Atomization. Atomization includes the four categories of gas, water, centrifugal and mechanical processes. Only the gas and water techniques are extensively used for the production of thermal spray powders. Atomization is extensively used for the production of metallic powders. The principles of the gas and water processes are quite similar in that a continuous stream of liquid metal is broken down into droplets by the impingement of a gas or water stream. A variety of process parameters allow the particle morphology and size distribution to be varied. A schematic of an atomization facility is shown in Figure 7. The principle components are:

- A melting and a dispensing crucible (or tundish);
- An atomizer and associated control system;
- The water-cooled spray chamber;
- A cyclone separator or a settling tank;
- A system for the storage and supply of gas or water;
- A cooling section for the effluent; and
- Powder collection devices.

Water atomization produces an irregularly

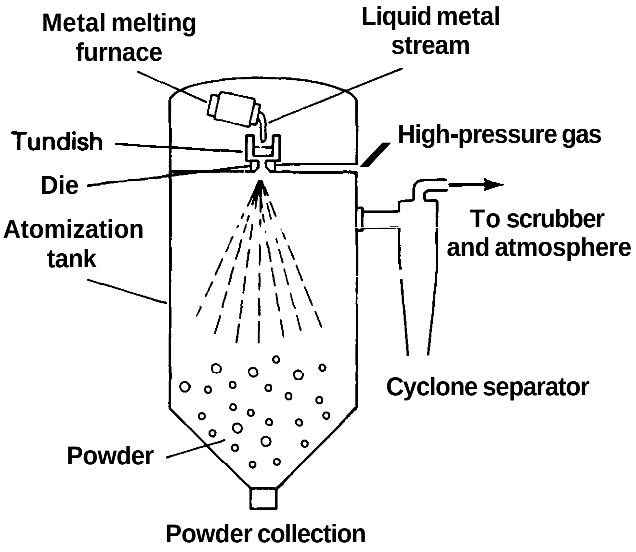


Figure 7. An overall view of an atomization facility.

shaped particle, although a more spherical powder can be obtained by giving the melt a large degree of superheat (i.e., a high temperature above the material melting point). Water atomization forms a large part of powder production techniques, primarily because it lends itself to high production rates. These powders usually require de-watering, drying and milling after production. If the high oxygen content of these powders is unacceptable, then a reduction stage is necessary. It can be noted that the high oxygen content of nickel- and cobalt-based powders produced by water atomization is not overly detrimental for thermal spray applications. Water-atomized powders have mean particle sizes in the range of 75 to 100 microns.

Gas atomization can be carried out with air, steam, nitrogen, argon or helium. The powder can be extremely clean (i.e., about 100 ppm of oxygen), especially for those materials produced under inert gas conditions. It is the only viable method of production for reactive materials such as titanium or titanium alloys and other reactive alloy systems. Gas-atomized products are typically spherical and have smaller-sized particles than those produced by water atomization (typically in the range of 25 to 100 microns). Powders produced by this technique include CoCr-based alloys, NiCr-based alloys, Ni-based alloys, Cu-based alloys, zinc, aluminum, stainless steels (all from 38 to 150 microns in size), and Ni-based superalloys and MCrAlY's (in sizes less than 75 microns).

The prime advantages of gas atomization over water atomization are the enhancement of a spherical morphology and the ability to produce a clean structure (i.e., the lamellae of coatings produced from these powders exhibit oxide-free surfaces and low oxide and gas contamination). These two factors influence, respectively, the powder transport and flowing characteristics of the powder, and the formation of oxide-free metallic bond coats. The material structures formed from these techniques are typical of those produced by rapid solidification processing. Water atomization results in particles that are cooled at a rate of 10^4 to 10^6 Cs^{-1}, whereas gas atomization cooling rates are an order of magnitude lower, i.e., 10^3 to 10^5 Cs^{-1}. Both processes rely on convective cooling in the liquid or gas and the cooling rate is therefore dependent on the individual particle size. Thus, cooling rates can be as high as 10^7 Cs^{-1} for 10 micron sized particles which are cooled by high thermal conductivity gases such as helium. This range of cooling rates (from 10^3 to 10^7 Cs^{-1}) is within the same range for the thermal spraying process, so the coating crystal structure may be quite similar to that of the as-received powders. The crystal structure for the milled and crushed powders is crystalline, these feedstock materials exhibit a marked structural change during thermal spraying since they melt and then are subjected to rapid solidification.

The melt size of each metal heat may vary from 20 to 500 kg (44 to 1100 lb.). The ability to rapidly induction heat the melting crucible either outside or within the atomizer ensures high purity of the metal or alloy. The typical flow rate of the metal through the atomizing process is from 20 to 60 kg min^{-1} (44 to 132 $lb./min^{-1}$); the total atomization time for the smaller specialized or laboratory runs may be less than 30 seconds. The metal runs directly from the ladle to the **tundish** of the atomizing unit. A gas atomizing unit that processes a 300 kg (135 lb.) batch of metal is typically 4.5 m (14 ft. 9 in.) in height and 1.25 m (4 ft. 1 in.) in diameter. The two basic designs of atomizers are termed as *confined* or open (equivalent terminology is closed orfree-fall, respectively). In a closed system the gas travels 1 to 10 mm (0.04 to 0.40 in.) before hitting the metal stream, whereas this distance is 30 to 150 mm (1.2 to 5.9 in.) for an open system. This distance greatly influences the manner in which the metal stream is fragmented.

There are a number of principle operating variables that influence the powder quality produced by atomization techniques. The features of the atomizer that control the product size and quality are shown in Figure 8. They are:

- Atomizing jet distance, geometry, and pressure;
- Nozzle geometry;
- Velocity of the atomizing media and the metal;
- Melt superheat (i.e., the temperature above the melting point of the metal); and
- Gas purity. This will control the purity of the atomized product. Usually inert gases of the 99.99% purity level are used.

Many of the operating conditions for an atomizer are empirically based. The essential principals are that less-coarse particles are obtained as the metallic stream is highly disrupted. Water atomization is performed at water pressures of 30 kPa to 65 MPa (4.4 psi to 9.4 ksi). The corresponding water flow rate may be near 90 kg/min (198 lb./min^{-1}) and this corresponds to a water flow rate of 40 to 150 m/s (130 to 490 ft./s). An important operating parameter is the water/metal ratio since there must be enough water to tear the metal apart. The normal ratio is 4 to 10 L/kg (2.3 to 6 gal./lb.) of metal and this provides sufficient cooling of the metal to avoid boiling and permits a range of metal flow rates to be accommodated. In gas atomization the typical gas usage rates are 0.5 to 2 m^3/kg (39 to 155 ft.^{3}/lb.) for nitrogen or air at pressures of 1.4 to 4.2 MPa (0.2 to 0.6 ksi). The gas flow rate is 50 to 150 m/s (165 to 490 ft./s).

Chemical Techniques

Sol-Gel. Sol-gel processing is a chemical engineering technique used to manufacture ceramic powders, especially oxides. The term sol refers to the initial solution of the chemical components from which the final powder will eventually be derived. ***Gel*** is a term used to describe the final product of the ceramic material.The methods are based on mixing solutions that enable reactions for the formation of distinct particles. It is important to distinguish that the particles are not precipitated from solution; rather, the mechanism of particle production is based on colloidal science in which the particles are sus-

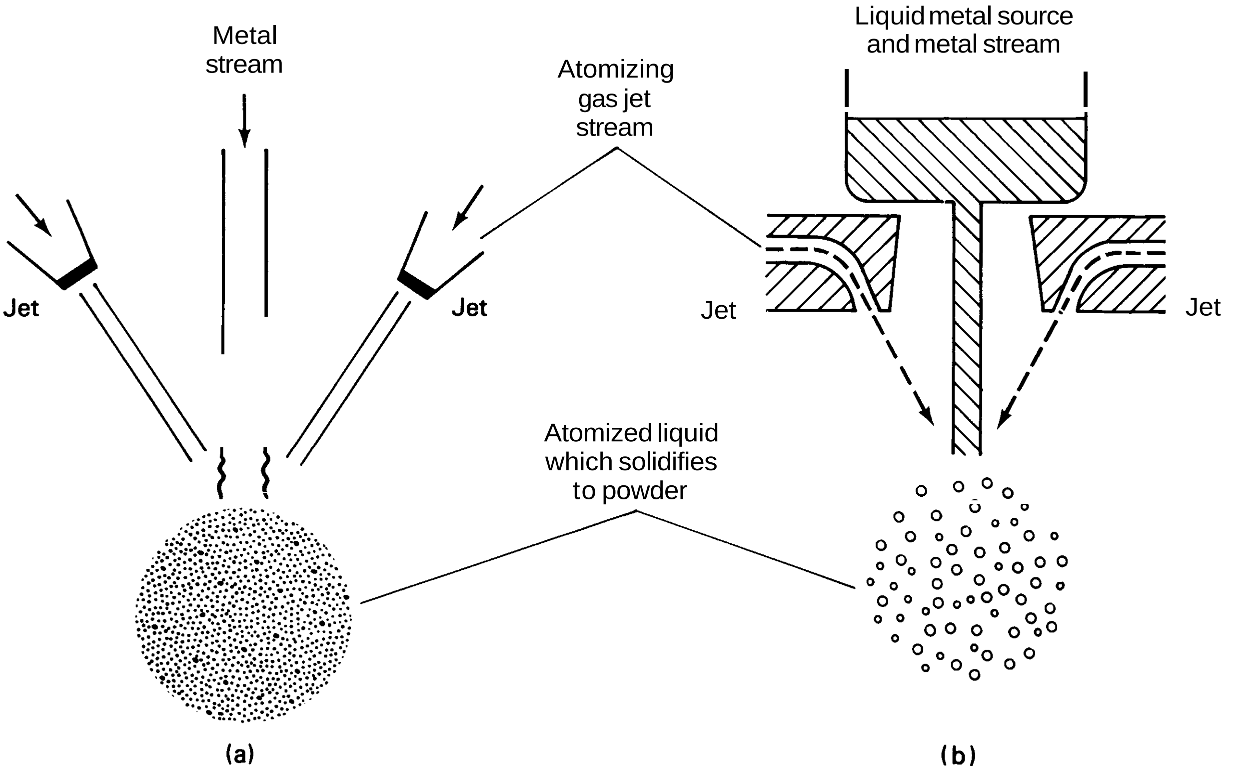


Figure 8. Prime variables that control the atomization process.

pended in the liquid. Typical ceramic powders that are produced by this technique include chromia, alumina and the stabilized zirconias.

The sol-gel technique and powders formed by this means have the following attributes:

- The technique is performed at low temperatures. The process is therefore more energy efficient than high-temperature methods that may also need particle attrition as described in the previous sections.
- Powders of a predetermined size and morphology can be produced. The techniques enable the production of high yields of a required size fraction. With reference to thermal spray powders it is important to state that spherical powders are routinely produced, since this morphological attribute enables the manufacture of free-flowing feedstock.
- The powder composition can be closely controlled; for example, multi-component powders can be produced. Also, the degree of particle aggregation can be used to control the pore structure of the product.
- The process is performed by solution chemistry methods. The technique is quite efficient compared to powder blending. Another benefit is that there is no dust hazard, thus minimizing or completely eliminating pyrophoric, toxic and environmental problems.

The steps that are followed to produce a powder via the sol-gel route are indicated in Figure 9. These steps are listed and discussed below.

Stage 1. The components are put into solution. The liquid component is usually water or an alcohol. The solute is either an inorganic nitrate chloride or based on metal-organic compounds. These components allow thorough mixing of the species on the atomic scale, explaining the extremely homogeneous compositions that are obtained. This solution is often referred to as the "precursor" since it is the basis of the following steps that lead to the final powder.

Stage 2. The above solution, if it is not already a sol, is converted to a sol. A sol is a colloidal dispersion (or a dispersed solid phase) that contains particles smaller than about 150 nm (0.15 microns or 5.9 x 10^{-8} in.). The sol can be made stable by appropriate adjustment of the pH; otherwise, the particles will grow to agglomerates that, in turn, may precipitate and thereby not enable the unique powder chemistries to be obtained.

Stage 3. The gelation step occurs next. This is essentially a step to remove most of the solvent (water or alcohol) so that a rigid body of well-defined chemistry is formed. The gel is still quite plastic or highly viscous. The processing variables are pH, temperature, and time.

Stage 4. The gel is shaped to the required morphology. This may be as spheres, fibers or coatings.

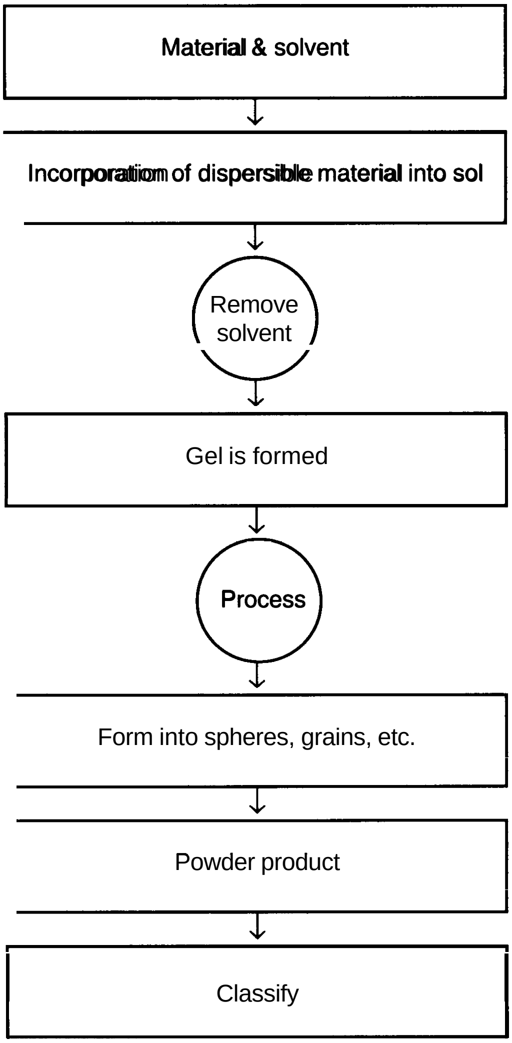


*Figure 9. The production steps for **a** sol-gel production route.*

Thermal spray powders of spherical morphology are produced by a process called ***spray drying.*** These morphologies may also be produced by controlling the **gelation** stage in the conventional process.

Stage 5. The remaining solvent is removed and the gel is calcined to form the final powder. This final temperature is significantly lower than the temperatures of conventional processing 'methods. For example, silica can be calcined at temperatures from 600 to 1000°C (1110 to 1830°F).

The sol structure can be in either an aggregated or dispersed structure so that varying degrees or particle alignment are obtained when the conversion to a gel takes place. Figure 10 is a ball-model of the structures that may be established. The density of the product can be altered from low (i.e., an aggregated sol) to high (i.e., a non-aggregated, or dispersed, sol).

Spray Drying and Spray Roasting. In this process the sol is normally concentrated by partial evaporation of the solvent to form an aero-gel. The aero-gel is then fed into an enclosed chamber heated by hot air. The process chamber is similar to that used for atomization (see Figure 7). The aero-gel is still quite fluid and forms a spray so that each droplet will, after evaporation, constitute the gel of the powder particles. The shrinkage of the individual drop may be quite high — on the order of 50% — and particles in the size range of 5 to 250 microns may be produced. These gel particles can now be calcined to form the feedstock for thermal spraying. The hot air is usually at 400°C (750°F) and the air leaving the spray drying device is at 200°C (390°F); the process is clearly not very energy efficient for small batches of material. Spray drying of powders is also referred to as ***spray roasting*** since the process temperature may be quite high.

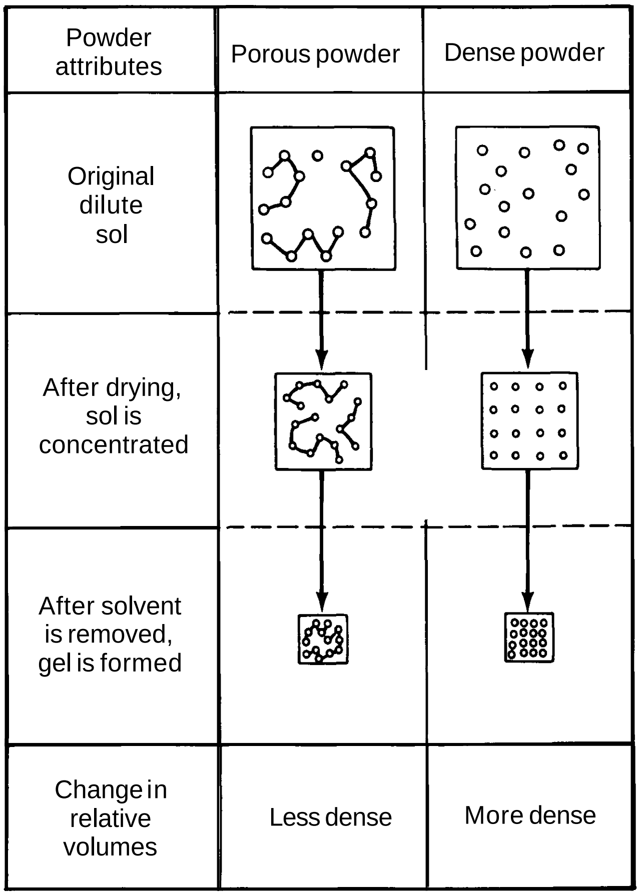


Figure 10. Formation of variable densities during the sol-gel process.

A spray drying facility can also be used to agglomerate fine powders, presently in the 1 to 10 micron range. The particles are mixed in a slurry that contains a binder. The slurry is then atomized at elevated temperatures and the various constituents form clusters of particles with the chemistry of the powders in the original slurry. The powder size is controlled by altering the nozzle geometry and the atomizing pressure. This process is not a true sol-gel method (in the scientific sense) since the chemistry of the product is inhomogeneous. The method is attractive because of its flexibility in utilizing the raw materials that are generally inexpensive, to produce free-flowing particles of a spherical morphology.

Freeze Drying. Freeze drying also uses a sol or another type of emulsion which contains the various components of the powder alloy being manufactured. The droplets of material are rapidly frozen by stirring into hexane at -30°C (-86°F). The material is now formed into solid phase that can be filtered out from the system. The final step is to evaporate any solvent by causing it to sublimate (i.e., the solid-to-gas transformation which bypasses the liquid state) at low pressures and ambient temperatures.

Agglomeration and Sintering. Agglomeration refers to the binding together of particles to form a coherent body. Two methods are used: those that use a binder and those that rely on a sintering operation. These methods also overlap with the sol-gel techniques that have been described in the previous section.

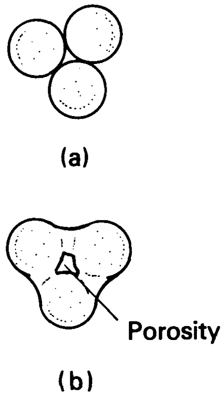


Figure 11. Sintering of particles.

The term ***sintering*** refers to the joining of particles through the combined actions of pressure and heating. The pressed material is heated to below its melting point and binding between particles occurs due to chemical diffusion between the particles. The particles become joined to each other and further comminution and sieving is necessary to achieve the required particle size distribution. Figure 11 indicates that the individual particles become coherently joined. There is also some residual porosity within the material. Porosity may be reduced by employing higher compaction pressures, higher sintering temperatures and longer sintering times, but such changes may be counter-productive with regard to the milling operations that are necessary since the strength of the product is greatly increased. The Co/WC class of materials are produced by these methods.

The process of binding particles together also enables the combination of different materials (e.g., alumina and titania, yttria and zirconia, etc.) and the recycling of fines from some other process. For example, particles produced from the sol-gel process may, in turn, be agglomerated. The binding material is generally of organic-base, such as polyvinyl alcohol or carboxy-methyl cellulose. The use of a binder is not mandatory since the fines can be consolidated by pressing and sintering; however, a further comminution and sieving procedure is always required for these materials. The agglomerated particles must be re-sieved regardless of whether any sintering is performed so that the new particle size distribution can be ascertained. The nature of the sieving operation is quite crucial since it may and usually does lead to a further breakdown of any loosely agglomerated particles and thereby lead to a false representation of the as-received feedstock.

An agglomerated morphology is not spherical; it can be described as being globular in appearance with many protuberances on any surface. Therefore it may be expected that such a powder is difficult to feed. This is generally true, but the thermal spray parameters for that particular material have been optimized and so these powders are viable from the technological viewpoint. A more important aspect is that the theoretical density of sintered powders is usually lower than powders obtained from the fused and crushed routes. Since there is an empirical relationship that relates low densities to low tensile adhesion strengths, then agglomerated powders (or spray dried and sintered powders) may exhibit lower strengths. It is emphasized that such powders are still acceptable for their intended applications and may have the additional benefit of being a lower-cost feedstock material.

Other Material Production Techniques

A number of processes are available that enable the production of powders with unique geometry or composition. Some of these will be described in this section.

Nickel-Aluminum Composite Materials. The bond coat materials which are based on nickel and aluminum metals are called ***composites*** and are produced by two main methods. One process is the same as the prior-described agglomeration technique but is referred to as ***cladding*** within the thermal spray trade. Particles of one of the components are coated onto a core of the other by the use of a binder. For example, 5 wt% aluminum can be clad onto a nickel core, or 80 wt% nickel can be clad onto an aluminum core. The other class of Ni-Al composite materials are termed ***prealloyed*** and are manufactured by using water or gas atomization methods; for example, a 5 wt% aluminum - 95 wt% nickel alloy is manufactured by this method.

Abradables. An important class of materials are those used for high-temperature abradable seals. These materials operate at 650 to 850°C (1200 to 1560°F) and consist, in some instances, of NiCrAl/bentonite composites. The feedstock material is a composite of a nonmetallic core that is coated with a layer of metal. The bentonite core powder is suspended by mechanical agitation in a nickel amine sulfate solution. The nickel can be reduced and allowed to deposit onto the bentonite by bubbling hydrogen through the solution at 180°C (355°F) and 2.4 MPa (35 psi) in a high-pressure autoclave. Bentonite particles of size 1 to 200 microns are used as cores and the nickel coating thickness is greater than 2 microns. The nickel coating can be further alloyed with chromium and/or aluminum by pack diffusion. The average diameter of the core particles is about 85 microns and the alloy coating thickness is about 6 microns. The overall powder composition is, typically, 20 wt% bentonite, 5% Cr, 3% Al and 72% Ni. The core material, which may also be graphite or a silicate, is abradable when thermally sprayed and allows the rotating components to cut and form a

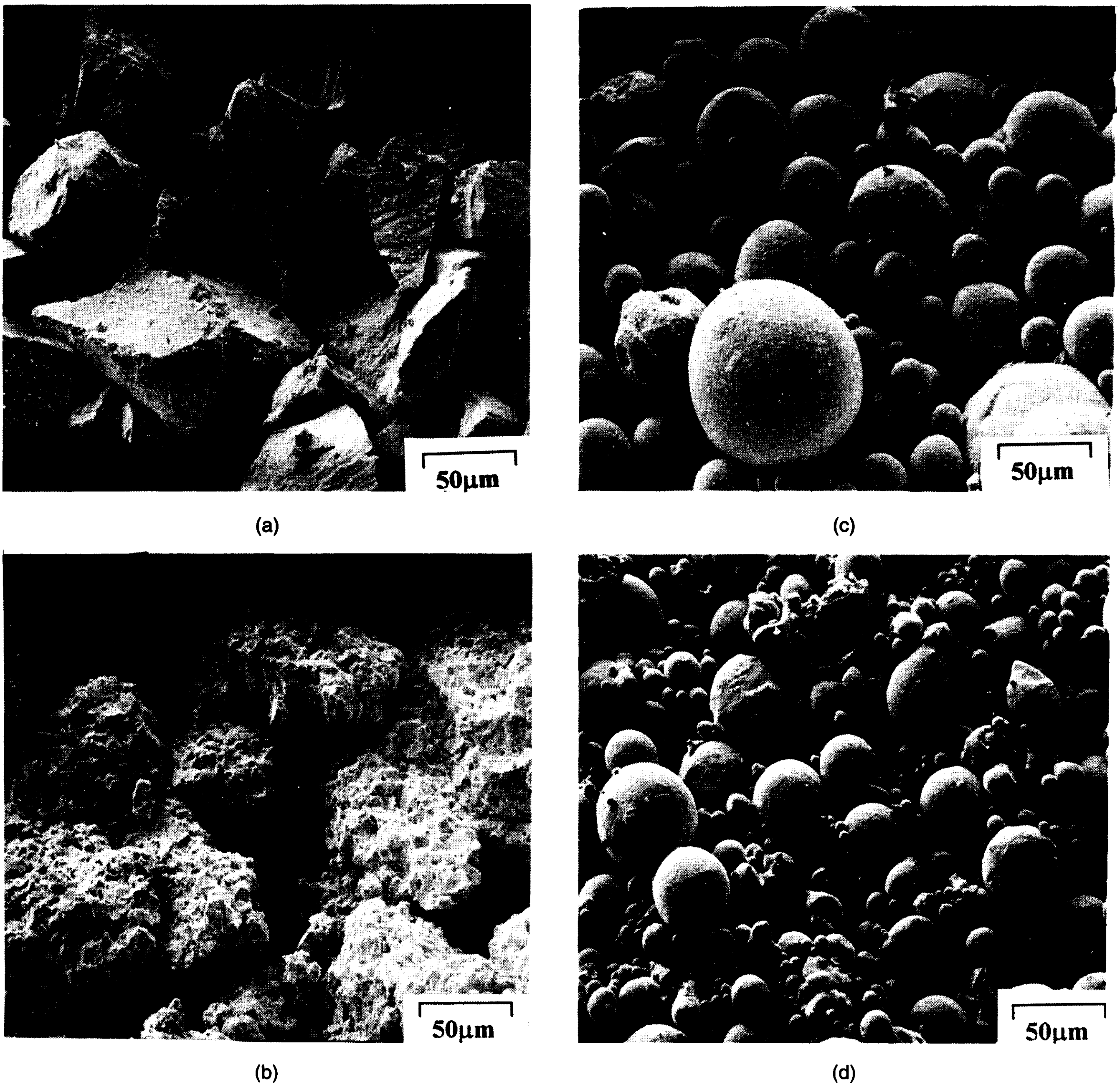
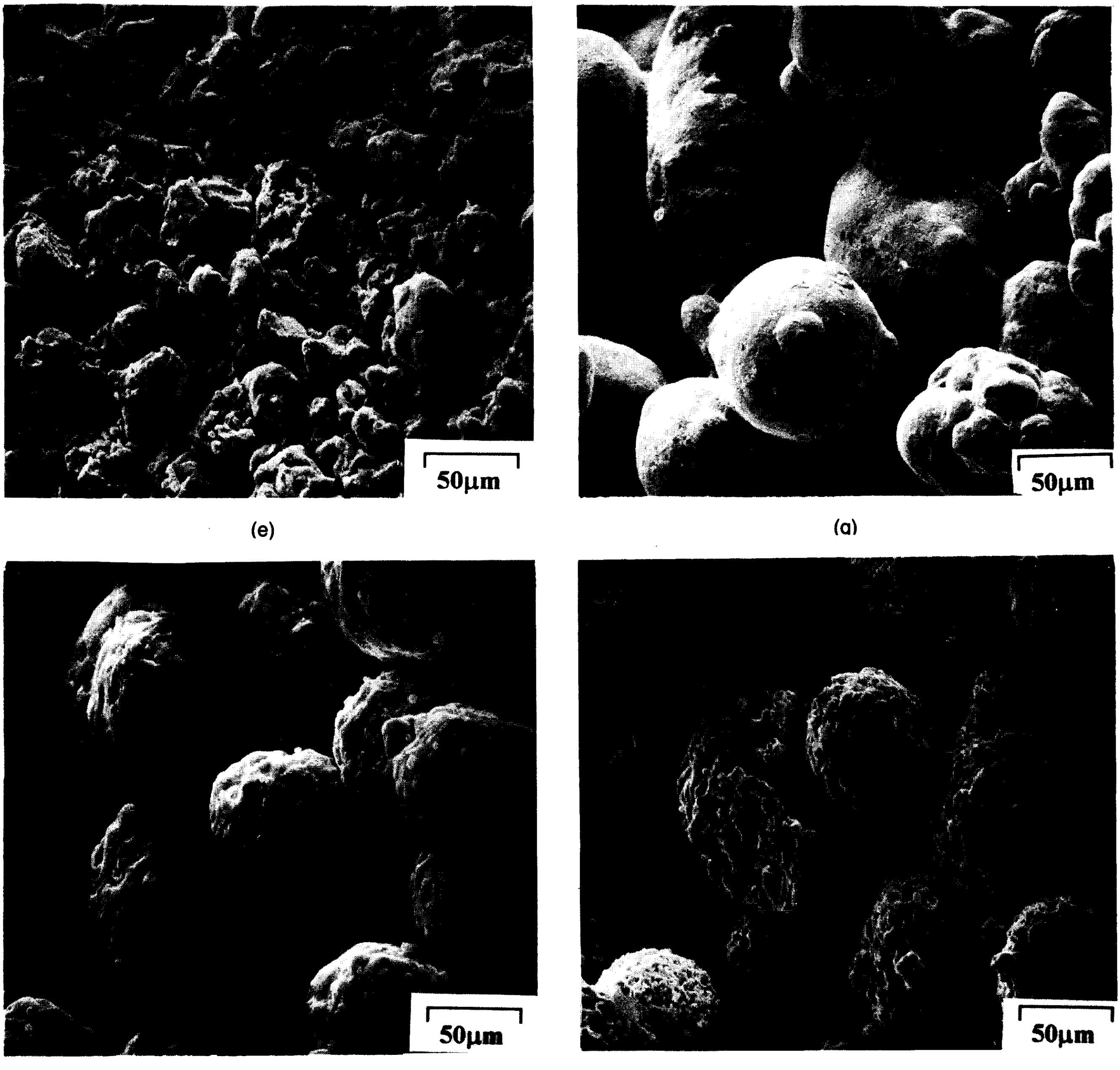


Figure 12. Scanning electron micrographs of different powders: (a)250°C (480°F) fused and crushed YSZ, (b)spray-dried YSZ, (c)plasma-fused YSZ, (d)gas-atomized Ni-Al, (e)water-atomized Ni-Al, (f) 5 wt % Al clad onto Ni, (g) 80 wt % Ni clad onto Al, (h) agglomerated materials.

close-clearance seal. The metallic components of these coating systems have good high-temperature resistance and permit satisfactory bonded porous structures to be formed for special purpose abradable seals.

Plasma Fusion. A representative process for a plasma fusion method is the technique used to manufacture the hollow-oven spherical powder (known as the HOSP process). The HOSP process involves the thermal spray processing of spray-dried materials. The powders are melted in the heat source to form spherical particles that are hollow. The particles are collected and then classified by the usual processes. The process is applied to ceramics such as alumina, zirconia and chromia-based materials to form powders with good flowing characteristics. The manufacturers of this product claim that the good powder feeding properties of so-formed materials, together with their chemical homogeneity



(e) (a)

(f) (h)

Figure 12. *(cont'd)*

and ability to melt high-melting-point materials within the thermal spray stream, achieves a superior coating to one obtained from powders produced from a fused and crushed route. The basis of the higher-productivity argument is that hollow spheres allow more-uniform melting behavior.

Agglutination. A new laboratory technique for producing ceramic / metal composite powders is the agglutination process. This is a low-temperature process, on the order of 50 to 85°C (120 to 185°F). The ceramic cores are first tumbled in a binder phase of polyethylene glycol to form a uniform coating. The binder phase is about 4 wt% of the powder. The second step is to cool down the mixture and break it up by conventional milling. The coated cores and fine metal powder (5 to 10 microns) are charged into a tumbling furnace where they combine to form a metal-coated core. The advantage of this process, other than the technical simplicity, is that it is very energy efficient and virtually unlimited with regard to the combinations of materials that can be formed into a composite.

Blended and Co-Deposited Powders. A final aspect of feedstock preparation that concerns the topic of composite powders is that of blended powders and co-deposited powders. Blended powders are mechanical mixtures of separate components; i.e., the blended powder is fed through only one power port. Care must be taken that the constituents of the powder blend have the ability to be sprayed with identical thermal spray parameters; otherwise no coherent deposit will form. These powders therefore require adjustment of the appropriate particle size distribution with respect to their weight loading in the composite coating.

Co-deposited materials have very similar structures as blended materials but are formed from a dual powder-feeding arrangement. The powder ports may enter the flame at either the same location (if they require the same thermal trajectory), or at different locations (if they need different thermal heating). One advantage of the dual port system over the blended powder technique is that a continuous adjustment of the composite chemistry can be achieved. Such coatings are referred to as graded.

Particle Morphology and Porosity

The prior sections have indicated that the particle morphology is not only an important part of the powder feeding stage but that it can also be controlled. The range of particle morphologies that may be produced is shown in Figure 12. Particle morphology is most commonly ascertained with either scanning electron or optical microscopy. The use of the electron microscope has the additional ability of checking the chemical analysis of the material if the microscope is equipped with an analytical facility.

The shape of the particles gives a most important indication of the processing methods for the material since the surface textures of the particles have quite different characteristics, depending on the method. These morphological features also allow the flow capability of the materials to be qualitatively ascertained, if it can be assumed that perfectly flowing material would be mono-sized and have smooth surfaces.

The spray dried, sol-gel manufactured composite and the agglomerated and sintered feedstock materials have characteristic globular features on the surface. The scale of these features is less than about 5 microns; however, they do not disrupt the overall spherical nature of the individual particles and therefore the flow characteristics of these materials are good. The fused and crushed materials are clearly distinguished as having smooth fracture surfaces which are indicative of their manufacturing method. Their shape is not spherical and can be thought of as being very irregular in shape. These powders are quite dense and they are sensitive to flow characteristics due to a non-ideal geometry.

The individual feedstock particles may also exhibit very fine particles (less than 1 micron) on their surfaces. These fines are indicative of either strong electrostatic forces within the body of the powder or

Table 1. Standard Sieves for Most Thermal Spray Powders

Sieve size number	Microns	Inches
100	149	0.0059
120	125	0.0049
140	105	0.0041
170	88	0.0035
200	74	0.0029
230	62	0.0024
270	53	0.0021
325	44	0.0017
400	37	0.0015
500	31	0.0014

moisture contamination. The overall result is that such particles may be indicative of either a poor powder classification procedure (as described below) or a potential powder feeding problem. A large range of particle sizes will cause irregular deposition due to inconsistent particle trajectories.

Particle Classification Methods

An important part of powder manufacture is the particle size distribution. This aspect of powder technology is often referred to as *classification* since powders are broken up into various groupings. The most common method of separating powders into their size fractions is by sieving. Sieving can be used either as a diagnostic method to determine the quality of a powder or as a manufacturing method to form powders of a specific size distribution.

The sieve with the largest holes (or largest mesh openings) is placed on the top of a sieve stack. The order of the sieves should follow the ASTM specification where the mesh openings of two consecutive sieves has a 1.414 (the square root of 2) ratio. Table 1 is a listing of the standard sieves that are of general use for thermal spray powders. A typical sieve stack might classify particles according to the following 6 micron ranges: +300 (oversize), -300 +150, -150 +106, -106 +74, -74 +45, -45 (fines or undersize). The particle fractions are, for example, stated as "minus 300, plus 150," which indicates that the powder fell through the (300 micron) mesh but was retained on the 150 micron mesh. The powder is fed into the top sieve and the assembly is vibrated or shaken. The particle size distribution is determined by weighing the amounts of powder in each sieve. The result is usually stated as a percent weight fraction, although some scientific studies find number fractions more useful.

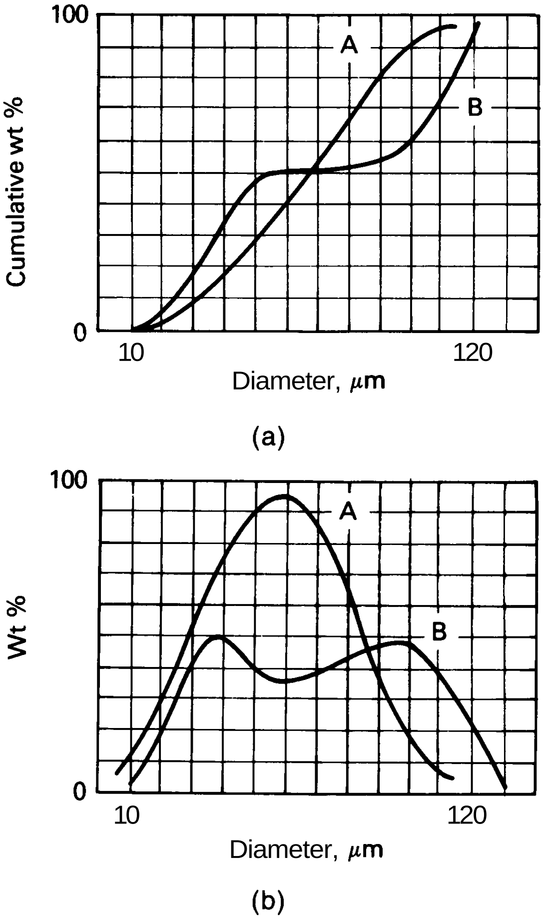
The particle size distribution can be graphically represented as a cumulative plot (Figure 13a) or as a frequency plot (Figure 13b). These graphs are basically identical since they can be derived from each other; the summation of the individual frequencies at each size (i.e., Figure 13b) enables the cumulative plot (i.e., Figure 13a) to be ascertained. The key features conveyed from particle-size distribution data are listed below.

- The most commonly occurring particle size (i.e., the mode value) is indicated by the peak in the frequency plot. The mean value can be ascertained from the diameter at the 50 wt% value of the cumulative graph.

- The sharpness of the plot gives a qualitative indication of the spread in the particle size. A sharp peak (for example, powder A in Figure 13b), indicates that the particles have a similar size; whereas a flat or broad curve (powder B in Figure 13b) indicates that the particle sizes are spread out over a range of values.

- The maximum and minimum values in measurable diameters allow the particle size range to be determined.

- It is important to look for a distribution that does not fluctuate since this is indicative of a bimodal distribution of particle sizes. Such powders, i.e., powder B of Figure 13, do not feed reliably.



(a)

(b)

Figure 13. Particle classification curves:(a) cumulative and (b)frequency.

A major problem with sieves is their maintenance and overall upkeep since they are prone to abuse and general misuse. The sieve wires may deform, be ruptured or corrode, resulting in changes in the particle sieving process that render it unreliable or useless. Individual particles may also become lodged in the sieve grids and this can decrease the sieving efficiency (since the total sieve area will be decreased) and may also cause contamination of any powder batches that are passed through the same set of sieves.

Sieving can be performed under either wet or dry conditions. Dry sieving is usually preferred for materials that are sensitive to corrosion or those materials that may tend to re-agglomerate in the sieving media. The advantage of wet sieving is that high through-puts can be attained under high pressure conditions. Also, in many cases, the powder may already be in solution and therefore wet sieving is a convenient and logical process for particle classification. A wet processing stage is usually encountered during the chemical production processes such as precipitation and sol-gel methods. Therefore, wet classification can be performed which has the added benefit of washing away any residual reactants from the powder. Very slight milling will separate any agglomerated particles with minimum particle breakup. Wet sieving is the preferred classification method when there is concern over environmental or toxic issues.

Quality Control and Feedstock Material Specification

The specification of a material is quite important since this often establishes its utility. A thorough knowledge of the appropriate standards and testing procedures is necessary for appropriate comparisons between materials. These considerations should not be considered alone but in conjunction with the thermal spray process since this also influences the coating quality. Some of the quality control tests that should be considered are: powder manufacture, particle size, powder quality control, material chemistry, Hall flow rate, spray tables, safety, porosity of powder, handling characteristics.

Any of the existing standards or potential standards for powder quality control should be implemented with respect to the intended use of the coating/substrate system. It is important that any coating not only be subject to some specific standard but that the standard be relevant to the particular feedstock material. The issue of "what standards are appropriate for the thermal spray industry?" has not been completely resolved.

The following aspects can be noted with regard to testing methods for feedstock materials. (Note: Further detail is included in Lesson 6.) There are two major methods of measuring the particle size (other than the sieving method described in Particle Classification Methods): by an X ray method or by a light-scattering technique. Batches of the same material, when tested by the two methods, have been found to give quite different particle size distributions. There are technical reasons for this discrepancy but, more to the point, it is disturbing that the particle size is dependent on the measuring technique since the thermal spray applicator desires this data in order to optimize the spraying parameters. The up-shot of this discussion is that the measuring technique for the mean particle size or the particle size distribution should always be referenced so that the applicator is able to adjust the spraying parameters accordingly.

A quality control issue which is related to powders concerns the examination of deposits. A simple method of determining whether the material or thermal spraying parameters are suitable is the "wiper blade" test. This involves passing a series of glass sides (or metallic substrates) in front of the thermal spraying torch during a trial powder spraying operation. Usually a series of glass slides are tested simultaneously by off-setting them on a rotor-type device so that a range of stand-off distances can be examined in one thermal spray run. The degree of particle melting can be quickly ascertained by optical microscopy and the optimum thermal spray deposition parameters quickly found. The best powder/spraying conditions will be obtained when a well-formed and coherent splat can be produced.

It is appropriate to comment on the powder feeding problem that was addressed earlier. Three common observations that infer a quality control procedure is necessary are listed below.

- Powder has irregular flow. For example, it is observed that the powder seems to be "turning on and off."

  The powder can only be fed at high carrier gas

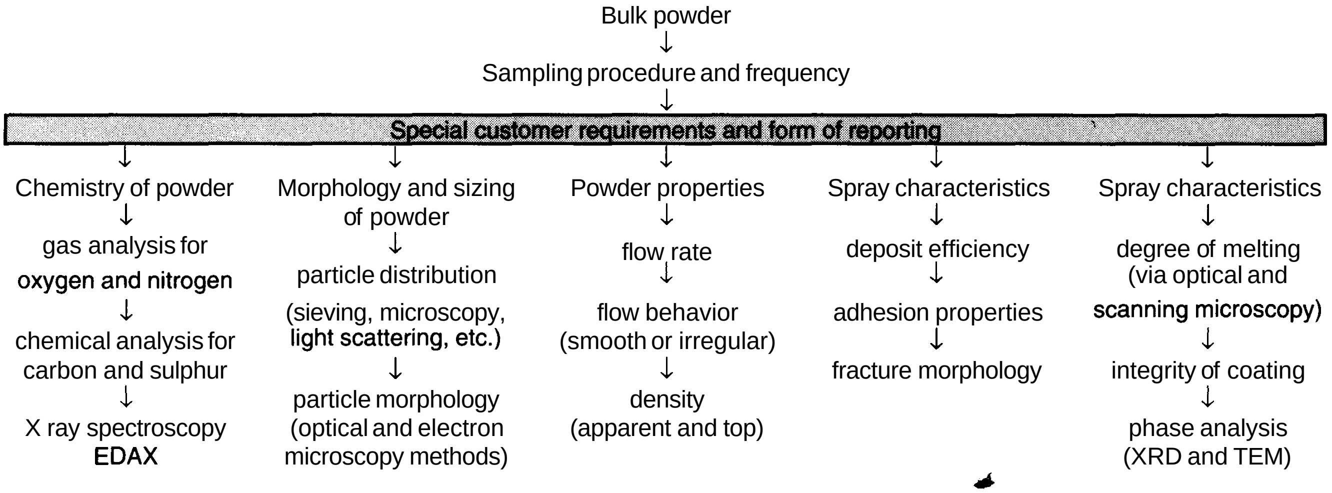


Figure 14. Quality control procedures for powders.

flow rates. These flow rates are high enough to carry the powder straight through the thermal source.

- The deposit efficiency (DE) is observed to be low.

Possible solutions to these problems would be: adjust the powder port and delivery tube sizes, dry the powder in an oven, and/or homogenize the powder by rotating the powder container for a few minutes. These are all essentially operator options and if the powder feed problem still exists then modification to the powder specifications may be required as the thermal spraying equipment (in particular the power feeder) may need maintenance.

Powder testing and the associated quality control must be a continuing task and a suggested schedule is shown in Figure 14. This figure emphasizes that it is essential to test coatings as well as specify only high-quality powders. Very few of the determinations have standards that are followed throughout the thermal spray community. Often any standards or specifications are supplied by the consumer of the feedstock and different consumers may have different requirements for exactly the same powder under different applications. These standards issues need to be resolved.

Summary

The quality of any thermal spray coating will always be dependent on the initial feedstock material. There is no technical procedure that can correct for a poor-quality powder. The coatings are formed from the repeated deposition of molten and semi-molten feedstock particles. The resultant lamellar dimensions are about 60 microns in diameter and from 1 to 4 microns thick. Metallographic characterization of coatings is often used as a basis for determining the suitability of the initial feedstock. However, such methods should be used with caution since no universal accepted standards for coating preparation (i.e., the basic metallography practice) and characterization exist at present. Advanced analytical techniques which involve image analysis and statistical quantification of the microstructure are currently being developed to overcome this.

Feedstock materials should be considered as part of a coating system that includes the substrate, possibly an intermediate layer and finally the bulk of the coating. The coating structure is highly oriented with the lamellae parallel to the surface of the substrate. The real contact area of lamellae is 30% of the available boundary area. Unmolten particles may become incorporated into the coating and these, along with the interlamellar oxide boundaries and porosity, constitute regions of poor bonding which ultimately may lead to failure of the coating system. On the other hand, melting of a material does not always infer good adhesion since substrate properties and material/substrate interactions are critical. The overall coating manufacturing schedule consists of the following components: production of the feedstock, then the production of the coating so that a material-specific structure is formed that, in turn, exhibits distinct materials properties for specialized applications.

Some feedstock materials, for instance alumina and stabilized zirconia, are manufactured by a wide range of different production processes. It is not surprising that each feedstock material needs to be optimized with regard to the thermal spray parameter of particular spray devices, and that the so-formed coatings exhibit variable material properties which are dependent on specific attributes of the feedstock. The material properties of coatings are generally independent of specific equipments (for the same type of thermal spraying) if the powder is optimized with respect to each equipment. Generally the material properties of the coatings such as hardness, wear, and tensile adhesion strength correlate well with the coating density. It is therefore essential that the thermal spray applicator appreciate that feedstock materials are not just designated by their chemistry and particle size. A simple substitution of one powder in preference to another (because it may be cheaper, more available, etc.) does not necessarily imply that the original performance characteristics of the coating will be maintained.

The focus of this lesson has been on the manufacturing methods for thermal spray feedstock materials. The ideal material will be matched to the thermal spray parameters so that a coherent and dense coating is produced. Guidelines have been put forward in this module to enable an understanding of feedstock/coating relationships. Particular emphasis has been placed on attributes of the powder that control its feeding into the thermal source.

Self Test Questions

1. Comment on the statement, "it is only necessary for the powder to flow in order to deposit well-adhering coatings."

2. List methods whereby spherical powders, either metallic or ceramic, may be manufactured.

3. Sol-gel methods are essentially precipitation of a stable reaction product; true or false?

4. Explain why comminution methods can not grind powders to particle sizes less than about 5 um.

5. Describe the principles behind a “volumetric-based" and a "density-based" powder feed system.

6. Angular particles are, generally, more difficult to feed; true or false?

7. Explain why powders are generally stored in warm ovens immediately prior to use.

8. Warning! This question is a bit tricky. What is the prime requirement for a thermal spray powder; is it to have a high flow rate, a high deposition efficiency or a high adhesion strength?

9. How is the particle size distribution related to powder feeding.

10. Distinguish between composite powders, blended powders and co-deposited powders.

Selected References for Further Study

References 1 through 6 consist of hundreds of papers presented at the National and International Thermal Spray Conferences hosted by ASM. References 7 through 10 are papers given at other major thermal spray conferences. References 11 and 12 are a book and an article, respectively, that address the *general* aspects of thermal spray technology, while references 13 to 18 focus on *specific* aspects of thermal spray technology that have been addressed in this lesson.

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Appendix: Answers to Self Test

Note that only a guideline to the answer is given.

1. Your answer would indicate that effects such as particle size distribution, mean particle size and process conditions (i.e., plasma, flame, LPPS etc.) are also very important during thermal spray deposition.

2. Gas atomization for metallic alloys, Sol-gel methods for ceramic materials.

3. False, since sol-gel methods rely on colloid formation and these stable atomic clusters are less than 150 nm in size.

4. The particles will agglomerate and weld together at these small sizes since the contact forces are very high.

5. A volumetric-based system would be, for example, one which has a rotating wheel to deliver material into the powder feed delivery tube. The fluidized powder feeding system, on the other hand, relies on density changes (among other factors) to transport powder. Descriptions of these systems can be noted within the text. Sketch diagrams of the apparatus which indicate the principles of operation should also be included.

6. Generally this statement is true. It has been assumed that we are comparing angular particles to spherical particles. The answer would be false if the other particles were dendritic, or flake or plate morphology or contained superfines (many particles less than 1 micron).

7. It is necessary to keep powders dry to enhance their flow characteristics. Any moisture in the powder tends to make the powder clump or agglomerate within the powder feed delivery system and this often makes it impossible to obtain smooth/non-pulsating delivery. This is observed as a fluctuation and pulsation in the powder stream density and is technically known as saltation.

8. The answer to this question is not clear cut since it relies on the joint topics of application and productivity. Generally, high powder flow rates may lead to poor adhesion strengths (since residual stress buildup is greatly increased), but this may be offset by a higher deposition efficiency.

9. Powders with a close distribution (i.e., mono-modal and not a large range) have been found to flow well. The analogy is that a group of 10 billiard balls move much more efficiently and evenly than an equivalent weight mass consisting of a mixture of golf balls and billiard balls.

10. The individual particles of a composite powder each consist of different components; i.e, there is virtually no metallurgical bonding between the separate species. A blended powder is a mechanical mixture of different powders. Co-depositedpowders is a general term that refers to the spraying of several materials simultaneously. Another aspect of this question would be to emphasize that each spray material may require different thermal spray parameters and that the coating quality is influenced to a high degree by the nature of the feedstock.

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C51L4
Test Series A
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THERMAL SPRAY TECHNOLOGY
Materials Production for Thermal Spray Processes

The Materials
Information Society

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MULTIPLE CHOICE

Place the appropriate letter (A, B, C, D) in the box. (Only one answer is correct and each question has a value of 10 points.)

1. Which powder property (or properties) control powder flow? ☐

   (A) Particle size.
   (B) Particle morphology & size.
   (C) Gas flow rate.
   (D) Particle morphology, size distribution & size.

2. The following statements address the physical and chemical nature of powders; which statement is most accurate? ☐

   (A) The physical/chemistry attributes of powders can not be generalized.
   (B) Metallic powders are always spherical since they are produced by atomization methods.
   (C) Ceramic powders of 100% theoretical density are never produced (since they are usually agglomerated).
   (D) Powders, whether of ceramic or metallic nature, are always homogeneous with regard to chemistry.

3. Which most accurately describes the powder-feeding problem? ☐

   (A) The inability to pour powder into the feeding hopper of the system.
   (B) A practical problem where low deposit efficiencies are observed.
   (C) A condition in the feeding system where powder does not reliably flow through the delivery tube.
   (D) None of the above statements are appropriate.

4. Which of these best describes a powder feed delivery system based on principles of creating a differential pressure within the powder hopper so that a Venturi effect is created? (NOTE: You should be able to sketch diagrams of the methods.) ☐

   (A) A rotating wheel feeder; either a slotted wheel or a grooved disk.
   (B) A feeder based on a fluidized bed.
   (C) A gravity feed system that depends on powder flowing through a rubber tube that can be constricted to stop powder flow.
   (D) A paper tape system where the powder is contained within predetermined powder shots.

5. The prime characteristics of a crushed and milled powder are: ☐

(A) smooth edges and close control of particle sizes.
(B) angular features and a tendency to manufacture sub-micron particles (fines).
(C) angular features and very wide particle-size distribution.
(D) a spherical morphology and ability to produce a homogeneous chemical composition.

6. An atomized powder has the following properties or characteristics: ☐

(A) Morphology may be either irregular or spherical depending on the operational parameters of the powder processing equipment and nature of the material.
(B) Atomized powders are always spherical since the individual droplets must freeze in-flight.
(C) Metallic materials are not usually atomized since they would be contaminated by the atomizing media.
(D) Atomization always leads to mono-modal particle size distributions.

7. Which of the following is not an advantage of the sol-gel manufacturing technique? ☐

(A) Ability to produce spherical powders which are free-flowing.
(B) Closely controlled powder composition, as sol-gel is based on solution chemistry.
(C) It is a high-temperature process; very dense materials can be produced.
(D) The degree of powder porosity can be controlled.

8. Which method is not allied to a low-temperature "chemical" method? ☐

(A) Spray drying. (C) Freeze drying.
(B) Agglomeration and sintering. (D) Sol-gel methods.

9. Which best describes a powder produced by crushing and sintering? ☐

(A) Usually of spherical shape with small rounded protuberances on the surface.
(B) Sharp angular features and nearly 100% theoretical density (since sintering has taken place).
(C) Almost completely spherical particles since full melting has taken place.
(D) Very fine particles (less than 3 microns) of irregular shape that tend to clump during feeding.

10. The only way to classify a powder into size ranges suitable for thermal spraying is: ☐

(A) to use a spherically shaped powder with small rounded protuberances on the surface.
(B) to control atomization so that only the specified particles are manufactured.
(C) to control the carrier gas flow rate so that only the required particles are entrained in the gas that flows to the thermal spraying device.
(D) to perform a sieving operation.

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This MEI Homestudy Lesson is dedicated to the memory of Paul B. Urban, Manager of New Product Development in the Education Department at ASM International until his untimely death in 1991.

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