An Analysis of Appraoches to High-Performance Powder Metallurgy

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Abstract

High performance comes with full density. Full-density, net-shape water-atomized powder compacts have been demonstrated for 40 years. Every few years there is a new invention that offers a new technology to make powder metallurgy competitive with wrought materials. The fact that full-density processes have long been known indicates the fundamental barriers are not densification; often they are cost, shape complexity, or tolerance issues. This presentation analyzes the response of water atomized steel powders to densification via changes in particle size, strain, stress, strain rate, temperature, and other basic parameters to map the conditions required for full-density in compaction or sintering. Economic criteria are laced with dimensional control and shape complexity issues, showing that densification is not the barrier. In other words, current efforts are solving the wrong problem. Our research shows the barriers come from holding tolerances in a cost-effective process in the context of long-established processing equipment.

Introduction

Without a sense of history we are doomed to repeat that history. Full-density processing has been known for coarse powders since the early 1960s [1]. The first point is that full density is not new and there is no miracle. Our intent here is to help the powder metallurgy community recall history and to be sure new options are really removing the barriers. As a point of perspective, one report shows press-sinter parts fabricated to 100% density by novel compaction technologies by IBM in 1964 [2].

This presentation reviews the basics of full-density processing for ferrous alloys. Some important first concepts are presented by performing a contrast and comparison of classic press-sinter powder metallurgy options with alternatives, including dynamic compaction, high temperature sintering, high pressure compaction, liquid phase sintering, and small particle sintering. This analysis provides an overview of the densification rates, costs, productivity, tolerances, and barriers for these options.

In basic textbooks [3], we teach that there are fundamentally three processing options and that all practical technology is a form of one of those options, as outlined in Fig. 1.

In each of these paths there are many subdivisions. For example, in sintering densification the powder can be a large prealloyed particle that undergoes softening

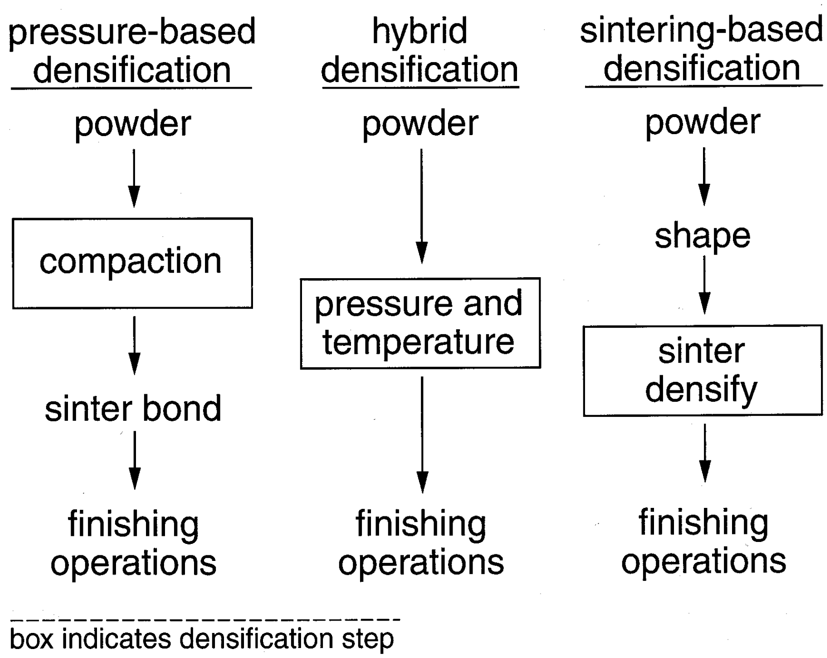


***Figure 1**. Three fundamental routes to full-density powder compacts based on densification using pressure at room temperature, simultaneous temperature and pressure, or densification in sintering. Each has its advantages, costs, and problems.*

and rapid densification as the solidus temperature is approached. This is known as supersolidus liquid phase sintering. Such a process is used in tool steels, stainless steels, nickel superalloys, and even bronze. An alternative would be to start with smaller particles that sinter densify because of the capillary stress. Such a process is common in powder metal injection molding (PIM) technologies for steels, stainless steels, electronic alloys, nickel, and copper. Others might be the contrast between the near instantaneous densification of hot powder forging and the slower, creep densification obtained in hot isostatic pressing. Here there is a fundamental difference in the strain rate reflecting the relative stress versus the inherent strength of the material (at temperature). Creep processes are low strain rate events that take a few hours for densification because the applied stress is low compared to the material strength at that temperature. Finally, full-density compaction technologies predominantly can be separated based on the rate of pressurization, with lower pressurization rates requiring higher pressures. Dynamic compaction simply is applied so quickly that the powder heats, softens, and densifies faster. We have found the unifying parameter in compaction is the total work (deformation and heating).

Thus the full-density subdivisions are as follows:

- **pressure-based densification**

1) **low strain rate**, high pressures
   sometimes called cold sintering
2) **high strain rate**, shock wave compaction
   called dynamic or high velocity compaction

- **pressure and temperature based densification**
  1) **lower temperature, higher stress**, yielding
     seen in hot powder forging, hot extrusion, spark sintering
  2) **higher temperature, lower stress,** creep
     seen in hot pressing and hot isostatic pressing

- **sintering-based densification**
  1) **small particles**
     used widely in carbides, refractory metals, and injection molding
  2) **large particles**
     mostly reliant on liquid phases, such as supersolidus sintering.

Sintered steels are used in large quantities because of the inherent low cost, good shape complexity, acceptable tolerance capability, and high levels of productivity. In general, a coarse powder is pressed in uniaxial tooling and then sintered with little dimensional change. To increase the final density requires either more pressure in compaction or a higher temperature in sintering. Dimensional control in the sintered product is held to within ± 0.025 mm of specification, with better control in the radial features and poorer control in the axial features. Table 1 shows some example strengths obtained from the sintered products, illustrating the combined effects of time, temperature, and composition. Both density and strength are listed, showing how the two are linked. In repeat tests these strengths typically exhibit a scatter of ±35 MPa.

Statistical analysis of the data shown in Table 1 helps defend the search for full-density powder metallurgy technologies. Strength (performance) had a very significant correlation to density (confidence over 99.9%). Higher temperatures, longer times, and higher densities contribute to strength, and the copper alloys tend to be better than the nickel alloys. The highest strengths come from the highest densities.

The CISP program at Penn State has been collecting data on the options to allow for an intelligent contrast and comparison by the member companies. Table 2 shows the routes under investigation and a synopsis of the progress in moving toward full density powder metallurgy compacts.

Table 1. Sintering Temperature Effects on Properties of Fe-2Ni-0.8C and Fe-2Cu-0.8C Steels

Alloy	Sinter Temperature °C	Sinter Time h	Density g/cm^3	Strength MPa
Fe-2Ni-0.8C	1120	0.5	6.70	243
Fe-2Ni-0.8C	1200	0.5	6.80	283
Fe-2Ni-0.8C	1300	0.5	6.75	338
Fe-2Ni-0.8C	1120	2	6.69	288
Fe-2Ni-0.8C	1200	2	6.77	316
Fe-2Ni-0.8C	1300	2	6.70	394
Fe-2Ni-0.8C	1120	0.5	7.32	345
Fe-2Ni-0.8C	1200	0.5	7.37	429
Fe-2Ni-0.8C	1200	0.5	7.30	461
Fe-2Ni-0.8C	1120	2	7.35	440
Fe-2Ni-0.8C	1200	2	7.25	479
Fe-2Ni-0.8C	1300	2	7.25	494
Fe-2Cu-0.8C	1120	0.5	6.70	329
Fe-2Cu-0.8C	1200	0.5	6.70	365
Fe-2Cu-0.8C	1300	0.5	6.67	364
Fe-2Cu-0.8C	1120	2	6.71	320
Fe-2Cu-0.8C	1200	2	6.67	437
Fe-2Cu-0.8C	1300	2	6.63	400
Fe-2Cu-0.8C	1120	0.5	7.26	465
Fe-2Cu-0.8C	1200	0.5	7.20	544
Fe-2Cu-0.8C	1300	0.5	7.17	584
Fe-2Cu-0.8C	1120	2	7.30	536
Fe-2Cu-0.8C	1200	2	7.22	613
Fe-2Cu-0.8C	1300	2	7.17	524

Table 2. Efforts in Full-Density Press-Sinter Powder Metallurgy

Approach	Equipment	Advantages	Disadvantages
high pressure compaction	150 ton tooling	standard press standard furnace	tool cost, design tool life, wear
high velocity compaction	small gas gun	controlled energy simple tooling	simple shapes long cycle time
small particle sintering	1650°C pusher 2000°C belt 2000°C vacuum 1400°C hydrogen	standard press builds on PIM	agglomerated powder powder cost high temperatures special polymers
liquid phase sintering	2000°C vacuum 1400°C hydrogen	coarse powder standard pressing	warpage temperature control
pressure-temperature creep	hot isostatic press	established process	must sinter first slow cycles high costs
pressure-temperature yielding	pneumatic isostatic forging	no tooling fast cycles many parts per run	rare equipment high starting density poor understanding

Contrast and Comparison of Full-Density Options

Pressure-Based Densification

The first option is based on pressure. A powder starts at the apparent density and can be pressed to higher densities with higher pressures. Two subdivisions exist under this option depending on the strain rate - a low strain rate such as seen in die compaction amounts to about 10 1/s while dynamic compaction is more in the 1000 1/s range. To drive the higher strain rates requires a high pressure applied over a short time period, and for ferrous systems we are typically looking at peak pressures of 500 MPa (to drive a strain rate of 10 1/s) and 2000 MPa (to drive a strain rate of 1000 1/s) - high pressures at slow strain rates and high pressures at high strain rates.

slow strain rates

Most metallic powder exhibit work hardening, so the rate of return - density change over pressure change - decreases as density and pressure increases. Densification is rapid at low pressures, but enormous pressures are required to press the powder to full density. Over twenty years ago Gutmanas [4] demonstrated stainless steel powders require pressures in the 2000 to 4000 MPa range (that is 20 to 40 kbar or about 300 to 600 ksi or 150 to 300 tsi). As one can imagine, the press, tool construction, cycle time, tool life, safety, and other issues are real limitations if we want to press alloy powders to full density at room temperature. One option was to use a rotary motion on the upper punch to reduce the press size while reaching the peak pressures.

We have fabricated a high pressure die compaction tool for a standard hydraulic press and have taken that tool to the 2000 MPa range with iron, stainless steel, and aluminum powders. These experiments have considered differences in particle size, particle shape, and particle purity, as well as alloying and lubrication technology. Indeed, with no lubricant it is possible to attain essentially 100% green density. We also have been working with some full-density compacts formed from novel materials such as tool steels and tungsten-copper materials. We plan to further extend the experiments to nanoscale powders, especially from refractory metals. Table 3 provides an assessment of the slow strain rate approaches to pressure-based full-density powder metallurgy.

Table 3. Assessment of Pressure-Based, Slow Strain Rate Densification

metric	**situation**
equipment cost	standard presses, but oversized for part
tooling cost	2-folder higher than traditional
tooling life	low (100,000 compacts)
piece processing cost	6-folder higher than traditional
production rates	6 per minute
shape complexity	simple shapes, single height parts, plates
tolerance capability	similar to conventional press-sinter technology, ±0.2%
first demonstrations	1960s
current maturity	in production at few companies
major barriers	automation, tool materials, tool life, lubrication, safety

high strain rates

An alternative is to rely on adiabatic heating in the compaction stage through a process known as dynamic compaction [5]. Here the pressure is delivered as a shock wave that passes through the powder compact very quickly. That shock wave heats the particle contacts, resulting in rapid local softening and flow. Some novel approaches have used repeat cycles, and densification gains have been reported with up to 10,000 repeat pressure cycles. So although the compaction occurs at room temperature, on a microscopic scale the particle contacts can be driven up to their melting points. Compaction equipment was available many years ago, but proved difficult to sustain and the early companies have all been dissolved. Today, there is no commercial success in this area, but that does not dissuade optimism and a new press is being developed for powder metallurgy. So in spite of significant research expenditures by government agencies, major corporations, and universities, the problems are many -

equipment cost
tool life
cracking of the compact from reflected shock waves
cycle times (some devices give 4 parts per hour)
inadequate equipment support
poor understanding by users and designers.

We have constructed a large experimental matrix and performed a full review of the field. Our experiments have included construction of a gas gun for higher velocity compaction and plans include working on new high velocity compaction equipment.

Further, the combination of experiments over a range of strain rates, powders, alloys, lubricants, particle sizes, and particle shapes has determined a few simple rules: i) irregular particles are best, ii) bimodal powders are best, iii) coarser powders with higher apparent densities are better, iv) traditional die wall lubrication is adequate, and v) the unifying parameter is total energy. Table 4 summarizes the situation.

Table 4. Assessment of Pressure-Based, High Strain Rate Densification

metric	**situation**
equipment cost	high, at least 2-fold more than conventional
tooling cost	3-fold or more higher
tooling life	reported demonstrations to 200,000 parts
piece processing cost	unknown in production, probably 3-fold over conventional
production rates	10 per minute
shape complexity	modest, mostly flat plates and tubes, promise of 2-level
tolerance capability	unknown
first demonstrations	1970s
current maturity	research status, failed in earlier pilot-scale demonstrations
major barriers	equipment repair, costs, controls, design rules

Pressure and Temperature Based Densification

Almost all engineering material soften on heating. Consequently, the yield strength falls with heating to a point where an applied pressure exceeds the materials deformation resistance. At higher stresses, deformation is by plastic flow. In powder forging the strain rates are high and hard tooling is required. Spark sintering usually relies on slower strain rates, but can still have fast cycles. For slow strain rates, deformation is by creep and higher temperatures are required to induce significant diffusion, but stresses are lower. Hot pressing, hot isostatic pressing, and related technologies are lower production rate options based on diffusion creep.

lower temperature, higher stress

Here powder forging is the clear benchmark for powder metallurgy. The press and sinter compact is heated, forged, and heat treated with final machining on critical dimensions. This was patented in the early 1960s. Densification is at a high strain rate where thermal softening and a high stress exceed the yield strength of the material during flow. Alternatives to powder forging include hot ceramic particles filing a die that is hit with a forging stroke and hot compacts in a hot isostatic pressing vessel, where the vessel is filled with liquid argon or nitrogen. The combination of phase change from liquid to gas produces rapid gas expansion with minimal compact cooling, giving an effective forging stroke. Various names such as quick-HIP and pneumatic isostatic forging are applied to these variants. Early demonstrations with sintered compacts produced dramatic results [6]. However, without a deformation model and clear understanding of densification events, the technology still struggles with a trial and error approach. A few units have been fabricated, but none have gone past the pilot plant level.

An alternative evaluated by CISP is the spark sintering route. First demonstrated in the 1940s, the technology came back in the early 1990s, failed, and has emerged again as a strong actor in Japan. Various names are now given to the approach, including spark plasma sintering, electric field activated sintering, and such [7]. They rely on an electrical discase through the compact or die with simultaneous pressurization, giving densification in just a few minutes. Only a few units exist outside of Japan, but there the technology has succeeded in forming sputtering targets and other high value products. CISP sent a student for one summer in Japan to work with the technology in the consolidation of functional gradient cemented carbides.

Table 5 lumps these approaches together into a generalized statement on the status.

Table 5. Assessment of Pressure and Temperature Based, Lower Temperature, Higher Stress Densification

metric	situation
equipment cost	very high, in excess of $450,000
tooling cost	moderate (often graphite)
tooling life	very low
piece processing cost	very high
production rates	fully automated demonstrations of 6 per h
shape complexity	simple shapes, disks, cylinders
tolerance capability	unknown
first demonstrations	1940s
current maturity	small scale production in Japan, pilot level elsewhere
major barriers	costs, only applied to high value materials and products

higher temperature, lower stress

Hot isostatic pressing was invented in the 1960s and reached widespread commercialization in the 1980s. There is no question as to its success in forming superalloys, tool steels, cobalt and nickel alloys, clad materials, titanium, and other higher value full-density components. From a scientific view, HIP works by diffusional creep and requires combinations of stress and temperature where diffusion is occurring. Since diffusion rates are slow, creep rates are slow, so HIP and related technologies require cycle times in the hours. Fortunately, the low stresses required for densification help reduce the containment vessel cost, making HIP affordable for many applications such as biomedical implants, jet engine components, computer peripherals, sputtering targets, wear components, and cutting tools.

Our research relies on HIP for routine densification of powder compacts. The design of the cycle and the design of the HIP preform or container are supported by reliable computer tools. As an approach to full density, HIP is well developed as indicated in Table 6. The problem is evident in terms of cost, tolerances, and cycle times, so HIP tends to better succeed with larger components. Our efforts have discovered means to remove the container and to significantly reduce the costs prior to the HIP cycle, but still that cycle is a dominant cost. Most HIP products are machined on critical surfaces, so there is less attention to exact tolerances and more attention to over sizing.

Table 6. Assessment of Pressure-Based, High Strain Rate Densification

metric	**situation**
equipment cost	high
tooling cost	high
tooling life	often single cycle
piece processing cost	ranges from \$3 per kg and up
production rates	one cycle per few hours, container may contain many parts
shape complexity	modest
tolerance capability	low, ±2% is good
first demonstrations	1960s
current maturity	very mature field, no growth
major barriers	equipment cost, cycle cost, equipment maintenance, safety

Sintering-Based Densification

Sintering densification is based on a combination of material softening and capillary stress. Small particles naturally have high capillary stresses, so they sinter densify, but coarse powders have low stresses and often prove difficult to soften sufficiently to sinter densify. One viable option with large powders is to form a liquid phase and supersolidus liquid phase sintering is very useful for prealloyed, larger powders. Thus, in sintering densification the approaches rely on small particles or the formation of a liquid phase in larger particles.

small particles

Sintering is driven by the elimination of surface energy. During random atomic motion, there is a change in atomic stability depending on location within the sintering microstructure. The Laplace equation gives the sintering stress associated with a particle system. During sintering concave surfaces are under compression and convex surfaces are under tension, and the natural tendency is for the random atomic motion to move both toward flat, stress-free surfaces over time. The change in curvature leads to a stress termed the sintering stress that puts the particle contacts under compression without an external pressure [8]. Thus, there is a natural compression or densification associated with sintering. This sintering stress increases as the particle size decreases. In powder injection molding, the typical 10 μm powder leads to a sintering stress near 0.4 MPa (about 60 psi). Smaller powders give higher stresses.

Table 7. Assessment of Sintering-Based Densification with Small Powders

metric	situation
equipment cost	same equipment as used for compaction and sintering
tooling cost	relies on standard tooling
tooling life	no data yet, should rival traditional compaction
piece processing cost	higher due to agglomeration step and sintering cycles
production rates	same as traditional press-sinter, 6 to 20 per min
shape complexity	no data yet, should be similar to die compaction
tolerance capability	lower than traditional die compaction, ±0.3% probable
first demonstrations	early work 1930s, current variants from 1990s
current maturity	laboratory scale, early pilot runs scrapped due to polymer
major barriers	building infrastructure for powders, agglomeration, design

Because of the inherent sintering stress, powder metallurgy has long used small particles to induce densification, a process widely used in sintering carbides, refractory metals, and injection molding components. At CISP we have taken the small powders produced for injection molding, and used a new emulsified polymer to die compact these powders to green densities much higher than obtained in PIM, as high as 84% of theoretical. Thus, sintering to full density is rapid without large distortions. The compaction process does create green body density gradients, so there is some loss of dimensional precision, but several demonstrations show the approach is an alternative for full-density powder metallurgy. The key attributes are as follows:

1) start with lower cost water atomized powders fabricated for injection molding
2) add a polymer for green strength and agglomeration for easy die loading
3) die compact at typical pressures
4) rely on high temperature sintering to densify.

Of all technologies, as shown in Table 7, this seems one of the most suitable for a drop-in solution to full-density powder metallurgy. The chief concerns arise from the unique polymer and the requirement for high temperature sintering. However, much of the press-sinter powder metallurgy industry has embraced this latter option. Dimensional tolerances are not great because of the shrinkage during sintering densification.

large particles

Since the sintering stress responsible for compact densification is inversely dependent on the particle size, then large particles naturally resist sintering densification. The one novel means to circumvent that problem is to rely on large, prealloyed particles using a technology invented in 1962 [9]. When a large, prealloyed powder is heated to the solidus temperature, a liquid forms inside the particle to weaken the structure. In such a case the low sintering stress combines with a weak semisolid particle to induce via supersolidus liquid phase sintering.

Supersolidus liquid phase sintering and variants based on alloy powders and liquid phases are one means to sinter to full density. Demonstration experiments at CISP with coarse water atomized steel powders resulted in compacts with more than 99% density and strengths over 700 MPa as-sintered. Ductility and densification turn out to compete with each other in these systems. An excess of liquid makes densification easy, but often the liquid will solidify as a grain boundary film that lowers ductility. Post-sintering heat treatments have been developed to eliminate such problems and Fe-Mo-Ni-B-C compositions have proven very successful. The assessment of the situation is given in Table 8.

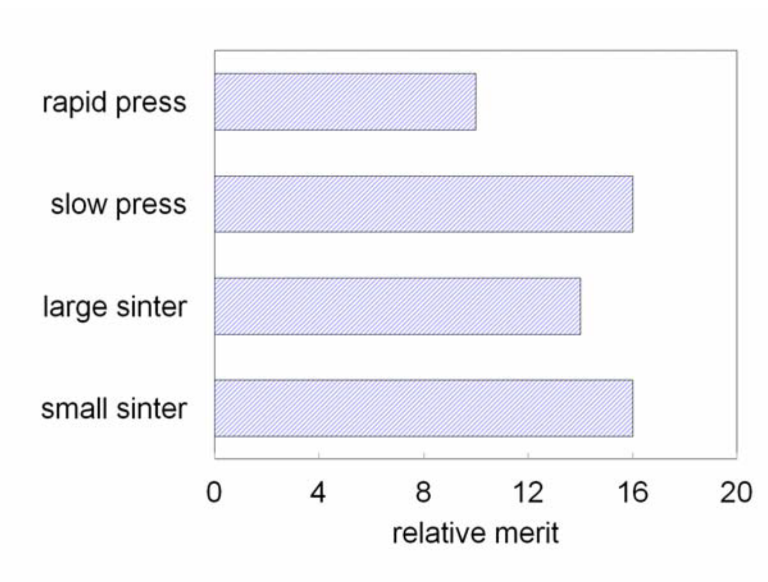
Table 8. Assessment of Sintering-Based, Large Particle Densification

metric	**situation**
equipment cost	same equipment as used for compaction and sintering
tooling cost	relies on same compaction tooling as standard process
tooling life	same as traditional powder metallurgy
piece processing cost	higher than traditional pres-sinter, especially sintering
production rates	same as standard compaction and sintering
shape complexity	generally limited to squat shapes to ensure support
tolerance capability	some warpage and distortion, ±0.5%
first demonstrations	1960s
current maturity	in production, used for several high performance systems
major barriers	dimensional control, excellent furnace control

Summary

Powder metallurgy approaches to full density abound. Generally the problem is not technology or equipment, so new approaches are not fundamentally required. The difficulty is the relation between dimensional control, processing cost (similar to conventional press-sinter technology), and shape complexity. As one company recently identified the situation - "Ideally we can find a technology that involves no additional capital cost" - and in that regard the sintering options are most attractive. High pressure compaction is not too different. Hence to summarize the compaction or sintering technologies for full density, Fig. 2 plots a diagram that shows the comparative attributes. We leave out the simultaneous heat and pressurize routes, since that would require new equipment.

Fig. 2. Comparison of pooled technology attributes for sinter and compaction routes to full density based on tolerance capability, shape complexity, processing costs, and equipment costs.



Conclusions

Powder densification to full density is possible through several variants that play on themes of particle size, sintering temperature, and compaction pressure. Today the offering of new technologies continues to grow. However in the core attributes, large differences exist in these offerings. Fundamentally, the barrier is not how to process to full density, since that has been known for many years. More typically the limitations are found in the following four attributes:

- shape complexity
- tolerances
- processing costs
- capital equipment costs.

When rationalized this way, the small particle sintering technologies coupled with high pressure compaction at slow strain rates becomes an attractive option. Sintering densification tends to have problems with holding tolerances, while the high pressure compaction is attractive except for the larger presses and higher processing costs (and questionable tool life).

Our preference is a hybrid solution for full density components based on agglomerated small prealloyed particles using new polymers, compacted in high pressure tooling using standard equipment and processing cycles. We see this as the best route to deliver full-density products, with shape, tolerance, cost, and

widespread adaptation.

Acknowledgments

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