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Influence of Moisture on the Properties of AlSi10Mg Powder for Laser Powder Bed Fusion

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This study explores the behavior of aluminum powder in laser powder bed fusion (LPBF) for additive manufacturing. Focusing on AlSi10Mg powders, the research investigates the effects of environmental conditions, such as temperature and humidity, on crucial properties like moisture content, fluidity, density, and agglomeration. Comprehensive tests, including particle size analysis and fluidity assessments, were conducted. Results showed increased flow time in the Carney funnel fluidity test after storage, but flow properties were maintained under high-temperature, low-humidity conditions. High humidity led to recurrent agglomerate formation. Moisture content analysis aligned with literature findings. Conclusively, these tests provide a reference for material acceptance and aid in supplier comparison for improved machine fluidity. While temperature and humidity impact aluminum oxide formation, humidity's greater relevance affects powder fluidity and quality. Other factors mentioned may cause permanent damage to the aluminum powder.

Keywords: *Laser Powder Bed Fusion, AlSi10Mg, moisture, humidity, Additive Manufacturing, powder.*

1. Introduction

In recent years, the rapid advancement of metallic additive manufacturing methods has propelled significant discussions regarding methodologies, parameterization, and various factors influencing the process. These factors encompass intricate aspects such as the interplay between powder and laser energy absorption, subsequent melt pool formation, effects associated with particle size distribution affecting densification and laser absorption, and the unique material composition characteristics[1]. Among the range of additive manufacturing techniques, Powder Bed Fusion (PBF) stands out, which offers distinct advantages such as tight fusion and heat-affected areas, effectively minimizing energy absorption losses compared to conventional manufacturing techniques. PBF integrates high energy density capabilities with design and manufacturing flexibility in the chambers, enabling the creation of complex 3D multi-material structures with open interface connections and precise material distribution[2].

With a specific focus on aluminum, the energy intensive molten pool formation process relies predominantly on the laser, particularly in the context of powder bed fusion. However, utilizing aluminum powder in this technique presents distinct challenges due to its unique characteristics, requiring meticulous control over the powder's properties[3]. As emphasized by Muñiz-Lerma et al.[3], primary aspects that require control include the sphericity of the powder, fundamental for the flow and densification of the powder, the particle size distribution that influences the apparent density of the powder, and the presence of satellite particles, along with critical physicochemical properties, which interact on the surface with particle behavior and friction.

Other critical considerations investigate the size distribution and absorption characteristics of the laser[4]. Aluminum,

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characterized by one of the lowest emissivity values among metals, presents challenges in the laser focus zone, leading to increased reflectivity resulting from the physical-chemical characteristics of the metal. Solving this problem involves carefully adjusting the powder roughness and particle size distribution[5]. In the laser powder bed fusion system, an integral step involves uniformly depositing successive layers of powder and subsequently exposing them to laser radiation, whereby the surface interacts through molten pools to construct an enclosing surface[6].

To guarantee the integrity and resistance of the final product, special attention is paid to the fluidity and densification of the powder layers[7]. Achieving higher flow requires powders with high fluidity due to gravitational forces, accentuating the critical role of factors such as particle size distribution, cohesive strength influenced by water adsorbed on the surface, and interparticle friction affected by surface characteristics[8,9]. Moreover, moisture content plays a pivotal role in determining flowability, necessitating adherence to specified conditions outlined in standard operating procedures[10].

In the domain of aluminum alloy, AlSi10Mg stands out as a low melting point alloy with a distinctive solid-liquid characteristic[11]. Notably, its melting point closely aligns with the eutectic composition of 12.6% silicon by mass at 577 ºC, resulting in minor contractions during solidification. This solidification process involves key elements such as Al and Si, while Mg forms dispersoids contributing to enhanced mechanical strength[12].

The main objective of this study is to empirically shed light on the impact of temperature and humidity on powder, with the aim of providing a comprehensive guide for powder analysis in laser powder bed fusion applications. This study ambitiously seeks to cover equipment, necessary precautions, and pertinent literature on material storage, offering a meticulous step-by-step outline to optimize results in the laser powder bed fusion manufacturing process.

2. Materials and Methods

The study utilized AlSi10Mg powders, specifically selected due to their significance in laser powder bed fusion applications. To examine the impact of different environmental conditions on these powders, a controlled environment was maintained for a duration of 5 days, with the atmospheric temperature and humidity was 19.5°C and 75%, respectively. The experimental conditions were established using a Design of Experiments created in Minitab, taking into account both temperature and humidity. The temperature settings were determined based on previous experimental studies, specifically those conducted by Cordova et al.[13].

1. High Temperature and High Humidity (50 °C, 60% Humidity): The materials were placed in a sealed muffle furnace set to a high temperature of 50 °C and high humidity of 60% to observe their behavior under elevated thermal and moisture conditions.
2. Low Temperature and High Humidity (23 °C, 90% Humidity): The materials were stored in a sealed box with water content to maintain a low temperature of 23 °C while exposing them to high humidity conditions of 90%, providing insights into their stability and properties at ambient temperature with high moisture levels.
3. High Temperature and Low Humidity (50 °C, 17% Humidity): The materials were exposed to a high-temperature environment of 50 °C with low humidity of 17% in a muffle furnace. This condition aimed to investigate the powders' responses under hightemperature conditions with reduced moisture levels.
4. Low Temperature and Low Humidity (23 °C, 29% Humidity): The materials were stored in an environment maintained at a low temperature of 23 °C with low humidity of 29%, achieved using a desiccant containing silica gel. This condition was designed to mimic a controlled environment with moderate temperature and minimal moisture content.

These controlled tests were performed to evaluate the powder's stability, flowability, and other pertinent properties in varying environmental conditions, providing valuable insights into their behavior and characteristics relevant to laser powder bed fusion applications. Specialized equipment and instruments were used to ensure precise data acquisition using sensor brand Mi temperature and Humidity Monitor 2 with Bluetooth monitoring hour by hour. The experiments were conducted multiple times to ensure reproducibility with variations inferior to 2°C and equal or less of 2% of humidity. The collected data was further analyzed and processed to draw meaningful conclusions and support the objectives of the study.

The particle size distribution was investigated using a HORIBA LA-930 analyzer, which employed laser scattering to measure the powder's distribution homogenized in Ethylene glycol, according to ASTM B822[14]. Additionally, pycnometry by helium was performed to measure the skeletal density by means of a Micromeritics AccuPyc 1330 equipment, following the ASTM B923-22[15]. Among the most important aspects of LPBF (Laser Powder Bed Fusion) are flowability using the Carney funnel and apparent density. The methodology applied to measure these properties is explained in the ASTM B417[16] and ASTM B964[17] standards. The 50-gram sample was tested five times for flow rate and apparent density to obtain an average of the results. The same test was reproduced after the exposition of the four ambient conditions previously described.

The powder was embedded in resin, subsequently sanded, polished, and coated with gold to evaluate its internal characteristics. Scanning electron microscopy (SEM) and the energy-dispersive X-ray microanalysis system (EDS) were employed for observation. The equipment used for this analysis was the FEI Quanta 400 model with EDS Oxford Instruments model 6650.

After obtaining the four different moisture characteristics, the flowability and apparent density tests were repeated for each of the four conditions using the same parameters as previously explained.

Three additional tests were conducted to evaluate the influence of moisture. The first test aimed to assess reflection. To accomplish this, four pellets measuring 20 mm in diameter and 3 mm in thickness were manually pressed at 50 tons and then examined using a Shimadzu

UV3600 Spectrophotometer. The spectrophotometer was utilized to obtain the light reflection within the range of 900 to 1200 nm, which corresponds to the wavelength range of the optical fiber laser used in LPBF.

The second test conducted was the moisture test, performed using the Karl Fischer method with a Mitsubishi Chemical Analytech CA-200 Moisture meter and VA-200 Vaporizer equipment. This method is in accordance with ASTM E203-16 standard[18]. The Karl Fischer reaction, which involves the reaction of iodine with water, is utilized to accurately determine the moisture content. This method is suitable for measuring moisture levels above 25 ppm.

Raman spectroscopy analyses were also carried out, in which the samples were excited using a laser with wavelength 633 nm with controlled power to avoid heating effects, and the scattered light was recorded by a spectrometer HR 800 Evolution micro-Raman - AFM Horiba equipped with a 600 gr/mm grating. Each spectrum consisted of an average of five accumulations of 60 s. The data obtained were normalized according to the Al-Al band (293 cm-1). In this study, the selection of the wavelength was strategically undertaken with the objective of minimizing the maximum reflectivity. This decision was influenced by the observed trend in the results, which clearly demonstrated that higher wavelengths were associated with elevated levels of reflectivity. The significance of this wavelength choice lies in its potential to mitigate reflectivity, a crucial factor in the context of our research. Our findings emphasize the nuanced relationship between wavelength and reflectivity, underscoring the importance of a deliberate wavelength selection in achieving desired outcomes in optical applications.

3. Results and Discussion

In the particle size and distribution analysis, it was possible to observe a distribution ranging from 8 to 200 microns, with the D50 value at 51 microns (Figure 1). In our experimental approach, all tests were conducted using the powder in its original state, as supplied by the manufacturer. This powder was specifically recommended for use in the Laser Powder Bed Fusion (LPBF) process. Adhering to the supplier's guidance ensured consistency and reliability in our testing conditions, emphasizing the importance of using materials in their as-received state to maintain the integrity and accuracy of our study. This standardized approach contributes to the validity of our experimental procedures and enhances the relevance of our findings to real-world applications of LPBF technology.. According to ISO ASTM 52907[9], this process, although simple and highly accurate, has limitations because the measurements must be performed on isolated particles, requiring a liquid suspension. Additionally, it is necessary to assume that the particles are spherical when calculating volumes.

During the conducted pycnometry, which encompassed five separate trials, an average density of 2.6583 g/cm^3 was determined. The margin of error was calculated to be within +0.0066 g/cm^3 and -0.0041 g/cm^3.

During the Carney funnel flowability test, it was observed that the newly opened powder, as provided by the distributor, flowed evenly once the box was opened. However, after treatment, even at low humidity, the flow time tended to increase. Notably, when the newly supplied powder was subjected to high temperature and low humidity treatment, it maintained the same flow properties as before (Figure 2).

The apparent density measured by the Carney funnel did not show significant variation during the tests, maintaining an average of 1.37 g/cm^3. On the other hand, Weiss et al.[19] observed the effect of humidity on AlSi10Mg exposed to 80% humidity at 50 °C for 14 h. This effect of higher humidity could potentially be attributed to the agglomeration caused by satellite particles due to their high surface energy, generating dust aggregation. Unfortunately, the particle size distribution of the powder was not evaluated in this article for direct comparison.

Using the Karl Fischer method, we successfully measured the moisture content of the powder under four distinct conditions, as illustrated in Figure 3. The results indicate

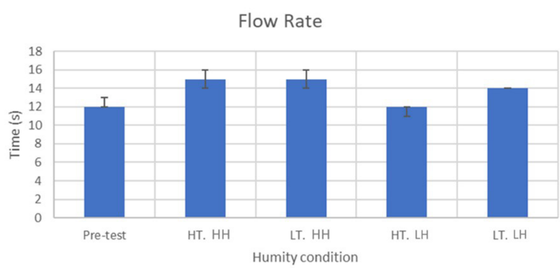

Figure 2. The average flow rate using the Carney funnel. HT: high temperature, LT: low temperature, HH: high humidity and LH: low humidity.

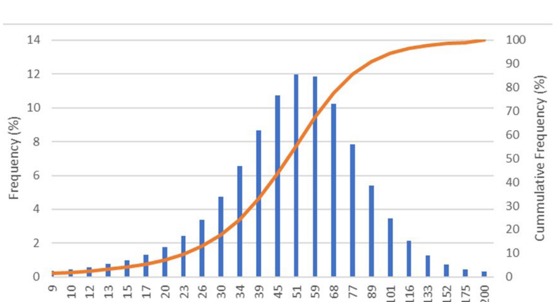

Figure 1. AlSi10Mg atomized powder as built diameter vs frequency.

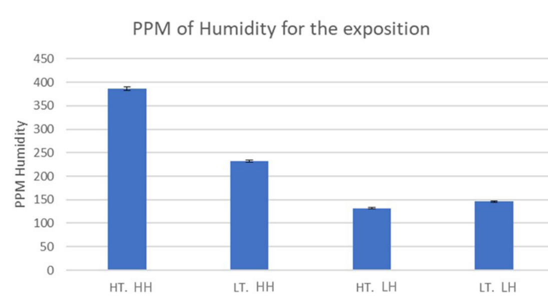

Figure 3. Relation between the humidity and temperature. HT: high temperature, LT: low temperature, HH: high humidity and LH: low humidity, and 1% error in the equipment measurement.

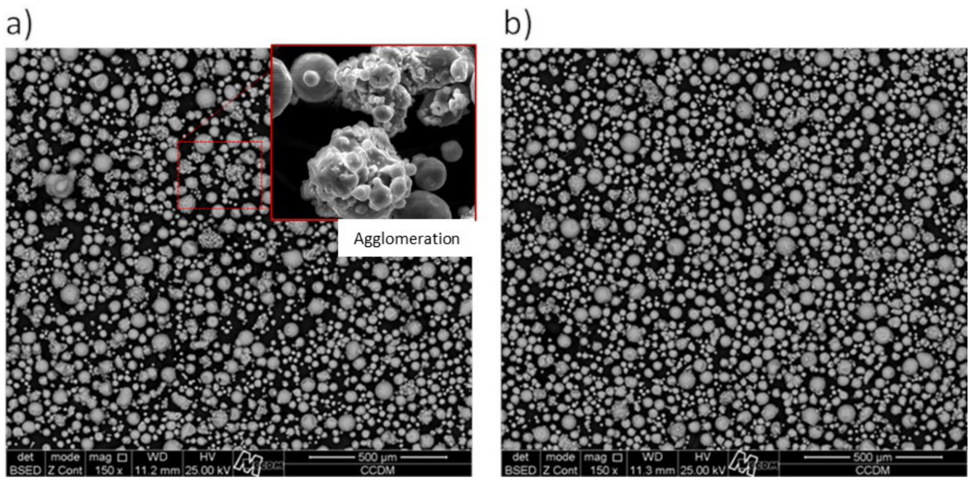

Figure 4. a) Powder distribution under high humidity and high temperature conditions. b) Powder distribution under high temperature and low humidity condition.

a rise in moisture levels under high temperature and high humidity, and conversely, a decrease when exposed to high temperature and low humidity.

Upon reviewing these outcomes, we note a congruence with the research conducted by Cordova et al.[13]. Their study demonstrated that the powder becomes increasingly sensitive to humidity with an elevated temperature, aligning with our findings. Specifically, their experiments at 80% humidity and 50°C for 72 hours elucidated similar trends. Upon examining these results, a convergence can be observed with the findings presented by Touzé et al.[8], wherein the temperature ratio renders the powder more susceptible to humidity.

The SEM images unveiled a notable disparity between the powder exposed to high temperature and low humidity versus high temperature and high humidity (Figure 4). The increased agglomeration was notably evident in the latter, revealing a substantial accumulation of satellite particles. These satellites were formed due to weak bonding caused by the presence of water among the particles. As previously noted, these satellite particles exhibit high surface energy, which likely contributes to the bonding process resulting in agglomeration.

The gas atomized material has excellent sphericity (Figure 5); However, due to the significant presence of satellite particles, it has a considerably high amount of free energy. This energy promotes adhesion between satellite particles and primary particles, resulting in the formation of small clusters of particles, even in dry environments, following the same characteristics shown by Cordova et al[13].

As determined in prior pycnometry assessments, the powder tested initially dis-played a notably high level of densification. In Figure 6, a distinct void within the sample is evident, a rare occurrence but of potential concern due to its possible implications for the material's structural integrity post-solidification during the LPBF process. The voids could couse material damage[13], a concern that can be quantified using finite elements and damage criteria , for this it is importantat know the voids and ditribuition inside the part[20], after the fabrication.

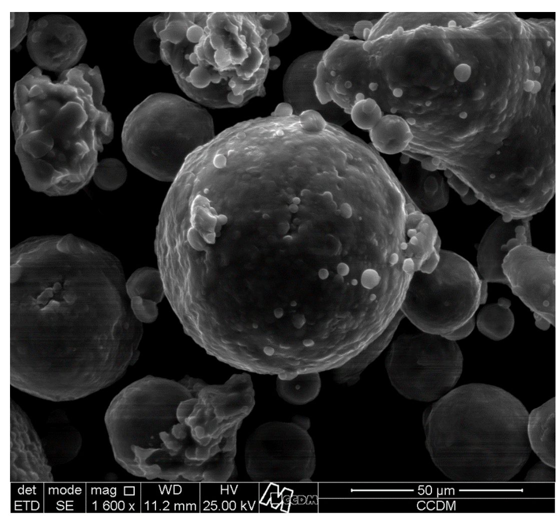

Figure 5. Morphology of the powder with high temperature and low humidity.

Numerous studies have investigated the application of Raman spectroscopy for identifying functional groups in aluminum alloys. When analyzing samples obtained under low humidity conditions, a significant distinction arises in the two spectra. Notably, the more prominent peaks at 474 and 515 cm-1 in Figure 7, section A, are attributed to Al-O bonding groups, which are characteristic of aluminum oxide. This clear observation points to an increased formation of aluminum oxide at elevated temperatures, as anticipated due to enhanced atomic mobility.

Observing the formation of aluminum hydroxide is crucial in our study due to its inherent characteristics. While aluminum hydroxide is less stable than aluminum oxide, it plays a pivotal role in surface protection against oxidation. Despite being a higher-energy form, the formation of aluminum hydroxide occurs on the surface,

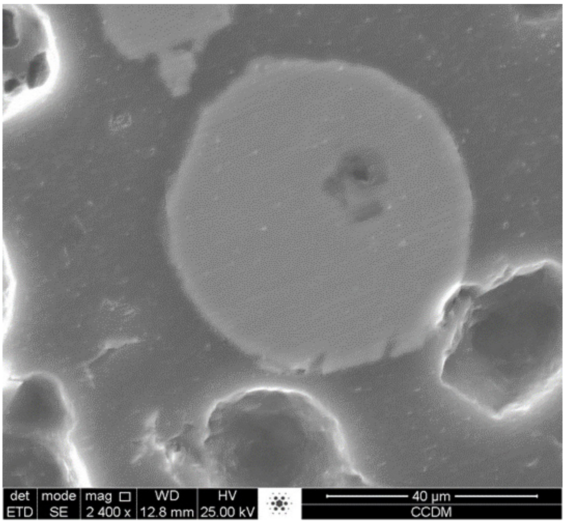

Figure 6. Internal porosity in a particle before the application of heating and humidity.

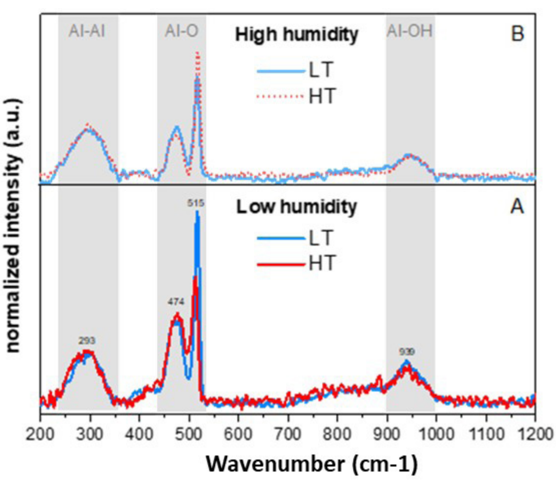

Figure 7. Raman spectra of samples analyzed at low humidity (A) and high humidity (B).

serving as a protective barrier that inhibits the oxidation process of aluminum. This nuanced behavior underscores the significance of monitoring and understanding the formation of aluminum hydroxide in our investigation, as it contributes to the overall comprehension of the protective mechanisms inherent in aluminum surfaces[21].

Conversely, under high-humidity conditions, the spectra exhibit two remarkably similar patterns. The variation in the Al-O peak is remarkably subtle. This finding suggests that under high-humidity conditions, the results are virtually indistinguishable, regardless of whether the temperature is low or high. This aligns with expectations since there is an ample supply of oxygen and water vapor, leading to extensive material oxidation in a non-equilibrium environment with a high partial pressure of oxygen. In this scenario, temperature exerts minimal influence, and the oxidation process proceeds in a similar manner.

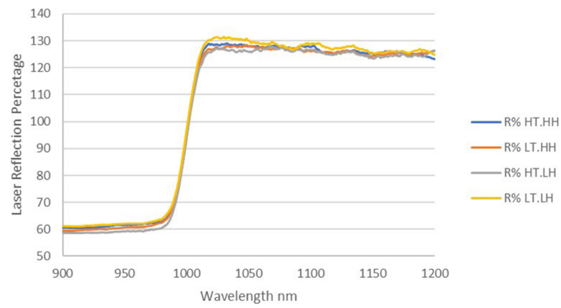

Figure 8. Percentage of laser reflection when compared to magnesium white calibration. Samples with different treatments being high temperature (HT), low temperature (LT), high humidity (HH) and low humidity (LH).

In conditions of high humidity, the formation of aluminum oxides is favored. Moisture can react with aluminum, promoting oxidation reactions and forming an oxide layer on the surface. The intensity of the Al-O binding bands, characteristic of aluminum oxide, may be greater in these cases. However, it is important to consider that temperature also plays a key role in this process.

The reflectance test conducted on the samples revealed the presence of oxides and hydroxides observed through Raman spectroscopy, but their impact on the material's reflectivity was found to be insignificant. Unlike the findings observed by Riener et al.[22], where they reported a decrease in the connection between humidity and laser ab-sorption due to hydroxides forming on the surface, our study did not reproduce the same effect. Their article suggested a potential link between humidity, laser absorption, and the reduction of laser dust rather than the alteration of reflectivity (refer to Figure 8). These results significantly contribute to comprehending the Laser Powder Bed Fusion (LPBF) process.

In the study of aging AlSi10Mg powder in the air by Silva et al.[23], the increased oxygen content led to decreased laser beam absorbance due to a thicker oxide layer on the particles. During DED processing, the increased oxygen content decreased surface tension, resulting in tracks with decreased height and increased width. Aging the powder also induced more porosity in the tracks, likely caused by hydrogen pores. The increased porosity led to lower tensile strength and a potentially coarser microstructure, while the increased elongation was attributed to the presumably lower cooling rate.

Given the observed consistent absorption leads one to consider whether pore formation can be avoided by simply pre-drying the powder before using for laser based additive manufacturing. This observation suggests the possibility that moisture, rather than surface phase formation, may be the predominant factor contributing to pore formation.

4. Conclusions

Our findings highlighted the substantial impact of different temperature and humidity conditions on the properties of aluminum powder, particularly moisture content, affecting fluidity, bulk density, and agglomerate formation. Raman spectroscopy has provided valuable information on oxide formation, revealing potential implications for laser absorption but not demonstrated when measured by a spectrophotometer.

Notably, the significant role of temperature in stabilizing aluminum oxide under high humidity conditions is uncovered by influencing powder oxidation reactions. The correlation between humidity exposure and agglomerate formation emphasizes the necessity of humidity control in powder handling. Managing agglomerated particles is crucial, as they can retain moisture, leading to increased porosity during LPBF. Unexpectedly, consistent laser reflection is revealed despite the presence of oxides and hydroxides, challenging established beliefs about their relationship with laser absorption.

This discovery hints at promising strategies like pre-drying the powder to prevent pore formation and potentially mitigate the adverse effects of moisture, that can cause permanent damage to the aluminum powder.

In summary, this research provides a comprehensive guide for comprehending and analyzing powder properties, aiming to optimize the LPBF manufacturing process. By highlighting the intricate interplay of environmental conditions and their impact on aluminum powder behavior, we aspire to propel the continuous advancement and enhancement of additive manufacturing techniques, particularly concerning metallic materials like AlSi10Mg. Future research and experimentation are warranted to fully unlock the potential and elevate the quality of components produced through LPBF utilizing aluminum powder. The current study may further contribute to defining how humidity parameters and storage conditions influence the powder and potentially impact the printing process, depending on the machine's design. Additionally, it has created opportunities for potential studies on solid-state flow simulation[24].

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