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Water Atomized H13 Tool Steel Powders for Binder Jet Additive Manufacturing

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CONTENTS

Table of Contents

ABSTRACT

ORNL (Contractor) and Ametek, Inc. (Participant) collaborated to determine the feasibility of adopting water atomized H13 tool steel powder for binder jet additive manufacturing (BJAM) for tooling applications. The capability to utilize water atomized powders can substantially reduce powder expenses for BJAM, enabling process competitiveness with casting whilst enabling new applications of high-temperature, net-shaped components.

1. FABRICABILITY OF WATER-ATOMIZED H13 TOOL STEEL POWDER IN BJ-AM

This Manufacturing Demonstration Facility (MDF) technical collaboration project was started on November 5, 2018 and was completed on December 31, 2023. The collaboration partner for this project was Ametek, Inc. In this project, ORNL and Ametek evaluated the applicability of water atomized H13 tool steel powder for binder jet additive manufacturing (BJAM) for tooling and other industrial applications. The focus of this work was to demonstrate the fabricability of water atomized H13 tool steel powders in BJAM.

1.1 BACKGROUND

H13 tool steel stands out as the most utilized hot working tool steel, finding applications even in cold working. BJAM presents a unique avenue for tool and die manufacturing, facilitating scalability in designs, cutting down lead times, enabling swift prototyping for quick design modifications, and integrating features like conformal cooling channels. Previously, experiments at the MDF have successfully showcased the viability of utilizing gas atomized H13 powders in BJAM of injection mold dies and various tooling components [1,2]. However, for BJAM to gain widespread acceptance, the cost of feedstock needs a significant reduction to fully harness its scalability. Water atomization of powder feedstocks is a feasible method to substantially reduce material costs compared to the more commonly used gas atomized powders for BJAM. Nonetheless, the water atomization process produces irregular shaped powders that are challenging to spread and can lower the green density during BJAM. This can cause higher distortions or reduce the extent of densification during sintering. Further, subtle chemistry changes can also impact the sinterability of the powders. Therefore, this collaboration between ORNL and Ametek focused on determining the feasibility of BJAM to deposit the irregular water atomized powders and the ability to densify the printed preforms via pressureless sintering.

1.2 TECHNICAL RESULTS

H13 tool steel powders with three different chemistries within the H13 specifications were produced by water atomization by Ametek. The three powders were characterized in terms of chemistry, density, morphology, powder size distribution, flowability, and apparent density prior to fabrication. These details are summarized in Figure 1 and Table 1. As expected, the powders did not flow through the Hall flow meter owing to a combination of their finer size and irregular shape.

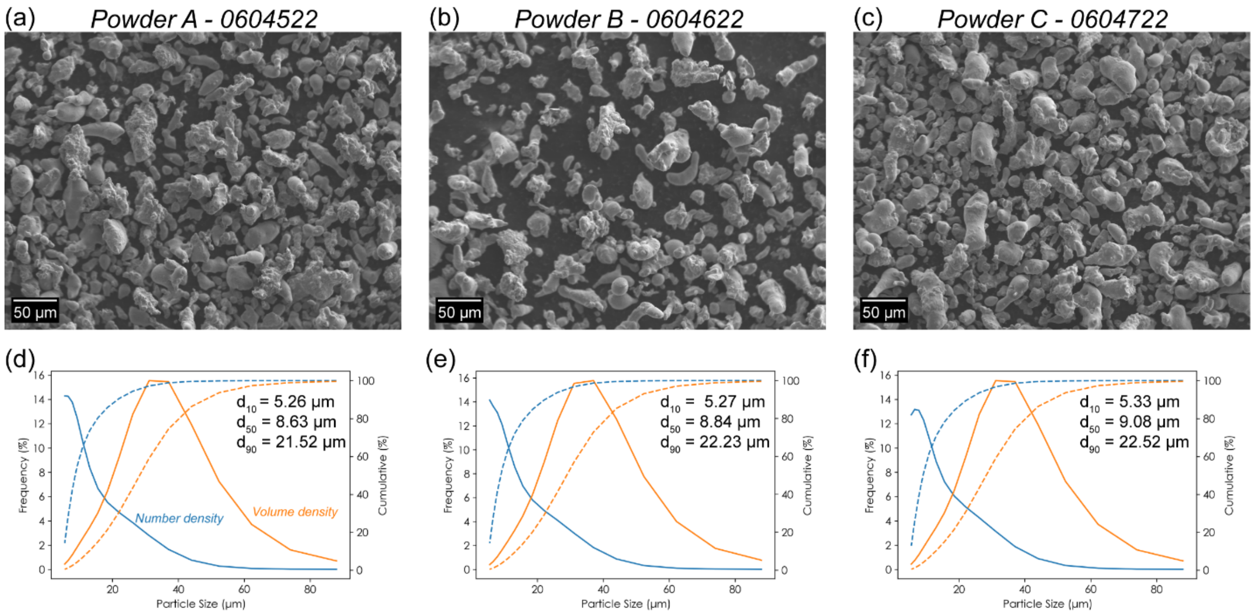

Figure 1. (a – c) Scanning electron microscopy (SEM) micrographs showing the morphology of the three different water atomized H13 tool steel powders. (d – f) Respective powder size distributions (PSDs) of each powder as measured by laser diffraction.

Table 1. Powder compositions as measured by inductively coupled plasma mass spectrometry (ICP-MS) showing distinct variations in Mo and Si concentrations. Powders also displayed variations in true (pycnometer) & apparent densities, nor were they flowable.

Powder	A	B	C
Composition (wt%)			
Fe	Bal.	Bal.	Bal.
C	0.274	0.294	0.345
Cr	5.16	5.39	5.29
Mo	1.50	1.54	2.14
Si	0.61	0.94	0.94
Mn	0.32	0.32	0.22
Ni	0.064	0.063	0.063
S	0.012	0.013	0.016
O	0.488	0.441	0.441
True Density (g/cm^3)	7.85	7.80	7.78
Apparent Density (g/cm^3)	3.12	3.11	3.11
Flowability	No Flow	No Flow	No Flow

An ExOne M-Flex BJAM system with ExOne Fluidfuse binder was used to successfully deposit 0.25” cubes, referred to as green parts, using the three different powders. Two binder saturation levels of 85 and 105 % were utilized for each powder, to assess the effect of binder saturation on final part density and properties. The green parts were debinded in a furnace in a flowing Ar atmosphere at 900 °C/3 h, followed by supersolidus liquid phase sintering [3] in a vacuum furnace at 1380 °C/5 h. These debinding and sintering parameters were based on previous investigations into BJAM of gas atomized H13 tool steel powders [1]. Figure 2 shows the morphological change between green parts and final sintered parts. Layer delamination was apparent in the green and post-sintered parts for all powders at 105 % binder saturation. This was possibly because of either nozzle malfunction or excess dry time. The study did not focus on optimizing the other parameters for a given saturation level. Post-sintered builds from powder C resulted in slumping at both saturation levels, indicating higher liquid volume fraction at the sintering temperature for powder C.

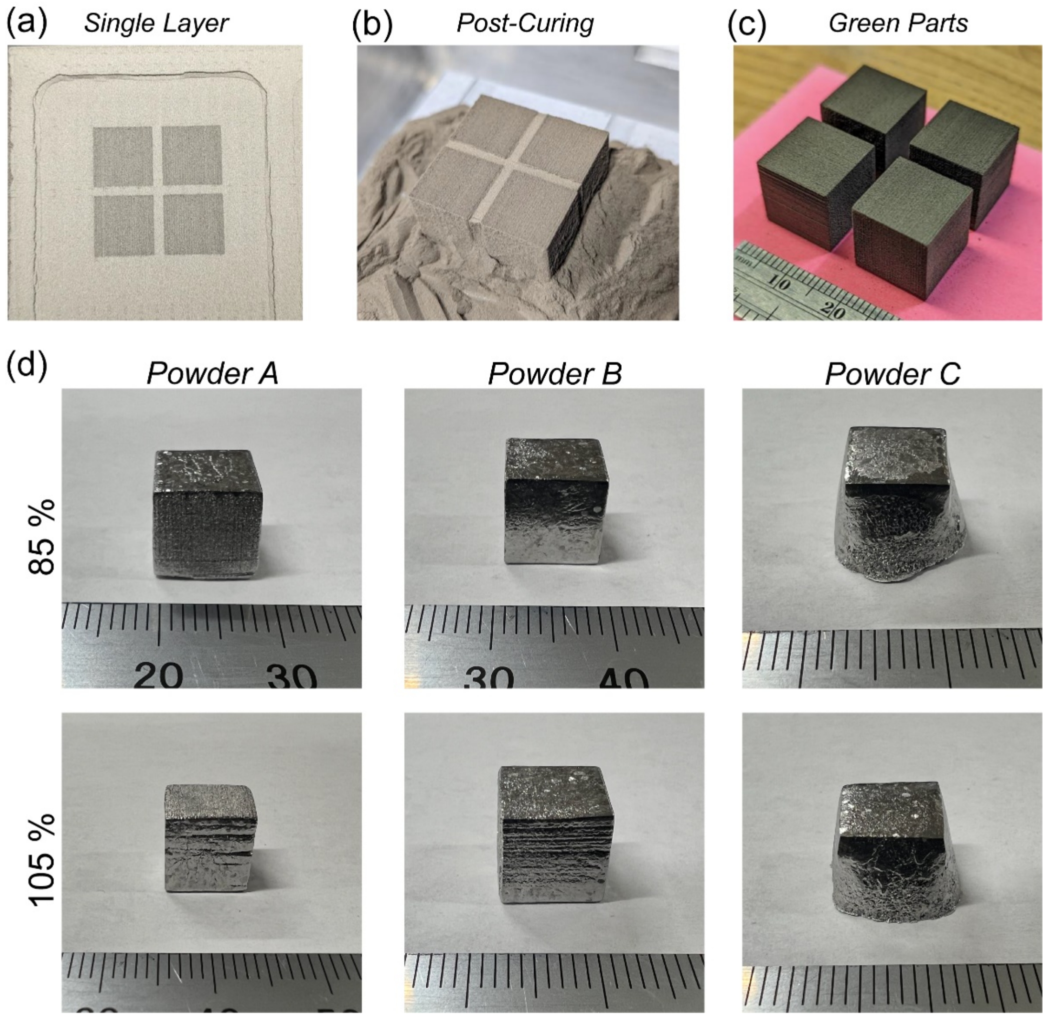

Figure 2. Optical images showing (a – c) sequence of BJ-AM fabrication steps and (d) builds post-debinding & sintering. Differences in delamination between saturation levels and slumping between powder types were evident. Ruler markings are in mm.

Differences in slumping behavior and porosity (Figure 3) indicated that the sintering temperatures used for each powder were sub-optimal, due to differences in powder chemistry. There were no differences in porosity between saturation levels. Builds from powder A, with the lowest Si concentration, resulted in the highest porosity whereas builds from powder C, with the highest Si concentration, resulted in the lowest porosity. Higher Si concentrations likely reduced the supersolidus liquid phase sintering window of the H13 powders [4]; resulting in excessive liquid phase formation at 1380 °C and hence, slumping, in builds produced from powder C.

Table 2 summarizes the change in volume from green part, relative true density in comparison to powder and Vickers microhardness of the post-sintered builds. Similar volume reductions from green parts were apparent in all builds. The relative densities agreed with porosity observations with builds from powder C having the highest density, due to the greater amounts of liquid phase sintering experienced. Among the three powders, powder B resulted in reasonable density of ~98% with minimal slumping for the sintering conditions used.However, despite no relative density variations apparent between saturation levels of builds from the same powder, differences in microhardness were apparent. This is possibly because higher binder saturation results in higher amounts of retained carbon post binder burnout that increases the hardness as has been discussed by Nandwana et al. in their work on gas atomized powders [1]. Microhardness was higher in builds with 105 % binder saturation and higher hardness correlating with higher relative densities.

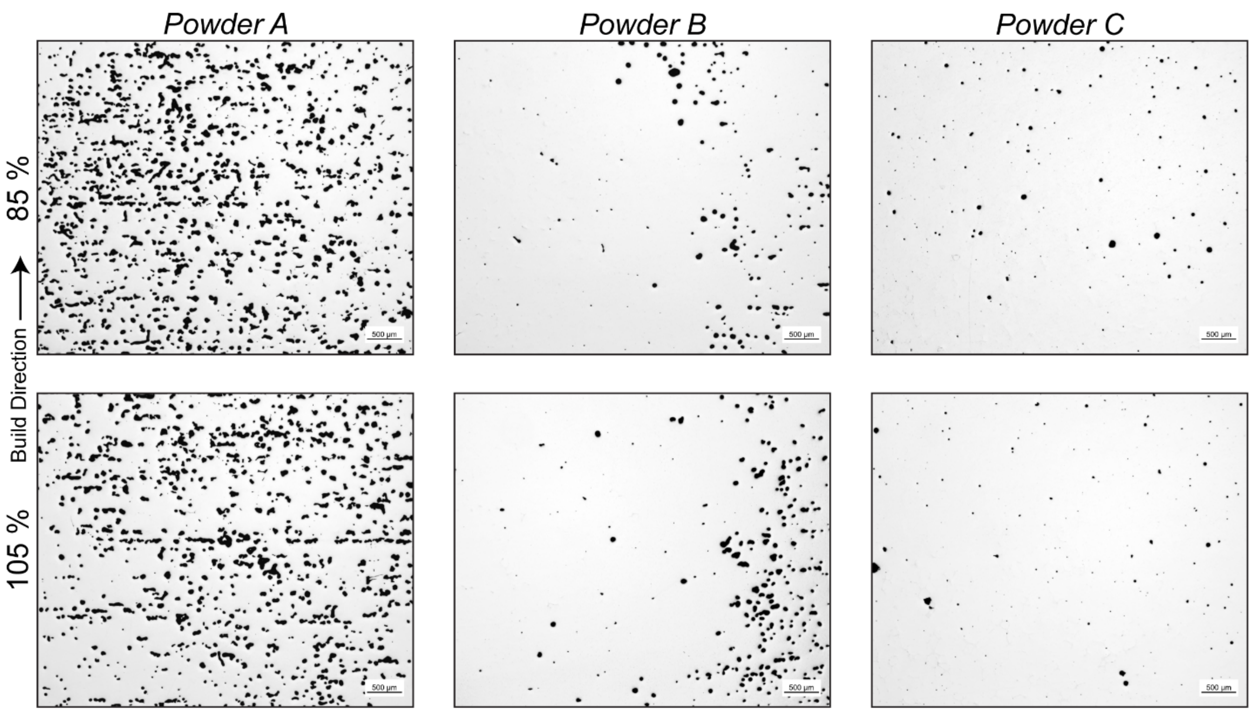

Figure 3. Optical microscopy (OM) micrographs of polished cross-sections of the sintered builds. Significant differences in porosity between powders but not between saturation levels. Builds from powder B exhibited 'banding' of porosity near the edges of the build.

Table 2. Summary of relative volume change from green part, relative true (pycnometer) density in comparison to powder, and average Vickers microhardness for both saturation levels of post-sintered builds.

Powder	A - 0604522	B - 0604622	C - 0604722
Volume Reduction (%)			
85 % Sat.	64.4	64.7	65.2
105 % Sat.	63.8	65.3	65.3
Relative Density (%)			
85 % Sat.	92.8	98.3	99.2

105 % Sat.	92.8	98.2	99.3
Hardness (HV0.05)			
85 % Sat.	191.4 ± 2.3	207.8 ± 1.4	247.7 ± 10.3
105 % Sat.	195.5 ± 3.8	222.7 ± 1.4	268.5 ± 16.4

In future, thermodynamic simulations to predict optimal sintering temperatures for all three powders will be performed based in their individual chemistries. This will minimize slumping, improve sintered density and improve mechanical properties. These predictions will then be experimentally validated and used to fine tune the sintering parameters for BJ-AM of water-atomized H13 tool steel powders.

1.3 CONCLUSIONS

In summary, this Phase 1 CRADA between Ametek, Inc. and ORNL demonstrated the feasibility of producing BJAM H13 tool steel using water atomized powders. H13 powder chemistry plays a key factor in sintering temperature windows, final part density, and properties, whilst the non-sphericity and non-flowability of water-atomized powders do not affect the fabricability of builds. The size distribution of the powders and sintering schedules can be further optimized to improve the densification during sintering.

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