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Electron Microscopy Analysis of 17-4 PH Powder for Additive Manufacturing

Yu Sun[1], Mark Aindow[1], Rainer J. Hebert[1,2]

[1.] Department of Materials Science and Engineering, and Institute of Materials Science, University of Connecticut, Storrs, CT, USA
[2.] Additive Manufacturing Innovation Center, University of Connecticut, Storrs, CT, USA

Metal additive manufacturing (MAM) has evolved into a production-ready manufacturing technology over a period of just a few years. The strong interest in MAM mainly derives from the ability to manufacture complex parts without the tooling and material waste typically associated with machining components from bulk alloys. Most of the attention for MAM has focused on Ti-6Al-4V and IN718 alloys, but several other alloy classes, such as stainless steels have gained traction as well. The stainless steels that have been considered for MAM include: 304, 316, and 17-4PH alloys, but to date none of these has been studied in great detail. 17-4PH is a heat-treatable martensitic stainless steel. Since MAM starts with the spreading or feeding of powders, a careful characterization of the starting powder is necessary to understand the way in which the microstructure and defects develop in the additively manufactured components, and how these lead to variations in component properties.

17-4PH powders were received from 3DSystems/Phenix for MAM on a 3DSystems ProX-300 machine. The powder was analyzed by XRD, SEM and TEM, using a Bruker AXS D2 Phaser diffractometer, an FEI ASPEX Explorer and Teneo SEMs, and an FEI Talos TEM/STEM, respectively. TEM samples were prepared by the FIB lift-out method on a FEI Helios G3 FIB. Particle size distributions were determined using the Aspex Explorer SEM in automated feature analysis mode.

The XRD analysis of the as-received powder reveals the presence of bcc alpha phase and fcc gamma phase (Fig. 1a). These two phases were also detected by Zhao and co-workers in nitrogen gas-atomized 17-4PH powder [1]. The as-received powder has an average particle size of 2.22 μm in diameter, and the particle size distribution is shown in Fig. 1b. Aside from particles at a size of 2-10 μm, a significant portion of the particles has a diameter of $<1\mu m$. The overall morphology of the powder is a mixture of irregularly shaped and spherical particles as shown in Fig. 2a. Unlike the results documented in [1], the particles in the current work do not reveal dendrites at their surfaces. Many of the larger particles appear to include smaller particles (e.g. Fig. 2b) that were presumably incorporated during the atomization and solidification stages. Fig. 3 is a HAADF-STEM micrograph and the corresponding EDX maps for the major elements in the 17-4PH powder. Si and Mn oxide nano particles are identified in the matrix, and these impurities might agglomerate during the MAM process, leading to deterioration of the component properties. We note that most of the as-received 17-4PH powder particles are considerably smaller than the commercial powders typically used for MAM. The small particle size and the roll coating of the ProX-300 machine yield a dense powder bed, which likely improves the surface roughness of MAM parts and possibly reduces the defect sizes. Further work is necessary to confirm these conclusions and to study the influence of the nano-oxide inclusions on the MAM component properties.

References:
[1] XM Zhao *et al*, Int. J Min. Met. Mater. **19** (2012) p. 83.
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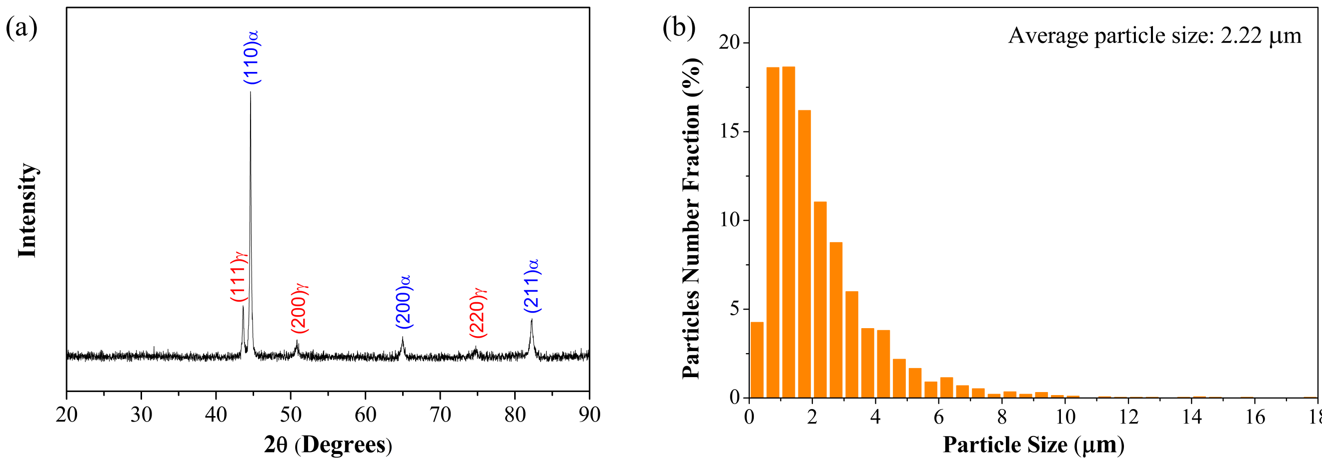

Figure 1. XRD pattern (a) and particle size distribution (b) of the as-received 17-4PH powder.

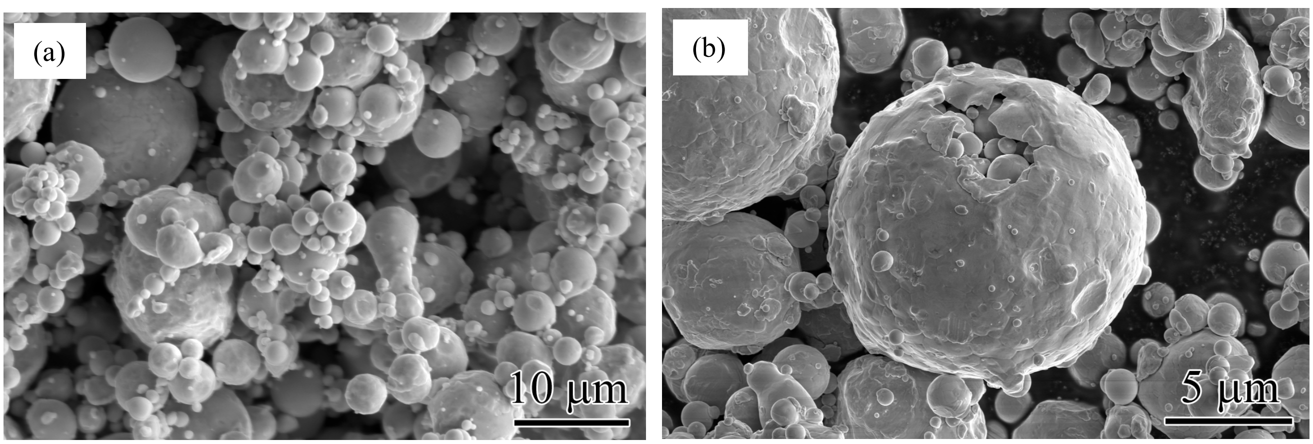

Figure 2. SEM micrographs of the as-received 17-4PH powder.

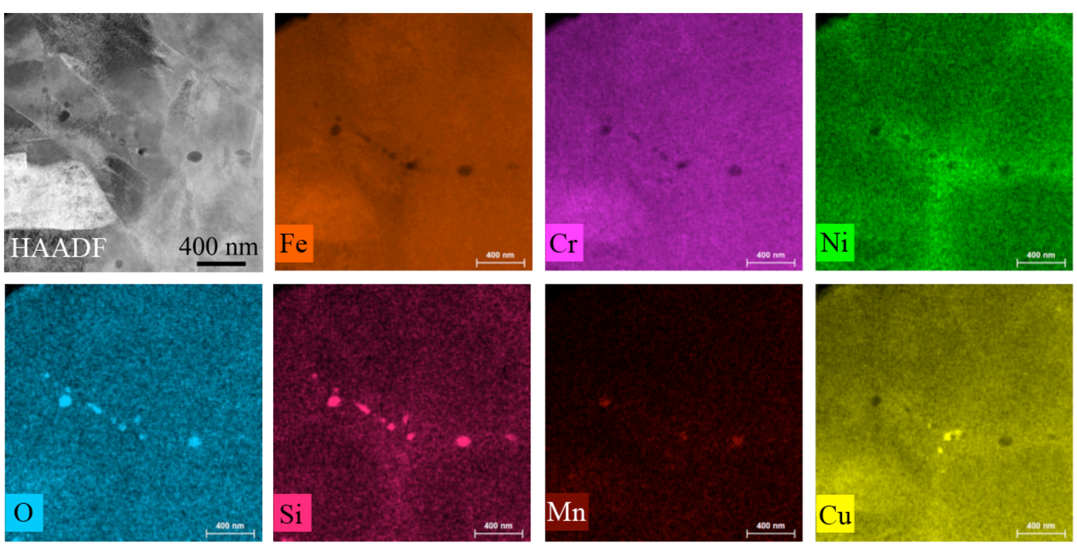

Figure 3. HAADF-STEM micrograph and EDX elemental maps from a 17-4PH powder particle.

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