

# Assessment of metallic FDM samples using x-ray diffraction

K. R. Aroom<sup>1</sup>, P. Y. Zavalij<sup>2</sup>, H. Huang<sup>3</sup>, and L. W. Schultheis<sup>1,4\*</sup>

<sup>1</sup> Robert E. Fischell Institute for Biomedical Devices, University of Maryland, College Park Maryland, USA

<sup>2</sup> Department of Chemistry and Biochemistry, University of Maryland, College Park Maryland, USA

<sup>3</sup> Advanced Analyzer Labs, Inc., USA

<sup>4</sup> Center for Excellence in Regulatory Science and Innovation, University of Maryland, College Park Maryland, USA

\* Corresponding author, email: lschulth@umd.edu

*Abstract: X-Ray Diffraction (XRD) characteristics were observed in additively manufactured metallic samples. Sample ‘coupons’ were printed with fused deposition modeling (FDM) systems. The coupons underwent post-processing steps to purge the polymer binder and sinter remaining metal. XRD analysis was performed to measure the crystallographic nature of printed samples at several stages of fabrication. A determination of grain/crystallite size can also be performed on sintered parts using XRD.*

## I. Introduction

Additive manufacturing enables the production of customizable, high-accuracy parts made from a wide variety of materials. For mission-critical applications such as medical implants, quality assurance of manufactured parts is of utmost importance. Validation of printed parts throughout the fabrication process would be a significant step in developing the next era of manufacturing.

### I.I. AM of metallic materials

Options for producing metallic parts through additive manufacturing include Direct Metal Laser Sintering (DMLS), Selective Laser Melting (SLM), Selective Laser Sintering (SLS) using a polymer/metal powder blend, or investment cast molding. Recently, extruded metal/polymer blends have become available for use with Fused Deposition Modeling (FDM) systems. DMLS produces fully densified parts directly from the machine, with limited post-processing requirements. As opposed to indirect SLS which often requires a binder burnout step and then an infiltration step, some FDM materials can be sintered to a final shape without the addition of an infiltrating metal. The FDM material sintering process requires the use of a vacuum furnace and refractory support material to maintain the shape of the part during sintering.

### I.II. X-Ray Diffraction (XRD)

X-Ray Diffraction is a measurement technique utilized across diverse fields as an authoritative method to determine crystalline structure, phase composition, grain/crystallite size, and many more. A standard polycrystalline XRD setup includes an X-ray source, a sample stage, and an X-ray detector, shown schematically in Fig. 1. The source and detector are mounted on a goniometer stage that enables both components to rotate about the goniometer axis, thus changing  $2\theta$ . As the goniometer scans through a range of angles, a profile of

measured X-ray intensity is formed. The analyzer used in this study (Fig. 2) utilized a 2D detector, from which the measured intensities were integrated to create a 1D profile. The generated profile is analyzed and compared to a database of scanned materials (known as the powder diffraction file (PDF)). Profile or full pattern fitting of different phases found in the PDF database is used for accurate quantitative phase analysis, refinement of precise lattice parameter (unit cell dimensions) and other metrics.

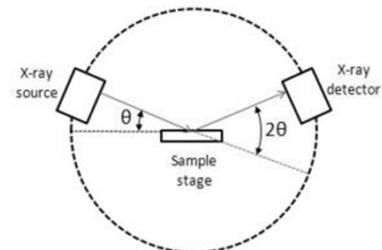


Figure 1: XRD schematic[1]

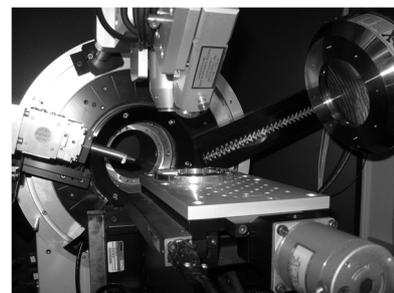


Figure 2: Bruker D8 Discover XRD parallel beam system.

## II. Material and methods

Test specimens (coupons) were fabricated from several stock materials. Table 1 describes the materials

Table 1: Materials analyzed by XRD.

Material Name (manufacturer)	Percent Metal (%)
Copper Filamet (Virtual Foundry; Wisconsin, USA)	~90
316L Stainless Steel Filamet (Virtual Foundry)	~80
Stainless Steel (Desktop Metal; Massachusetts, USA)	~80

Printing of the square coupons (size 15mm x 15mm x 4mm LxWxH) were performed by the manufacturers. Desktop Metal has a brand-specific printer, while Virtual Foundry can be used with various consumer-grade FDM systems. Sintering of green parts was also performed by the manufacturers and presented to authors for analysis. The coupons were analyzed using a D8 Discover X-Ray Diffractometer equipped with a  $\text{CuK}\alpha$  radiation  $\lambda=1.5418$  Å from sealed X-ray tube, Göbel mirror, XYZ stage with video camera and laser for sample positioning, and state-of-the-art Vantec-500 area detector (Bruker Nano Inc., Madison WI, USA). Analysis occurred before printing (i.e. stock filament), after printing, and after sintering. Some samples were intentionally over-sintered to explore differences in a defective part.

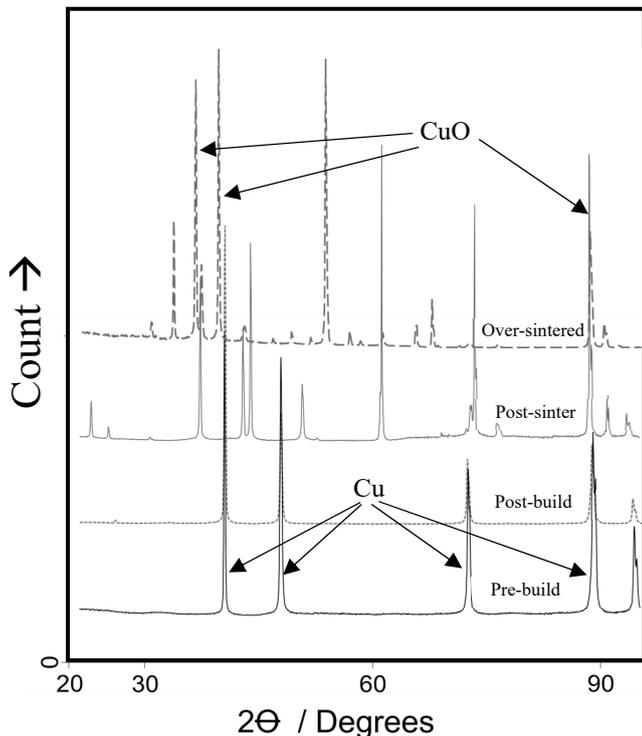


Figure 3: XRD profiles of copper coupon at different points of fabrication and post-processing. Significant peaks in Pre-build and Post-build measurements match copper signature, while Post-sintering and Over-sintered show oxide signatures. Note: the plots have been spaced vertically for better separation.

### III. Results and discussion

Build quality of FDM-derived parts requires constant

vigilance and monitoring of process conditions. Early-stage XRD sampling of a green part gives insight to the phase composition of the build material, and may also act as a baseline for subsequent measurements. XRD becomes more valuable in comparing a part after sintering. Imperfections in terms of impurities, or oxides formed are readily apparent on the XRD profile. Fig 3 shows a series of XRD profiles from a copper coupon sample at different points of fabrication and post-processing. Grain size distribution along with stress/strain can also be evaluated in a non-destructive manner by reviewing the X-ray detector scan, shown in Fig. 4. Six different scans are shown in Fig. 4, the left column are from points in a normal sintered region of a stainless steel coupon, and the right column are from points in a discolored region of the coupon, possibly due to oxidation. Each scan is a composite of images collected as the source and detector change the angle of incidence with the sample. The resulting image contains 'diffraction rings' that appear as nearly vertical lines. Small grains create a uniform distribution of ring intensity, due to the grains being randomly oriented. However, as grain size increases, the larger crystals result in more heterogeneous intensity rings.

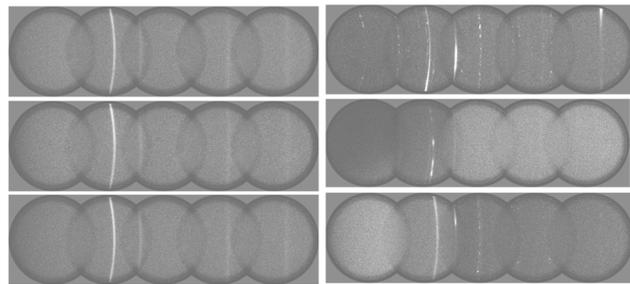


Figure 4: XRD intensity scans of stainless steel coupon focused on defect-free points (left column), versus discolored points (right column)

### IV. Conclusions

XRD shows promise as a non-destructive tool to determine material composition in additive manufacturing. Clear changes to the diffraction profile occur throughout the fabrication process. Further investigation may yield an effective set of measurement conditions that can efficiently identify issues or imperfections in a part. XRD analysis after intentionally adding impurities, changing build parameters, or loosening post-processing tolerances would allow a better understanding of how imperfections modify a part's profile. Integrating XRD as part of the fabrication process could allow for real-time process parameter adjustment to maximize the probability of a successful build.

#### AUTHOR'S STATEMENT

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#### REFERENCES

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