Water Atomized Fine Powder Technology

Jack Hamill, Christopher Schade, Neal Myers*

Hoeganaes Corporation, Cinnaminson, New Jersey USA
*Pennsylvania State University, Center for Innovative Sintered Products

Abstract - Industry trends indicate the need for economical fine powder grades for a growing number of applications. Particle size, shape and percentage yields are the important characteristics associated with the manufacture of suitable powders. This work identifies our development efforts, corresponding achievements and commercial applications. Various metal injection molding binder formulations, to be used in conjunction with the fine powders, are also reviewed.
KEYWORDS: ATOMIZATION, METAL INJECTION MOLDING, METAL POWDERS, FEEDSTOCK

I. INTRODUCTION

High-pressure water atomization has proven to be a viable, low-cost process to achieve fine particle size distributions for iron, stainless and low-alloy metal powders. The economic advantages and prealloying capability provide desirable advantages over competing technologies. Previous shortcomings relative to powder characteristics, i.e. irregular particle shape, lower tap densities, oxidized surfaces, have been refined to more closely replicate gas atomized powder properties.

Internal development efforts focused on more sophisticated alloy compositions, improved melting practices and further development of water atomizing techniques. This work identified useful process control guidelines that assist in achieving desired particle characteristics. Understanding the interrelated variables was the key to our successful effort.

II. BACKGROUND

Previous work [1] identified various important aspects of fine powder production. We found two notable variables, melt viscosity and surface tension, are chiefly influenced by composition. Lower viscosity and surface tension are both thought to be elements that contribute to production of finer particle size distribution [2]. However, analysis is not conclusive as to which of these physical characteristics has a greater influence. Our investigations using select combinations of deoxidizers found little impact on particle size. Instead, we realized select combinations can influence particle shape toward a more spherical morphology along with having the ability to modify surface oxides to aid in passivation or reduced susceptibility to oxidation.

In comparison, water velocity has the most pronounced influence on particle size. Initial studies determined increasing atomization velocity, by coordinating water pressure and modifications to jet angle configuration, provided an 85% yield of minus 40 μm particles. The effect of other noteworthy variables, i.e. superheat, water/metal ratios and chamber atmosphere, did impact the outcome but to a somewhat lesser extent. After a considerable number of trials, it became apparent that many variables show strong interrelationships which often compromise each other in respect to optimizing specific particle characteristics.

III. MATERIAL CHARACTERISTICS

Results of our fine particle iron (FPI) material development effort indicate high pressure water atomization, with an optimized product yield, represents somewhat coarser particle size distribution than gas atomized powders, Figure 1. It should be emphasized, that not all particle size comparisons are truly representative. Often values are misleading because not all powder producers use the same reporting or identification practices when referencing particle size distribution. Figure 2 represents the actual laser diffraction results of water atomized 316L.

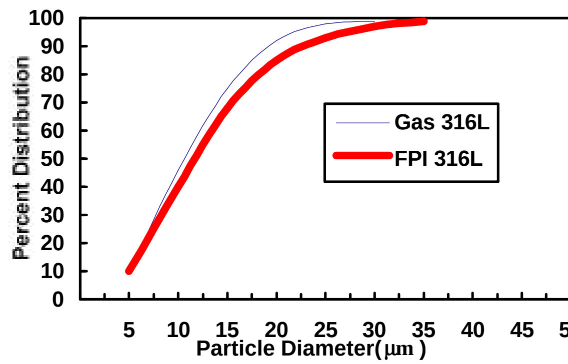

Figure 1. Water vs. gas atomized powders

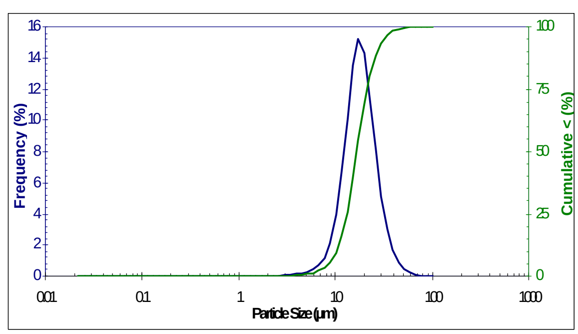

Figure 2 - Particle size distribution for 316L

Table 1 identifies typical powder characteristics for two commercial fine particle grades processed under standard production conditions.

Table 1. Typical Powder Characteristics

FPI Grades	17-4 PH	316L
D_{10} µm	10	10
D_{50}	15	14
D_{90}	22	22
Tap Density, g/cm^3	4.10	4.40
Carbon, w/o	0.03	0.025
Oxygen, w/o	0.25	0.25
Nitrogen, w/o	0.02	0.02
True Density, g/cm^3	7.67	7.87
Area, m^2/g	0.2	0.2

Particle shape is the prevalent characteristic that distinguishes water atomized materials from those produced using an inert gas media. Spherical powders generally exhibit improved metal injection molding performance relative to higher tap densities, but have a propensity to slump or distort if not adequately supported during the debinding process. Consequently, our fine particle efforts attempted to achieve near-spherical particle shape as a suitable compromise. The particles illustrated in Figure 3 represent typical water atomized material with a corresponding irregular particle shape and greater overall surface area. In comparison, Figure 4, exemplifies FPI material with shape modification having the same composition and particle size distribution.

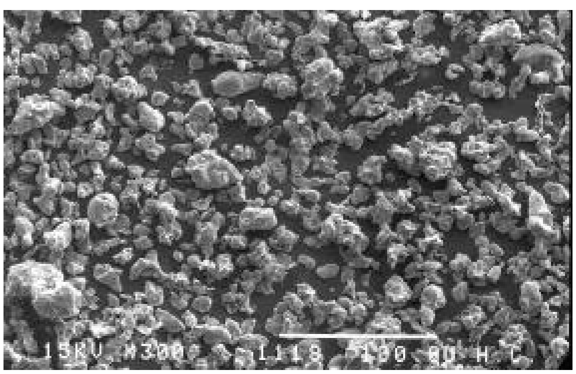

Figure 3 - Particle shape associated with typical water atomization process.

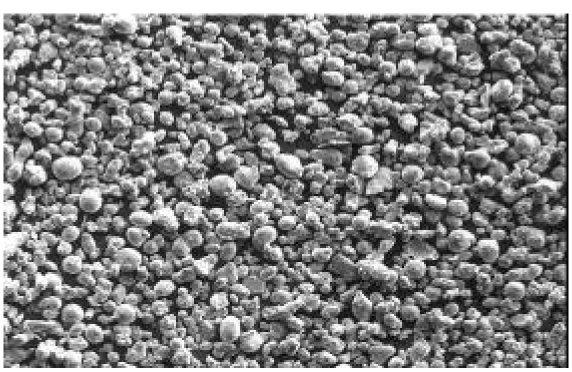

Figure 4 - Surface Modified FPI material with 45% less surface area.

The surface modification equates to a substantial reduction in surface area. Brunauer-Emmet-Teller (BET) surface analysis determined that the typical water atomized particles reflect 0.638 m^2/g, whereas the modified material exhibits 0.352 m^2/g or a 45% reduction.

Previous investigation [3] identified that surface oxides are a function of the alloying elements. Compositions that include oxides with high melting points form a hard shell around the solidified particle. Figure 5 illustrates a cross section of the modified surface using SEM. In this instance, there is an Fe_3O_4 oxide skin surrounding the particle. The structure is uniform with no appreciable amounts of internal porosity. TEM analysis indicates grain sizes are extremely fine with some that approximate the radius of the respective particle diameter.

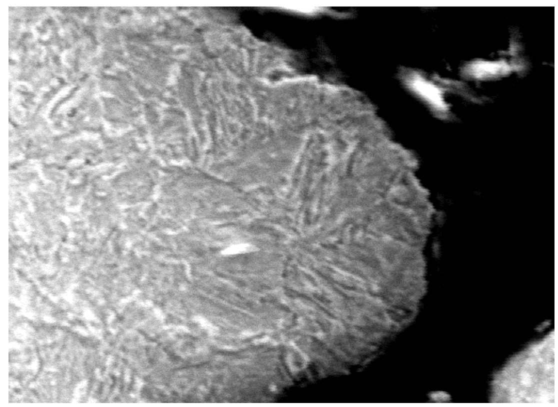

Figure 5 - SEM photo of modified iron particle cross section

Development efforts [4] determined that we could manipulate the type of oxide along with the thickness by controlling the atmosphere in the atomizing chamber. Thickness and permeability of the oxide layer can be altered to influence the surface area of the particles. Figure 6 shows the variation of surface area (measured by BET analysis) as a function of oxygen content. In all cases, atomizing conditions were held constant and the materials represented the same particle size distribution.

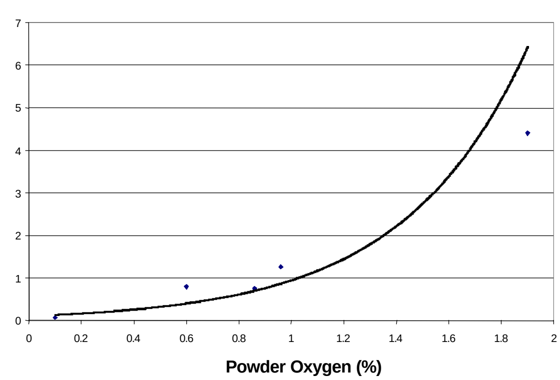

Figure 6 - Surface area versus Oxygen content.

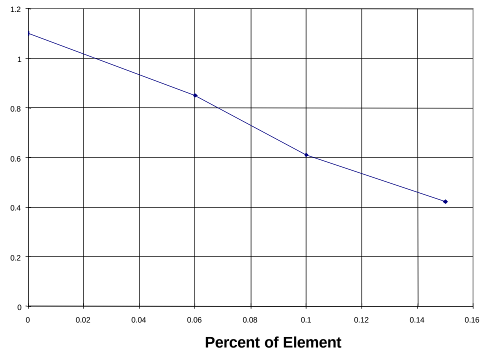

Figure 7 - Effect of oxide forming element on powder oxygen content

In addition, our investigations looked at the ability to reduce the oxygen content of ferrous powders with additions of alloying elements. The type of oxide and thickness can be modified with specific additions or combinations of alloying elements. Figure 7 shows the oxygen content of an iron based powder with small additions of a proprietary oxide forming element(s) made during the process. Without addition of the modifiers, oxygen can be as high as 1 %, whereas modification can reduce oxygen to < 0.40 %. The oxygen is primarily concentrated on the particle surface.

IV. MIM FEEDSTOCK

Compatibility between binder system and metal powder, in terms of feedstock rheology and molding parameters, is extremely important. Development work, done on our behalf at Pennsylvania State University, used both commercial, polypropylene-wax-stearic acid, and newer, unique binder formulations that were compounded into feedstocks for molding trials. All work attempted to replicate standard conditions typical of those used in MIM production environments.

The most promising of the new binder formulations includes 50% paraffin wax (Dussek Campbell), 25% polypropylene (Polyvisions ProFlow 5000), 25% polyethylene with surfactant (DuPont Fusabond) combination. Compounding trials on 316L stainless achieved 58 v/o solids loading using a Readco twin-screw unit at 160°C and 100 rpm mixing speed. Tensile specimens and assorted shapes have been molded without difficulty. Typical molding parameters reflect 170-175°C barrel and 17-25°C molding temperatures with injection speeds of 15-25 mm/sec. and 400 bar switchover pressure.

Weight variation in the as-molded condition is represented in Figure 8. The absolute weight values are somewhat different because of differences in solid loading and/or molding parameters between gas atomized powder and the FPI materials. However, the overall variation of both FPI grades compares favorably with gas atomized 316L.

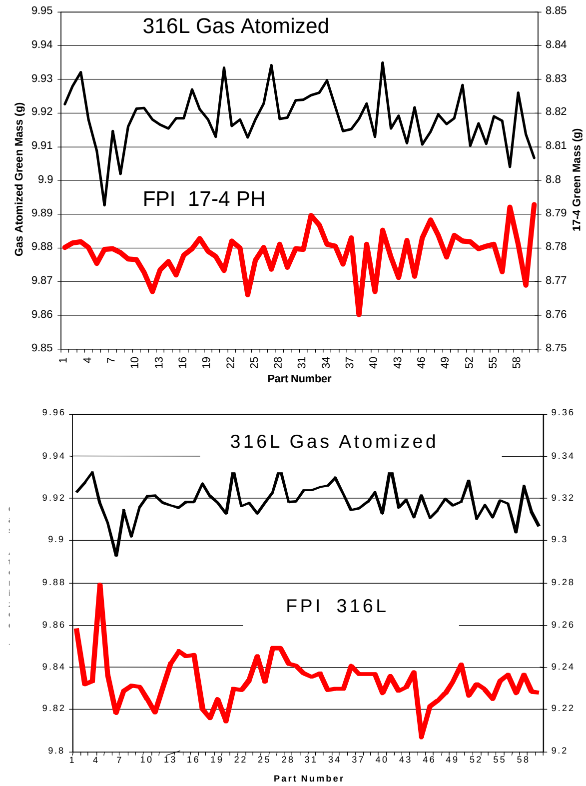

Figure 8a,b - Compares as-molded weight variation of gas atomized 316L to FPI 17-4 PH (top) and FPI 316L (btm).

The respective debinding conditions included an initial heptane immersion at 55°C for 5 hours with a secondary thermal debind incorporated into the continuous pusher furnace sintering cycle. We experienced no unusual circumstances or need for support fixturing during either step of the process. Both green and brown strengths appeared to be adequate.

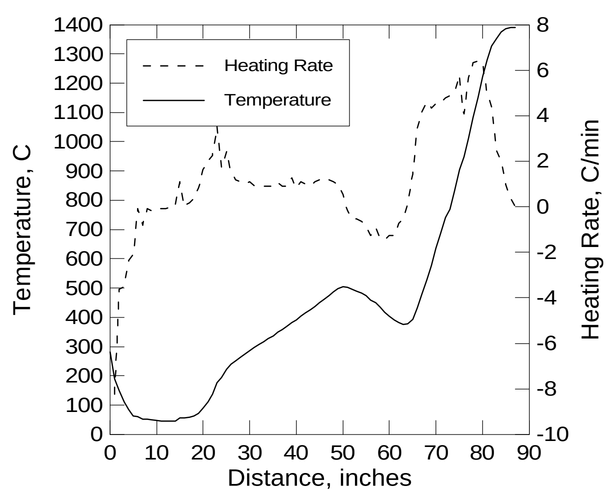

Figure 9 - Furnace profile for initial 316L sintering trial.

Sintering cycles reflect common industry conditions for both 316L (1360°C for 1 hour), Figure 9, and 17-4 PH (1300°C for 1 hour) in 100% H_2 atmosphere. We did not have sufficient opportunity to optimize either set of molding or sintering conditions for the respective materials. Microstructural indications suggest somewhat lower temperatures may enhance mechanical properties along with reducing the overall dimensional variation. The 17-4 PH solution treatment represents a one hour cycle at 1000°C in 100 % hydrogen atmosphere with a water quench. The subsequent aging cycle included 3 hours at 480°C in the same atmosphere and furnace cooled. Performance properties, Table 2, exceed those of previous investigations associated with water atomized powder [5].

Table 2 - Sintered and Heat Treated Properties

Grade:	316L	17-4 PH	17-4 PH
Condition:	Sintered	Sintered	Heat Treated
Density, g/cm^3	7.88	7.60	--
UTS, MPa	500	900	1225
Elongation, %	67	3	2
Hardness	44 HRB	28 HRC	40 HRC

We did not evidence appreciable distortion or cracking associated with either debinding - sintering processes. Shrinkage from die dimensions was isotropic for the small, thin tensile specimen measurements shown in Table 3. The relationship, although not in absolute terms, compares with results of gas atomized stainless and represents considerable improvement over other types of water atomized powders [6].

Table 3 - Shrinkage Rates for FPI 316L

Orientation	Length	Width	Height
Shrinkage, %	16.7	17.0	16.9

V. SUMMARY

Advancements in high pressure water atomization technology can now produce fine powders with unique physical characteristics. Fine particle size distributions with shape modification, without requiring additional mechanical or thermal secondary operations, provide suitable alternatives to more costly inert gas atomization processes. This has been accomplished by understanding the exacting balance and/or control of process variables required to achieve suitable product yields along with the desired performance characteristics.

Collaboration with the Center for Innovative Sintered Products at Pennsylvania State University resulted in the development of a new PIM binder formulation that complements the FPI metal powders. Favorable performance associated with the homogeneous feedstock has been achieved in respect to both compounding and molding conditions. Resultant sintered mechanical properties reflect similar values as benchmark standards referenced by the PIM industry. The isotropic shrinkage suggests reasonable dimensional control to support part production given suitable tooling factors. The ability to optimize molding and sintering parameters should further improve overall perfromance along with reducing total variation.

VI. REFERENCES

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