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Additive Manufacturing of 94% Alumina and Tracking its Chemical Signature During the Thermal Post-Process

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Abstract

Alumina ceramic materials possess material properties such as thermal stability, oxidation resistance, and high hardness that make them desirable for energy applications [1]. The traditional manufacturing of alumina has been thoroughly investigated and done by using techniques such as dry pressing and slip casting [2]. Within the past 35 years, research of ceramics additive manufacturing has flourished following the invention of Binder Jetting in 1989 [3]. Vat photopolymerization (VPP) is an additive manufacturing technique that uses a light source to solidify a photosensitive ceramic slurry layer-by-layer until the 3D model is completed. While additive manufacturing of ceramics can enhance the freedom of design, cut fabrication time, and waste reduction, there are defects that may arise during the thermal post-process. The thermal post-process is designed to effectively disintegrate the photopolymer binder to have a fully dense ceramic part.

In this work, VPP technology was utilized to fabricate ceramics components layer-by-layer, using a layer thickness of 25µm. More specifically, the Lithoz's Cerfab 8500 printer was used to print alumina components using lithography ceramic-based manufacturing, a subset of VPP. The chemical composition was tracked by using an array of chemical characterization techniques such as Fourier transform infrared spectroscopy, thermogravimetric analysis, Raman spectroscopy, and X-Ray fluorescence. Cross sections of the 3D printed ceramic components were analyzed through scanning electron microscopy.

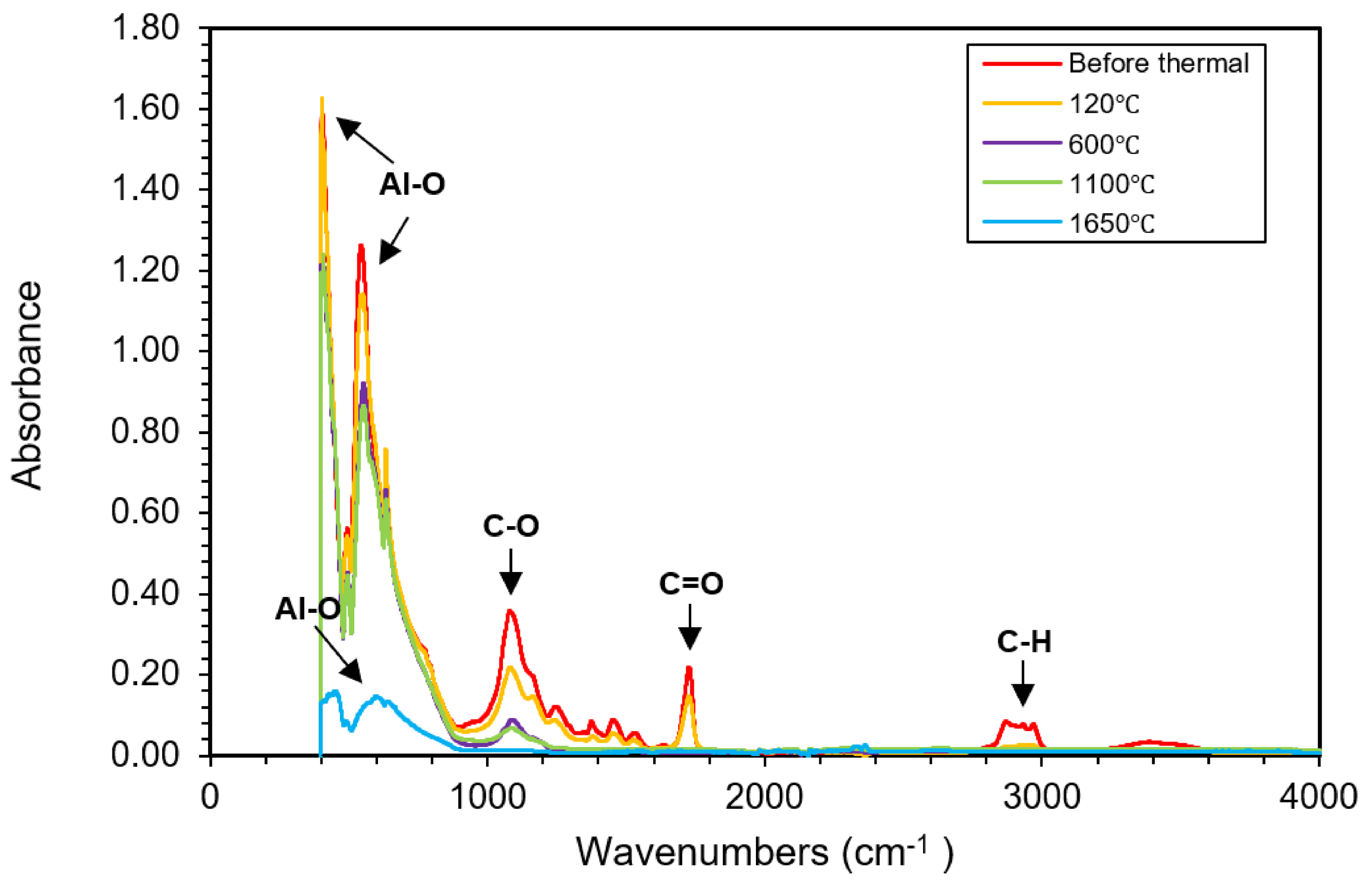
Introduction

Ceramic slurries consist of dispersant, monomer, photo-initiator, and ceramic particles.

- *How can we get a effectively burn out all of the photopolymer binder and be left with a fully-dense ceramic 3D printed part?*

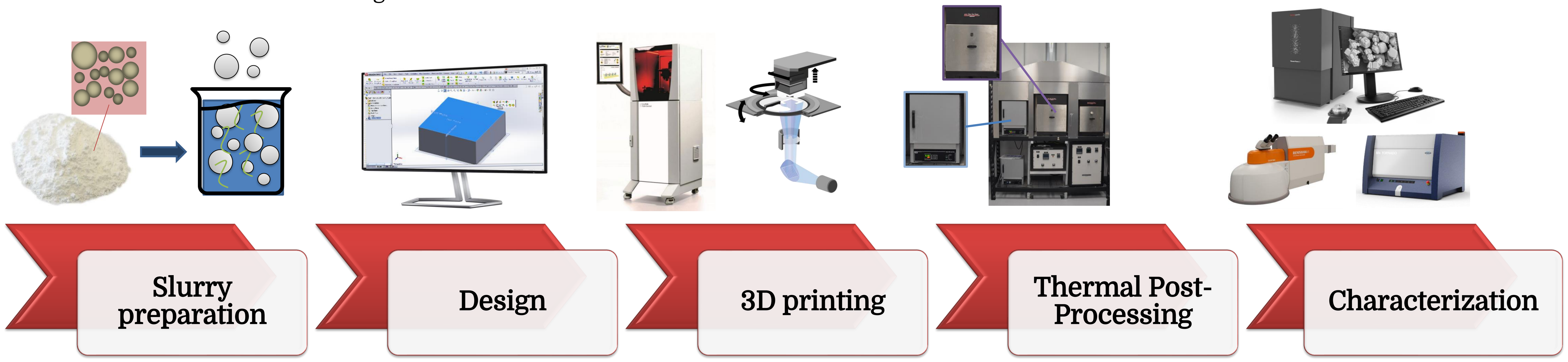
A series of chemical characterization techniques can aid in tracking the chemical signatures of the 3D printed ceramic. In this work, we 3D printed a 94% alumina and used the following techniques to track its composition at each stage of the thermal process:

- FTIR: identify characteristic peaks of the photopolymer binder.
- Raman: compliment FTIR results.
- SEM: analyze cross-sections of 3D printed alumina.
- Cr3+ Fluorescence in Raman: visualize stress distributions.
- XRF: create element maps.



Methodology

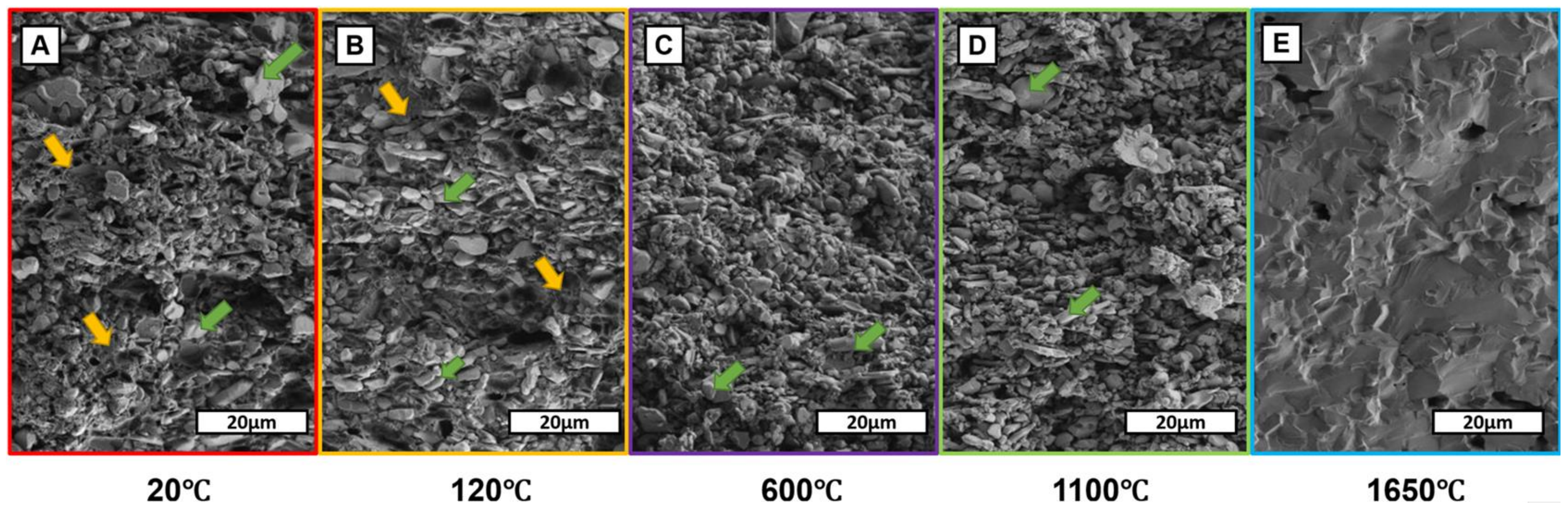
Ceramics Additive Manufacturing Workflow



Results

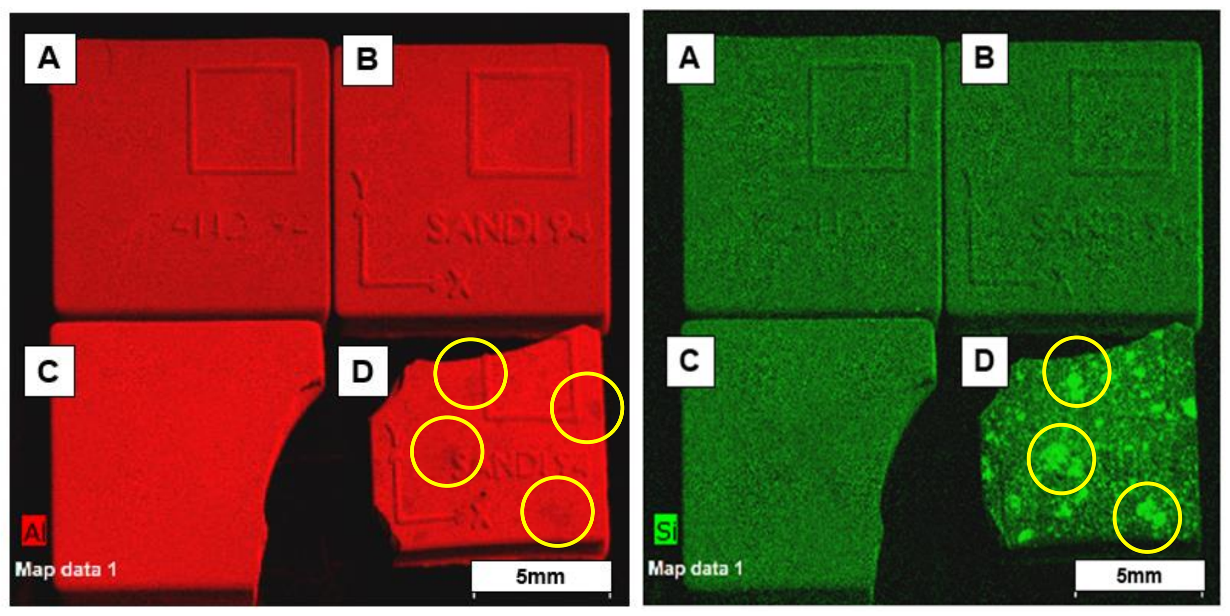
Scanning Electron Microscopy (SEM)

- Photopolymer binder content (yellow arrows) were present after 3D printing (a) and after pre-condition (b). Alumina particles (green arrows) are sandwiched in between photopolymer.
- Debinding at 600 °C (c) and 1100°C (d) completely disintegrated photopolymer binder.
- Sintered alumina (e) has porosity



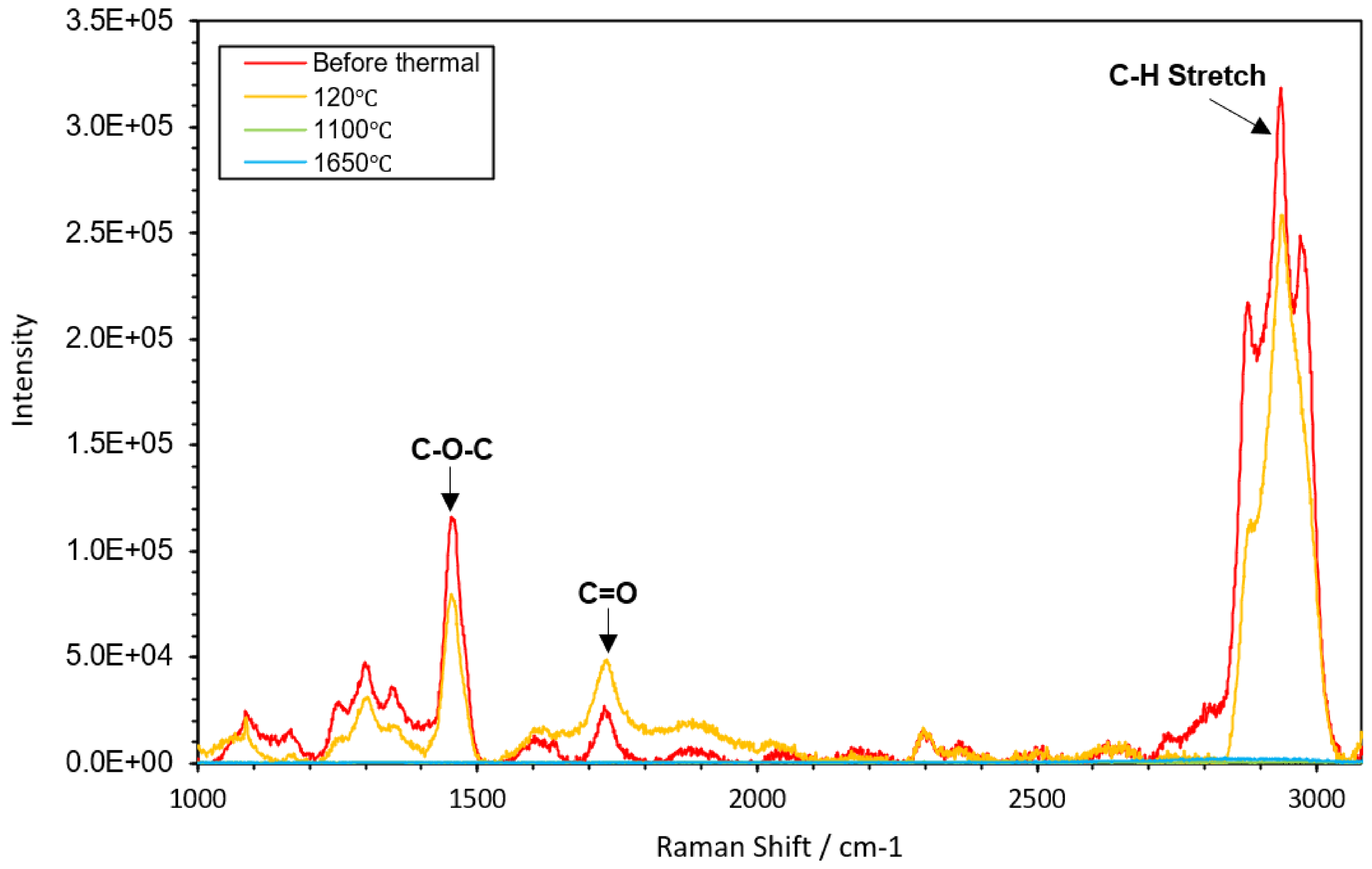
X-Ray Fluorescence (XRF)

- Element mapping of alumina (red) and silica (green) reveal distributions as a function of thermal.
- As the temperature increases, alumina domains intensify (c & d) and silica domains segregate (d).
- As-printed (a) and pre-condition (b) thermal stages showed homogenous element distribution.

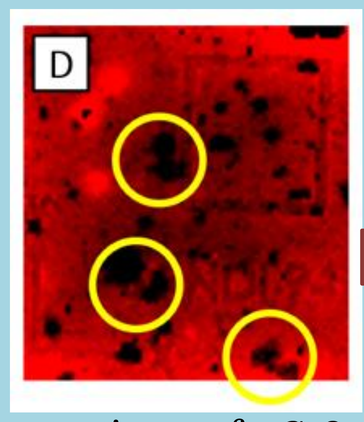


Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy

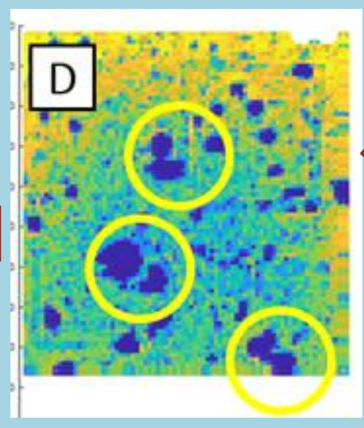
- C-O, C=O, and C-O-C bonds were found to be characteristic of photopolymer constituents.
- Photopolymer signature peaks decreased in intensity with increase of temperature.



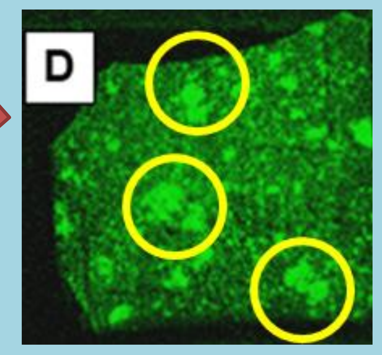
Conclusion



Low concentration of Cr3+ % was identified in compressive stress domains.

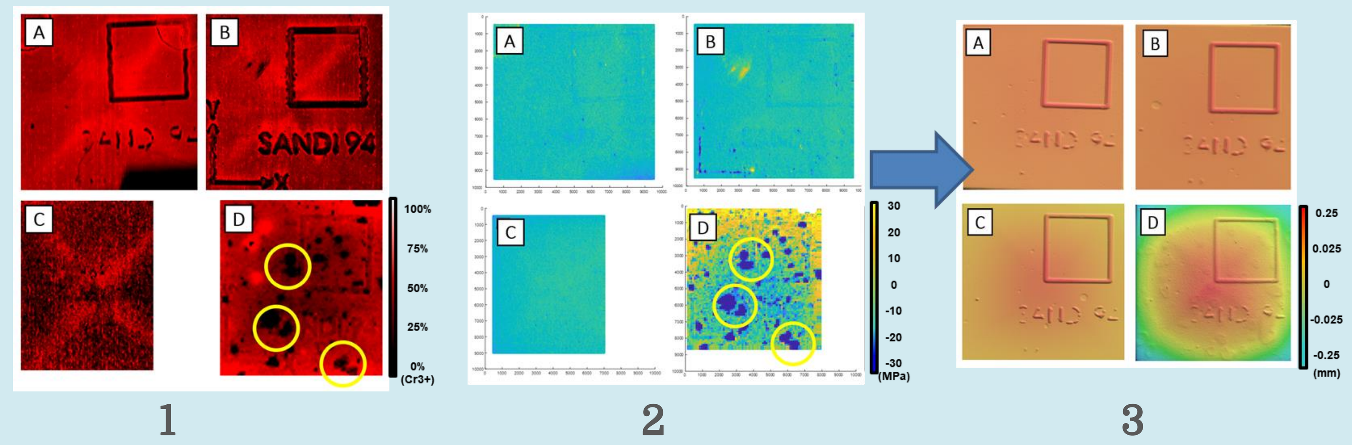


Si aggregates were identified in compressive stress domains.



Future Work

(1) Intensity maps of Cr3+ Peaks, (2) Stress maps of 3D-printed alumina can be used to infer dimensional defects seen in the (3) height maps of 3D-printed alumina. Samples analyzed after (a) green, (b) pre-condition, (c) debind, and (d) sinter stages.



Acknowledgements

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