POWDER METHODS

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Summary

In one form of powder methods, the powder is compressed in a die and subsequently sintered. The final dimensions are determined by the powder, die, compaction pressure, and sintering densification cycle. The higher the compaction pressure, the higher the final density and properties. Minimal densification occurs in sintering if the powders are large and the sintering temperature is low. This is the means for forming simpler shapes in high volumes, such as ceramic insulators and automotive timing sprockets.

In contrast, if the powder is small and the sintering temperature is high, then densification occurs in sintering. Higher temperatures lead to pore elimination in sintering and deliver high final properties. An often used powder method relies on low external stress during shaping and high temperatures in sintering to densify the compact, leading to large sintering shrinkages. Such routes are used for form more complicated shapes such as wristwatch cases and ceramic turbochargers.

When both temperature and pressure are applied simultaneously, then rapid densification occurs. High temperatures weaken most engineering materials, resulting in easier compaction, although some materials are degraded, decompose, or evaporate and make high temperature powder methods difficult. The combined action of temperature and pressure produces full density for most powders. Selection of the processing conditions depends on particle size, density, rate of heating, and many material-specific factors. The complexity to the processing cycle couples to the high equipment and tooling expenses to create barriers to widespread use of hot deformation in processing any but high value systems. It is used for diamond tools, high performance oil well drilling equipment, high stress automotive components, biomedical implants, sputtering targets for microelectronic processing, and jet engine components, as examples.

1. Introduction

Powder techniques are widely employed in the fabrication of cermets, metals, ceramics and even some polymer systems. Widely recognized by the term P/M, the group of techniques are variously interpreted to mean powder methods, particulate materials, or powder metallurgy. Overall P/M processing techniques involve the production of powders and conversion of these powders into engineered structures. The approaches all generally rely on first shaping a powder followed by thermally bonding the powder into a coherent solid by sintering. In the classic process, the powder is first compacted in rigid dies and subsequently heated in a second operation to sinter the particles together.

In all P/M approaches, the powder starts as a fluid-like substance, where it is neither a liquid nor a strict solid. This fluid-like character makes P/M similar to casting, since the powder can flow to fill out the desired shape. A dramatic difference is that in P/M the powder flows into a die cavity at room temperature. Under pressure, the powder is densified by deformation, with the formation of weak friction bonds. Although weak at this point, the compacted powder is now like a solid. To improve on the strength, the structure is heated to a temperature where the particles sinter to one another to form strong bonds. This sintered product has many benefits—it holds the shape imparted by the shaping or compaction step, but builds considerable strength in sintering. Many variants exist to this basic process.

Powder techniques are most attractive for forming components with moderate thicknesses and sizes, with a weight typically in the 1 to 1000 g range. The various processes match well to discrete engineering shapes with moderate levels of complexity and moderately smooth surfaces, but not too complex or too smooth. P/M can then be applied where dominant concerns are strength, stiffness, cost, and toughness. Because of possible residual porosity, most applications try to avoid corrosion, oxidation, and wear situations, and focus more on applications where surface finish, shape complexity, and mechanical or electrical or magnetic properties are of concern at low manufacturing cost.

The production of metallic, carbide, and ceramic components by compaction and sintering is a large industry that produces many products for modern industry—ranging from eyeglass frames to wood cutting tools, and includes bearings, connecting rods, oxygen sensors, fiber optic connectors, golf club heads, and transmission gears. Because of a high tooling cost, most P/M production is targeted at structures fabricated at high rates (measured typically in parts per year), such as for automobiles, lawnmowers, home appliances, telecommunication devices, sporting equipment, business machines, computers, and other electro-mechanical structures. Some components, such as tantalum capacitors, bronze bearings, and molybdenum heat sinks, are produced at rates as high as 100 million per day. More typical are production rates of millions per year, often for automotive applications. An example of the latter are engine valve seats which are die compacted and sintered for automobiles at production rates in the range from 250 to 300

million per year.

Today, the press and sinter powdered materials industry is made up of many companies, and probably contributes nearly \$50 billion in annual products worldwide. Most of the companies have specialized on certain compositions, processes, applications, and production techniques. The largest activity is associated with cemented carbide cutting tools, followed by electronic ceramics, and ferrous structural components. Thus, P/M materials have a few key segments of the economy where large quantities of similar shapes are used, with modest shape complexity-cutting tools, electronic components, automotive components, consumer tools and products, and sporting equipment being a few. To attain the desired mechanical properties requires tailoring of the composition, but high strength only arises from sintering. The combination of low production costs and high sintered strength dominate the P/M selection criteria. Since a high density is a precursor to high strength, the desire is to eliminate porosity in the sintering stage. Production technologies that allow high final densities include high temperature sintering, powder forging, powder rolling, injection molding, infiltration, hot isostatic compaction, and pneumatic forging. For this article, the emphasis will be on the basic processes of either low pressure shaping or high pressure die compaction, followed by sintering.

Overview of Processes and Steps

The P/M processes can be divided between two extremes. One extreme is encountered in injection molding and other processes that start with loose powders. These are sintered to nearly full density at high temperatures. Here, the powder is shaped in the forming step, but not compacted. Most important, the particles are densified in sintering, not in shaping. The other extreme is encountered in traditional die compaction, where densification occurs in pressing. Here, sintering has little impact on densification and is largely used to bond the particles. Between these two extremes are various technologies where sintering and compaction are combined for densification. Some examples occur in hot pressing, hot isostatic pressing, and hot forging. This categorization is illustrated in Figure 1, where key steps in the conversion of powder into a component are outlined. Pressure-based densification relies on lower sintering temperatures, larger particle sizes, and higher forming pressures to densify the powder. Alternatively, sintering-based densification relies on low forming pressures, high sintering temperatures, and small sized powders. The hybrid densification techniques apply temperature and pressure simultaneously to obtain a pore-free product.

With respect to the sintering step, some technologies rely on low temperatures to bond the particles, while others rely on high temperatures to densify the structure. Sintering densification results in a volume change, while bonding only results in a strength gain. Again, each process is characterized by a typical temperature and time. Various P/M fabrication techniques rely on a combination of pressure and temperature, for example hot isostatic compaction might involve 200 MPa at 800 EC for 1 h to densify a steel powder or 1350 EC and 5 MPa for 1 h to fully densify a cemented carbide (WC-Co). In each case the cited conditions are only examples: there is much variation between production facilities and materials.

A variety of P/M processing techniques are known, and many are well documented in standard handbooks, such as provided by ASM International. However, when analyzed as to importance to component production, two P/M techniques are dominant: die compaction and injection molding. On a relative use basis, the press and sintering technology is very large on a tonnage basis, providing about 85 to 90% of all sintered P/M products. Next is injection molding, which constitutes about 5% of all sintered P/M product sales. These are followed by smaller, but important technologies such as hot forging and sinter forging, followed by other technologies such as slip casting and sintering, tape casting and sintering, warm extrusion and sintering, cold isostatic compaction and sintering, hot isostatic compaction (simultaneous pressure and temperature), hot pressing, spray forming, and even explosive compaction. Many of these variants are detailed in standard references attached as suggested readings for more details. Each of these production routes has certain advantages and disadvantages, usually leading to selected component shapes, sizes, and applications.



box indicates densification step

Figure 1. A Schematic Categorization of P/M Production Processes Differentiated based on where densification occurs in the cycle, ranging from the forming step to the sintering step, with hybrid technologies that simultaneously compact and sinter

As an example, for situations such as automotive components that will see high fatigue stresses, full density is necessary; thus, hot isostatic compaction or hot forging are recommended. Forging is easier to automate for high production volumes, has a short cycle time, and provides a high final strength. Accordingly, forgings are used for connecting rods, where millions are produced every year, while hot isostatic compaction is the choice for lower volume production of high performance aircraft landing gears. In any selection of a P/M process, consideration of the production economics is a necessity.

1.1. Shaping Versus Compaction

Several approaches exist to P/M technologies. Die compaction at high pressures is the dominant approach, where densification and shaping occur simultaneously. After

compaction, the structure will be near 85% dense. In the subsequent sintering step, the particles are bonded, but little sintering densification occurs. Strength increases in sintering by a factor often near 100.

An alternative is to use low pressures and high binder contents to shape a powder. Largely, the binder acts as a glue to hold the particles into the desired shape. Most binders are polymers, which might be mineral oil or polyethylene. In shaping, the porosity ranges from 40 to 60% (ignoring the polymer which is sacrificial), so sintering is performed at temperatures where densification occurs. Small particles assist in densification during sintering. Thus, key differences between compaction and shaping are in the forming pressures, initial porosity levels, particle sizes, and sintering temperatures. Compaction relies on high pressures and the porosity after compaction is low, while shaping is the opposite. The third alternative, full density processing, relies on the simultaneous application of pressure and temperature and is usually applied to larger structures aimed at high performance levels.

1.2. Shaping Technologies

The differences between compaction and shaping technologies impact on production costs. Key considerations are the component size, desired final density, and shape complexity. A general categorization of shaping technologies is as follows:

- Binder-assisted extrusion: long structures, small particles, constant cross-section, relatively simple shapes,
- Injection molding: complex, small components, high performance materials,
- Slip casting: very large structures, constant wall thickness, low precision,
- Tape casting: flat sheets, very simple shapes, small particles.

The use of small sized powders and polymeric binders that are extruded, cast, or molded in various forming devices is the most rapidly growing aspect of P/M. The polymer is required to lubricate particle flow, since the forming stresses are insufficient to deform the powder. These forming approaches use similar powder-binder mixtures. An emerging area of rapid prototyping and rapid tool production will be discussed as a new option. All of these have similar concerns with powder characteristics, binder formulations, dispersants, mixing, viscosity, and polymer extraction prior to sintering.

Injection molding is a productive and widely used technique for shaping plastics. An evolution in powder shaping has been to form powders into feedstock customized for injection molding. Small particles are used to obtain good sintering densification. The polymer provides the lubricity for flow into the mold cavity, where heat is extracted to cause the polymer to freeze the particles into the desired shape. Subsequently, the polymer is extracted and the powder is densified by sintering at a high temperature. Thus, injection molding inherently relies on sintering densification and small particle sizes.

Although the process is more than 70 years old, it was only applied to inorganic materials in recent years. Examples of products formed by injection molding include alumina or stainless steel orthodontic brackets for straightening teeth, high magnetic

permeability iron for controlling computer disk drives, small stainless steel gears for electric hand tools and toothbrushes, stainless steel surgical tools such as scalpels, copper electrical connectors, tungsten-copper computer heat sinks, cemented carbide drills and cutting tips, steel handgun components, stainless steel automotive air bag actuator mechanisms, and iron-nickel alloy microwave filters for high frequency computer chips.

There are several variants to the injection molding technology, but as conceptualized in Figure 2, all essentially consist of four basic operations—formation of a powder-binder mixture as feedstock, shaping the feedstock in an injection molding machine, extraction of the binder (debinding), and sintering. The process is best applied when the components are complex in shape, with concern over production costs and properties in the context of tight tolerances and high production volumes.



Figure 2. Overview of the Powder Injection Molding Process Powder and polymer are mixed to form a toothpaste consistency feedstock. Subsequently, this feedstock is heated and rammed into a cold die, freezing the particles into the desired shape. The polymer is extracted in the debinding stage and the powder is densified in the sintering stage

In molding, cold feedstock is loaded into a hopper where it is transported and compressed by the molding screw. Outside the screw is a heated barrel that melts the binder. At the end of the barrel is a nozzle that fits against the tool set. Tooling is constructed to hold the molten feedstock into the desired shape, with flow passages from the nozzle via a sprue, runner, and gate system. Cooling in the tooling is assisted by water passages buried in the die. To counteract the pressures associated with mold filling, the tool cavity is clamped in the closed position, until ejection.

The feedstock is mixed to form a homogeneous material consisting of typically 60 vol% powder and 40 vol% polymer phase. The best powders are small, rounded with particle sizes in the 0.1 to 20 μ m range, and typical examples are carbonyl iron and gas atomized steels, but in some instances, water atomized steels can be used if the particle size is small.

In its simplest form, molding consists of heating the feedstock to a sufficiently high temperature such that it melts, then forcing this melt into a cavity where it cools and assumes the shape of the tool cavity. The cycle starts by clamping the mold closed, injecting preheated feedstock into the cavity, packing out the cavity until the gate solidifies, and preparing the next charge while ejecting the cooled component. The melt must have sufficiently low viscosity to flow freely into the mold. At the same time, the component must be produced without defects at minimum cost in the shortest possible cycle time.

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Biographical Sketch

Randall M. German is the Brush Chair Professor in Materials in the Department of Engineering Science and Mechanics at The Pennsylvania State University, where he also serves as Director of the Center for Innovative Sintered Products in the College of Engineering. He has worked in powder metallurgy, ceramics, intermetallics, metal matrix composites, and cemented carbides for his entire career, with prior positions at Rensselaer Polytechnic Institute (Robert Hunt Professor), Ney Corporation (Director of Research), Mott Corporation (Director of Research and Development), Sandia National Laboratories (Technical Staff Member), and Battelle Columbus Laboratories (Materials Scientist). His Ph.D. in Materials Science and Engineering is from the University of California at Davis, with the M.S. from the Ohio State University in Metallurgical Engineering, and B.Sc. from San Jose State University in Materials Science and Engineering. Professor German is author of nearly 600 articles, 10 books, 16 patents, and has edited over 20 books. He is a fellow of two professional societies-ASM International and APMI International, and has been recognized by several professional organizations, including being awarded the Distinguished Service to Powder Metallurgy Award by the Metal Powder Industries Federation, Penn State Outstanding and Premiere Research Awards, Tesla Medal, Kuczynski Prize, Samsonov Prize, Geisler Award, San Jose State University and University of California at Davis Distinguished Alumnus Awards. He lives in State College, with his wife Carol and is the father of two sons Eric and Garth who live in New York and California, respectively.