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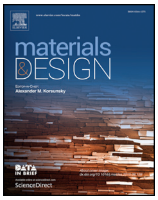

Treatment of ferrous melts for the improvement of the sphericity of water atomized powders

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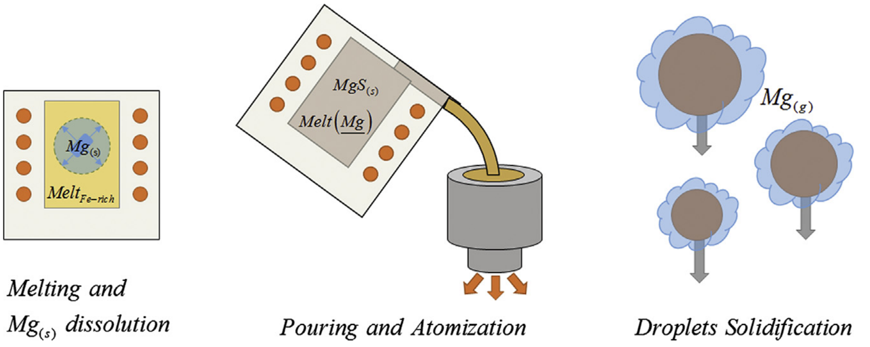
HIGHLIGHTS

- A magnesium treatment of the melt before water atomization improved the sphericity of ferrous powders.
- The Mg-treated powders have better flow, larger apparent and tap densities and contain fewer and smaller internal pores.
- Mg lead to longer solidification times by creating an insulating gas layer that renews itself during solidification.
- Mg increased the surface tension of the melt by reacting with dissolved sulfur.

GRAPHICAL ABSTRACT



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ABSTRACT

This study investigated the effect of a magnesium treatment of ferrous melts before water atomization on powder properties such as their shapes, flows, and apparent and tap densities. Three different ferrous alloys were studied, a high carbon steel alloyed with silicon, a hypereutectic cast iron, and a 304 stainless steel. All the powders that were treated with Mg were more spherical and contained fewer and smaller internal pores. All the Mg-treated powders had better flow and larger apparent and tap densities. The improved sphericity of the Mg-treated particles is caused by a larger solidification time and a smaller spheroidization time of the droplets. The larger solidification time is the result of the creation of a continuously renewed insulating Mg gas layer during solidification of the droplets. The smaller spheroidization time is a result of a larger surface tension of the melt from the reaction of Mg with dissolved sulfur. The decreased amount and size of internal porosities is also caused by the larger surface tension of the melt. This new technology is highly cost-effective and can benefit to the development of the additive manufacturing market.

1. Introduction

In recent years, the numerous advantages of additive manufacturing (AM) compared to more conventional processes have led to a significant growth of AM processes. The continuous reduction in the price of the equipment, their improved reliability and the increasing number of available processes point to a significant growth of the AM market that could almost double by 2017 compared to the market

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value of 2012, reaching about 3475 million dollars [1]. A wide range of materials can be used in AM such as polymers, composites, ceramics, and metallic powders. Some examples of AM processes are selective laser sintering (SLS), laser engineered net shaping (LENS), electron beam melting (EBM), selective laser melting (SLM), laser metal deposition (LMD), and 3D printing to name a few. Each of these processes is either classified as powder-bed or powder-fed systems. For powder-bed systems, a blade is used for spreading the powder from a reservoir to the top of the build space. Powder-fed systems use the same feedstock as the powder-bed systems, but the material is added through a nozzle and is being melted and/or deposited directly on the part that is being constructed. Even though AM provides many novel opportunities, there is a lot of development that needs to be made for larger scale production. The variation in properties from machine-to-machine as well as across machine types are issues that need to be addressed [2,3]. Different avenues are investigated, including better process controls and sensors for real time data acquisition [1], the development of physics-based models to improve the prediction of microstructures and properties and an increased amount of available alloys [2].

For both powder-bed and powder-fed systems, the ideal powder characteristics that are sought are a spherical morphology, a controlled size distribution and a smooth surface [3,4]. The main requirement being the flowability since the powders must be able to be distributed with precision and repeatability; the powder mass flow rate is a significant factor to porosity [5,6]. Indeed, these characteristics will also impact on how well the particles will pack together, which in turn influence the minimum part layer and the final density of the parts [7]. Powders with a high apparent density will provide parts with larger final densities, which will result in overall better properties [8]. For these reasons, powder feedstocks used in AM processes are typically gas atomized (GA) as the powders are more spherical and contain less oxides. However, the cost of GA powders is substantial, especially compared to water atomized powders (WA). Moreover, the required powder size distribution is specific to the AM process of interest. For instance, the preferred size distribution for the EBM process is typically between 45 and 105 μm, for powder-fed processes it is between 45 and 150 μm and for most laser AM processes the preferred size distribution is between 10 and 45 μm [5]. Powder particles smaller than 10 μm are usually removed as they negatively impact flow [5]. This additional step is costly and increases the price of GA powder feedstocks for AM applications compared to that of GA powder feedstocks for metal injection moulding (MIM) applications. Other processes were developed to produce more spherical powders, for instance plasma spheroidization which heats existing powders in plasma to increase their sphericity [9, 10]. Another technique is the liquid-solid (LS) method that takes advantage of the low wettability of metals droplets mixed with a particular solid powder that depends on the metal to be spheroidize. Spheroidization occurs as a result of the influence of interfacial and surface tension between the liquid droplet and the solid powder [11,12]. These two examples are secondary operations that necessitate the raw materials to already be in the form of powder, which increases the cost of these processes. There is also a concern of contamination with the solid powder material in the LS method as well has interrogations on the possibility of larger scale production and control over the right size distribution for various AM processes. Considering that currently, a major part of the production cost of AM parts is material related [13], economy on the production of the powder feedstocks would have a considerable impact on the cost-effectiveness of AM processes.

Some authors studied the possibility of using WA powders in AM processes since the production cost of WA powders is estimated to be 3 to 9 times lower than that of GA powders [14]. WA powders are more irregular than GA powders since the solidification rate is larger and they also contain more oxides as a result of the reaction with the oxygen in water [15]. Nonetheless, parts made with WA powders with final properties equivalent to those produced with GA powders were obtained. For instance, Pinkerton and Li [16–20] investigated the differences between GA and WA powders of 316L stainless steel and H13 tool steel using the direct laser deposition (DLD) powder-fed system. They showed that parts made with WA powders have superior surface finish, deposition uniformity and bonding between layers, compared to those made with GA powders under the same experimental conditions. In their experiments, the differences between GA and WA powders were attributed to an increased vaporization of the WA powders, a hotter melt pool and more powerful Marangoni flows. It was concluded that these differences were caused by a lower reflectivity and a greater surface area to volume ratio of the WA powders. However, the main issue was that deposition rates for WA powders were about 10 times lower than those obtained with GA powders. Differences between GA and WA powders were also investigated in powder-bed systems. Li et al. [21], using 316L stainless steel powders in a SLM process, found that GA powders densified more than WA powders and the difference was attributed to the lower oxygen content and larger packing density of the GA powders. Olakanmi [22] performed SLS trials with 5 different aluminum powders of various shapes that were atomized using different methods (air, water and gas atomized). A strong correlation between the sintered density and the bed density was established and was explained by the different degree of sphericity and amount of surface oxides. Parts made with WA powders densified less since the powder bed density was lower (apparent densities of about 1.0 g/cm^3 for the WA powder compared to about 1.4 g/cm^3 for the GA powder). Irrinki et al. [23] did SLM experiments with WA and GA 17-4 PH stainless steel powders and obtained superior densification and mechanical properties for the GA powder processed at low energy density (80 J/mm^3). They explained their results by the larger packing density and a lower oxide content of GA powders. They also found that similar results can be obtained with larger WA powders if the energy density is increased (104 J/mm^3). However, a significant variation of the properties of the parts with relatively large densities was observed. This phenomenon appears to depend on the type of powder that was used, but work to explain these results is still ongoing.

Techniques to improve the quality of WA powders for applications that require more spherical particles were developed. Seki et al. [24] and Okamoto, Sawayama and Seki [25] investigated the possibility of using high pressure water atomization (HPWA) for the production of fine ferrous powders (under 10 μm) for MIM applications. It was shown that their V-jet nozzle allowed the production of fine powders with flow properties equivalent to those produced by the carbonyl process. However, even if the powders were described as having a near spherical shape, the images that were presented in their paper also showed particles that are irregular. Moreover, as stated earlier, the fine size distribution needed for MIM applications does not apply for most of AM processes [5]. Larger particles produced with this process will probably not be spherical enough for AM applications as small particles are in general more spherical than larger ones [26]. Schade, Murphy and Walton [27] developed a process specifically aimed at the production of WA powders for AM. Although the exact process was not disclosed, they described it as a combination of HPWA to produce a high yield of fine powders and different steps to remove the more irregular particles such as classifying, dry and wet spiral separation, magnetic and frictional separation. Using such processes on a WA iron powder, they were able to increase the apparent density from 3.51 to 4.20 g/cm^3 and to improve the flow from 20.1 to 15.1 s as the number of irregular powders was lowered. However, the size distribution of the powder at the end of the process and a comparison with the size distribution of the initial atomized powder was not given. In addition, the yield after removing the irregular particles was not discussed. From the aspect ratio distribution curves included in the paper, it can be estimated that about 50% of the initial powder was removed by the different classifying techniques, which lowers the cost-effectiveness of this process.

In this paper, a novel technique for the production of ferrous WA powders for AM applications is presented. The technique uses a