Effects of Processing Parameters in P/M Steel Forging on Part Properties: A Review

Part I Powder Preparation, Compaction, and Sintering

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In the last decade, powder metallurgy (P/M) technology has made marked advances in competitive manufacturing. P/M offers design opportunities that are not possible with other methods, as well as significant cost savings. In the automotive industry, P/M forgings are being developed for applications requiring good high-cycle fatigue properties. The processing parameters, material characteristics, individual stages of compaction and parts production, deformation and densification mechanics and tooling, and preform design influence the properties of the P/M part and related economics. Therefore, a review of the various parameters involved in the different stages of P/M steel forging in net-shape manufacturing and their implications on resulting properties of the P/M parts is presented in a three part review. Key parameters in steel powder preparation, compaction, and sintering of P/M forging and their effect on part properties are described in Part I. Part II discusses issues of forging the sintered compact, and Part III reviews currently available analysis methods for studying the powder forging process.

1. Introduction

POWOERmetallurgy (P/M) has developed from being used primarily for processing refractory and other special materials to a competitive forming method. This technology covers the gamut of materials and components for applications in automobiles, aircraft, lawn and garden equipment, sporting goods, appliances, etc. Powder forging is particularly attractive because it blends the cost and material saving advantages of conventional casting and forging by providing better dimensional and weight control. Connecting rods are an excellent example for the favorable synthesis of powder metallurgy with hot deformation processing. Prior to forging, a sintered compact of powder is produced. The compact is produced to a required size, geometry, and composition of material, possessing sufficient integrity to be handled and moved. The sintered compact also possesses sufficient initial density and homogeneity due to the processes of powder preparation, blending, powder consolidation, and sintering. This review identifies the key parameters in the above stages and discusses their influence on the properties of the compact produced.

2. Classical P/M Processing

The classical powder metallurgy processing method (Fig. 1) begins with powder preparation and blending, from which a green compact is produced by axial and/or radial compaction. The green compact has all the geometrical features of the finished component; however, the mechanical, physical, and chemical properties are not yet fully developed, because at this stage metallurgical bonding has not yet occurred. Sintering is performed during which the admixed lubricants (added earlier for protection of compaction tools) are burned off, and metallurgical bonding occurs between adjacent powder particles producing an agglomerate. Sintering may also be accompanied by shrinkage and growth, resulting in dimensional change; the sintered compact has a substantial volume of residual voids (porosity). To enhance the mechanical properties of the sin-

Fig. 1 Flow chart of the powder metallurgy method for production of components. $[1]$

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tered powder compacts, deformation processing is done, which simultaneously densifies the material and produces the final desired part shape.

3. Metal Powder Production and Powder Characteristics

Characteristics of the starting powder dictate the compaction, sintering, and forging parameters that must be applied to ensure the required composition, homogeneity, impurity level, microstructure, and residual porosity for acceptable properties in the finished part. Metal powder production methods may be classified into the following characteristics, namely:

- Physical
- Chemical
- Mechanical

One of the common physical methods used for producing powders is the process of atomization, in which a stream of molten metal is broken up into droplets that solidify into metal powder particles, cooled primarily by a stream of fluid, usually water, or gas. The atomizing process variables affect the properties of the powders such as average particle size, particle size distribution, particle shape, particle chemistry, and structure. The above parameters determine to a considerable extent the properties of finished compacts and the behavior of the powder during processing by compaction, consolidation, and sintering. The practical aspects of metal powder atomization have been reviewed in Ref 2 through 4.

Control of particle shape is of major importance for further processing of the metal powders. Spherical atomized powders are needed when the powders are to be sintered loose or hot pressed. For cold pressing applications, irregular particle shapes are preferred, because spherical shapes, except for soft powders, produce inadequate green strength due to lower surface area for a given volume.

Chemical methods of producing metal powders involve decomposition of a compound of the metal, which includes a large group of reduction reactions. The principles of thermodynamics, reaction kinetics, etc., involved in chemical reactions are discussed in Ref 5.

Powders are also produced by mechanical comminution processes, generally used for brittle materials such as iron ores. However, these powders are of limited application, because they cannot be cold compacted without modification. Other variations, combinations of processes for producing powders, are discussed in Ref 6.

A predetermined composition of powder is often achieved by (1) blending elemental powders, (2) prealloying powders produced by atomization, (3) prealloying particulate produced by a rapid solidification process, and (4) blending of elemental and master alloy powders. Tests are conducted to determine parameters related to specific processing behaviors of the metal powders. During compaction of metal powders on an automatic press, a given volume of powder flows from the filling shoe into the die cavity. To ensure uniform flow and reproducible amounts of powder filling the die cavity for each stroke of the press, control of apparent density and flow is necessary. These parameters depend on particle shape, size, and size distribution. Compressibility, compression ratio, and green strength of powders are some of the other powder characteristics that are measured using ASTM tests.^[6] Also, dimensional changes in compacts occur during sintering depending not only on the above powder characteristics, but also on other factors such as type of sintering furnace, heat-up rate, sintering time and temperature, cooling rate, and sintering atmosphere.

Powder mixing is more an art than a technical science. The most complicated problem in powder mixing concerns the quality of mixing and evaluation of the degree of uniformity in the mix. Further details on powder mixing and on the role of statistics in evaluating blend samples are given in Ref 7.

4. Relationship Between Properties and Particle Size of Iron Powder Compacts

The process of sintering a metal powder compact depends, apart from other parameters discussed earlier, on the total surface area of the metal powder. The surface area is in turn a function of the particle size and shape. A study of the effects of the granular size of the powder particles on the elongation and ultimate tensile strength (UTS) of a P/M part was made by Veidis and Geiling.^[8] Tensile specimens from iron powder were sintered and normalized and tested on a Super-C-Tinius Olson (60,000-1b capacity) unit. The total surface area of all particles in each compact (assumed to be spherical) was expressed as:

Fig. 2 Ultimate tensile strength versus *1/R* for different sintering times. [8]

where R is the radius of each particle and d is the density of the metal. Therefore, as $At \alpha 1/R$, the measured mechanical properties were examined as functions of *1/R.* Figures 2 and 3 show the relationships between the ultimate tensile strength, the radius of the particle, and sintering time, with ultimate tensile strength increasing with sintering time and decreasing with increasing R . Therefore, Veidis and Geiling^[8] conclude that selection of powder particle size and sintering times in the manufacture of P/M parts is an important aspect of the process to obtain desirable ultimate tensile strength.

Although powder characteristics are important in the production of compacts for forging, they are of secondary importance in determining the mechanical performance of a forged component. Lally *et al.[9l* determined that particle size in the compact and grain size in the forging for low-alloy steel powders were not related. Hirschhorn and Bargainnier^[10] found that forgings produced from coarse steel powders possess greater toughness compared to those produced from fine powders. However, it was not clear whether the difference in toughness observed was directly related to particle size or oxide content. The oxide content, which is higher in fine powdered compacts, often leads to lower interface strength and possibly toughness.

5. Compaction

The consolidation of metal powders at room temperature often under applied pressure is termed "compaction." Compaction is often performed in rigid dies made of tool steel or cemented carbides. Pressures ranging from 70 to 700 MPa (10 to 100 ksi) are often used. The compacts so produced, called "green compacts," are strong enough so that they can be safely ejected from the die and handled by conventional means. These compacts are porous and have a lower density (green density) than cast or wrought parts of the same metal.

There are several methods available for the compaction of metal powders (Table 1), and selection of a particular method

Fig. 3 Ultimate tensile strength versus sintering time for different values of $1/R$. [8]

depends on the type of powder, powder composition, size and shape, as well as the number of parts to be compacted. Most of the compaction methods involve application of pressure on the powder. The basic variables in pressure compacting are:

- Method of compaction
- Compaction pressure, temperature, and time
- Speed of compacting
- Compacting atmosphere
- Lubricant and other additions
- Die design

Compaction of P/M parts is intended to produce a reproducible volume and mass from part to part to ensure proper dimensional control within stringent tolerance specifications. Also, compaction should achieve uniform density within each part so that uniform shape change occurs during sintering, thus leading to homogeneous properties. The densification process produced by compaction should avoid creation of defects such as laps, folds, and internal and surface cracks. Mass control in the preform is critical during compaction to produce required density and defect-free parts, as opposed to dimensional control of the final part shape, which is achieved during forging.

In preform fabrication by compaction for further forging, it is necessary to deviate from conventional compaction practice. For example, a large fillet radius is required during forging to prevent lap formation in a multi-level preform. This would be unacceptable in conventional P/M practice, because a large radius would produce a lower density in its vicinity. However, in forging of the preform, the flow that occurs around the radius assists in additional local densification and is thus preferred. Another example of deviation from convention is in pressing an axial taper, which can cause density gradients due to friction and radial pressure changes along the taper and is therefore unacceptable in conventional practice. In forging preforms, however, these gradients could prove significant by directing metal flow appropriately and preventing buildup of tensile stresses that can cause failure.

Table 1 Methods of Powder Compaction

With application of pressure

Unidirectional pressing Single-action pressing Double-action pressing Isostatic pressing Explosive compacting Powder rolling Powder extrusion Unidirectional Isostatic Powder swaging Powder forging **Without application of pressure** Loose powder sintering in molds

Slip casting Vibrational compacting

From Ref 7.

Fig. 4 Density distribution in compacts pressed only from the top and from both top and bottom.^[6]

Fig. 5 Distribution of densities in green nickel compact. Unidirectionally (single action) compacted at $103,000$ psi.^[6]

The equipment used for compaction, such as the type of press (mechanical, hydraulic, etc.), the tooling design and arrangement (single-action, multi-action, floating die systems, etc.), die construction (single block, sectional dies, ribbon wound dies, etc.), and friction conditions, are some of the other parameters that influence compact characteristics. Figure 4 schematically shows the density distribution in compacts pressed by single-action and double-action tooling, indicating more balanced distribution in double-action tooling. Figure 5 includes the density isocontours in green compacts produced by unidirectional pressing. The density distribution is very nonhomogeneous due to the differences in the motion and frictional characteristics of the tooling and powder interface. The magnitudes and directions of stresses during pressing metal powder compacts in rigid dies are quite complex. Vertical pressure in unidirectional compaction generates a lateral pressure

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Fig. 6 Pressure distribution during single-action die compaction with lubrication.^[11]

from the die walls, and friction between the powder and die causes nonuniform stress distributions, as shown in Fig. 6. Lubricants are therefore added to decrease friction, increase lateral pressure, and produce more uniform densification.

Triaxial compaction methods, involving application of both axial and lateral pressure by separate motions, produce shear stresses, which results in higher green density and strength. There is, however, a limit to the pressure applied to a compact contained in a nonrigid mold, as is the case in triaxial compaction because shear failures occur. Also in triaxial compaction, tool life remains a practical production problem, and the process is limited to simple and regular shapes to ensure uniform pressure and densification. Different stress paths produced by different compaction methods result in different times for compaction, density of resulting compacts, and required compacting pressure. Figure 7 shows comparisons of green density and strengths of compacts produced by different compaction methods.

"1 ksi = 6.9 MPa.

Fig. 7 Comparison of green density and transverse rupture strength of iron compacts formed by die, isostatic, and triaxial compaction methods.^[12]

In the case of compacting elements with a high slendemess ratio, it is impossible to obtain uniform density distribution throughout the whole volume of such compacts due to friction between the powder and die wall. Currently, two methods for pressing sleeves are used--double-action pressing and pressing with puncture, producing a characteristic density minimum midway along their length. Attempts to improve density distribution include a new method of powder pressing in an ultrasonic field. $[13]$

6. Sintering

As mentioned earlier, green compacts cannot be used in load-bearing applications due to their poor strength. Sintering performed under controlled temperature and atmosphere provides the required mechanical integrity to the compact. During sintering, bonding occurs via atomic diffusion between adjacent particles of powder to form a coherent mass with the required density, strength, and other properties. When two particles of powder are in contact at sufficiently high temperatures, the atoms in the surface layers become so mobile that they enlarge the contact area and group, or diffuse, into one of two dif-

Fig. 8 In the course of sintering (a), the compact acquires permanent strength while the volume shrinks (density increases) as a result of (b) elimination of most pores between particles. [14]

ferent lattices. Usually, one lattice then grows at the expense of the other and the interchange of atoms, or diffusion, is more pronounced as the sintering temperature and time are increased.

In the beginning of the sintering process, interatomic bonds are established between adjacent surfaces, and necks grow by the movement of atoms from the surface and bulk of the particles toward the necks. Plastic or viscous flow may also occur with more massive diffusion, reducing the size of pores (Fig. 8). Shrinkage in achieving a certain sintered density is greater for lower green densities. If phase changes occur during heating, shrinkage may be negligible, or growth may even occur. The formation of the common interface between two particles or a particle and a crystal block is achieved by one, or a combination of the following, mechanisms—viscous or plastic flow, evaporation and condensation, volume diffusion, and surface diffusion. Time cycles for sintering and the temperature of the furnace are determined by the composition of the powders involved and by the properties desired in the finished product. In addition, chemical homogeneity is developed by diffusion of alloying elements and the alloy itself chemically refined by reduction of metal oxides, further enhancing mechanical properties. Prolonged heating causes grain boundaries to migrate, grains to grow, and stabilizes remaining pores, resulting in weakening of the part.

Sintering is a complicated heat treating operation because it not only includes most of the well-known problems involved in the heat treatment of solid metals, but also several that are peculiar to the sintering mechanism alone such as dimensional growth or shrinkage, the effects of internal and external gaseous reactions, and the alloying of metals in a solid form. There

Fig. 9 Tensile strength of compacts of six grades of iron powder, sintered 1 hr at 1100 °C as a function of compacting pressure. $[6]$

Fig. 10 Density of compacts from six grades of iron powder, sintered 1 hr at 1100 °C as a function of compaction pressure.^[6]

are a variety of sintering equipment, and details on the different sintering furnaces can be found in Ref 1, 6, and 7. Typically, the furnaces have a preheat (drying or burn-off) zone, a high-heat (sintering) zone, and a cooling zone.

The micromechanics of the sintering process, such as the mechanisms of crystal growth, orientation in recrystallization and grain growth, structure and behavior of grain boundaries, self-diffusion, nucleation and allied phenomena, the different models developed to represent sintering process such as the microstructural model, kinetic models, single- and multi-phase models, finite-element models, the driving force, material transport mechanisms, plastic flow considerations, etc., are discused in the literature.^[1,6,7,15,16] Finite-element models for

Fig. 11 Tensile strength of compacts from six grades of iron powder, sintered 1 hr at 1100 °C as a function of sintered den $sity$ [6]

Fig. 12 Rockwell F hardness of compacts from six grades of iron powder, sintered 1 hr at 1100 °C as a function of sintered density.^[6]

Fig. 13 Rockwell F hardness and microhardness of iron powder compacts as a function of density, expressed as percent of solid density.^[6]

Fig. 15 Brinell hardness of compacts from carbonyl iron powder pressed at 76, 138, and 386 MPa, sintered for 2 hr as a function of sintering temperature.^[6]

modeling compaction and sintering processes have been developed, one of which is by Dawson et al.,^[17] are covered in more detail in Part III.

Solid-state sintering is most common in conventional P/M processing, because density change can be minimized and part dimensions can be tightly controlled. Liquid phase sintering is normally not used for steel powders, because oxide reduction in the solid state is possible. Other variations of sintering include loose powder sintering, where low porosity is not critical, slip casting of metal powders similar to slip casting of ceramics, and infiltration, in which the pores are filled with liquid metal or alloy.^[6]

Fig. 14 Elongation of compacts from six grades of iron powder, sintered 1 hr at 1100 °C as a function of sintered density.^[6]

Fig. 16 Effect of sintering conditions on physical properties is strongly influenced by compacting pressure. Loosely heaped or slightly compressed powders, for example, show a rapid increase in density and mechanical properties, whereas densities of powders compacted at very high pressures tend to decrease with increased sintering temperature.^[1]

7. Effect of Sintering on Mechanical Properties

In the early studies of sintering, mechanical properties of sintered compacts were represented as a function of compacting pressure, sintering temperature, and sintering time, together with the determination of sintered density. Mechanical properties such as tensile strength and elongation also increase with increased density, compaction pressure, sintering temperature, and sintering time. Increasing sintering time does not effect further increase in strength, but ductility continues to increase due to grain growth.

Table 2 Tensile Properties of Slag-Containing P/M Type 316L Stainless Steel

	Yield strength $\sigma_0 \pm \sigma$, MPa			Tensile strength, $UTS \pm \sigma(MPa)$			Fracture elongation, $\frac{3}{0} \pm \sigma$		
		Cold	Cold		Cold	Cold		Cold	Cold
Material									As extruded worked 40%worked/annealed As extruded worked 40%worked/annealed As extruded worked 40%worked/annealed
A (reference)	294 ± 12	785 ± 35	270 ± 15	642 ± 20	910 ± 62	638 ± 1	51 ± 3	20 ± 3	68 ± 1
	267 ± 31	599 ± 18	289 ± 6	586 ± 15	694 ± 6	611 ± 0	$47 + 1$	42 ± 2	68 ± 3
C (surface oxides)	308 ± 14	\cdots	\cdots	667 ± 30	\cdots	\cdots	$44 + 4$	\cdots	\cdots
D (Ex. Al ₂ O ₃)	260 ± 103	645 ± 8	310 ± 5	684 ± 9	750 ± 8	636 ± 0	$49 + 1$	$33 + 4$	$56 + 3$
E (Large furnace slags)	252 ± 102	661 ± 46	288 ± 6	672 ± 10	758 ± 17	624 ± 2	$44 + 4$	29 ± 0	66 ± 3
F(Small furnace slags)	274 ± 45	585 ± 20	318 ± 4	667 ± 7	712 ± 5	628 ± 0	41 ± 0	39 ± 1	64 ± 1
From Ref 22.									

Fig. 17 Effect of oxygen level on the toughness of 0.5%Ni-0.5%Mo steel powder forgings. [19]

Figures 9, 10, and 11 illustrate the relationship of tensile strength versus compacting pressure, density versus compacting pressure, and tensile strength versus density, respectively, as determined by Squires' method.^[18] Tensile strength and density increase with increased compacting pressure, as does tensile strength with increasing density. Powders with rough particle surfaces develop higher levels of sintered strength. Figures 12 through 14 illustrate other relationships such as hardness and microhardness, elongation, and impact resistance versus density, respectively. Rockwell hardness increases with increased density, whereas microhardness remains constant. Figure 15 shows the relationship of Brinell hardness versus temperature for different pressures. Higher compaction pressures produce higher Brinell hardness, but at higher compaction pressures hardness decreases in the temperature range of 300 to 500 °C before increasing further.

Further details on the relationships of properties in sintered compacts of iron and steel powders, effects of alloying, sintering atmosphere, etc., are discussed in Ref 6 (p 401-420). The shrinkage mechanisms affect sintered density, apart from particle size, briquetting pressure, and plasticity of powder. This in turn, along with sintering time and temperature, contributes to the physical properties of the sintered compact (Fig. 16).

8. Effect of Oxygen Content and Surface Oxidation

Oxygen levels present in the form of oxides act as contaminant particles and are detrimental to fracture-related properties such as toughness. Oxide films at the grain interface are more deleterious than discrete oxide particles or inclusions, because they provide a continuous weak path for crack propagation. Figure 17 shows that the toughness of powder forged low-alloy steels increases with decreased oxygen levels. For steel powders, it is recommended that during sintering the oxygen levels be kept below 500 ppm, preferably below 300 ppm. $Crown^[20]$ reported that a ductile-brittle fracture transition occurs at approximately 650 ppm oxygen for 4640 steel powder forgings. Higher sintering temperatures result in lower oxygen levels. The evaluation of fatigue resistance with respect to oxygen content is more complicated. The fatigue resistance generally increases with decreasing oxygen content up to a certain point, beyond which no further increase in fatigue limit has been observed. It has been proposed that oxide size and size distribution at levels less than 300 ppm are below the level that affects fatigue crack initiation or growth rates.[21]

For the properties of P/M parts to be comparable to conventional steels, the intrinsic problem associated with slag inclusions in these materials has to be controlled. One origin of slag inclusions in P/M alloys is oxidized powder particle surfaces. Arnberg and Karisson^[22] investigated the effect of powder surface oxidation on tensile properties and impact strength of a martensitic steel produced by hot isostatic pressing. Table 2 reflects the variation of properties with oxygen content. The anomally of an alloy with 150 ppm oxygen was attributed to experimental error. The decrease in ductility and impact strength was found to be closely associated with the density of slag particles on the powder particle boundaries. However, yield and tensile strengths are relatively unaffected by oxidation. Therefore, it is critical to control and limit the amount of oxidation by choosing appropriate temperatures and processing.

9. Conclusions

The sequence of operations in the classical P/M processing method was described and some key parameters in the stages of powder preparation, compaction, and sintering were identified, and their effects on the resulting sintered compact for subsequent forging were discussed. There are a variety of methods for powder production, blending, mixing, compacting, and sintering, and therefore, a judicious combination of the above processes is required to produce the desired sintered compact. Particle size, shape, distribution, composition, and powder characteristics contribute in the preparation of compacts. Lubricants, compaction tooling configurations, friction conditions, flow, and the compaction equipment have to be carefully selected to produce a compact for structural integrity, required density distribution, homogeneity, and strength. Sintering process parameters such as equipment, sintering time, sintering method, and the reactions during sintering have to be selected and controlled to remove the admixed lubricant, provide adequate densification and strength, produce the required microstructure, and control the dimensions of the compact.

The subsequent parts of this review details forging parameters and their effect on part properties (Part II), and Part HI reviews some of the analysis methods for powder forging processes.

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