Radiation responses of additive manufactured stainless steel

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Motivation

Additive-manufactured (AM) alloys have been proposed for nuclear reactor applications, for the advantages in reducing materials manufacturing costs and facilitating urgent on-site replacement. The method is particularly attractive for the flexibility to produce complicated geometries. Additive manufacturing, however, creates various unique micro-structures, which can lead to unique behaviors under neutron and ion irradiation. The impact of these structures on overall performance needs to be systematically evaluated. The project aims to identify the correlations between irradiation responses and characteristic features of AM alloys. Hence we can establish the relationship between irradiation tolerance and processing parameters. At the later stages of the project, we will proceed to ion beam analysis of AM alloys, as a way to characterize alloys' composition and porosity in a non-destructive manner.

Tasks

Task 1: Advanced manufacturing methods screening and technology survey
- Database of advanced manufacturing methods and commercially available technologies
- Database of input/output materials involved in advanced manufacturing

Task 2: Correlation of irradiation responses and microstructures in additive manufactured alloys
- Void depletion as a function of boundary misorientation angles
- Boundary segregation as a function of boundary misorientation angles
- Swelling tolerance for multiple samples manufactured under different conditions

Currently In Progress

Task 3: Nondestructive high-resolution composition analysis of advanced manufacturing technologies
- Advanced manufacturing samples library
- Sample interrogation using advanced nuclear methods (focused ion beam, RBS, PIXE, NRA, ERD)
- Sample mapping and advanced manufacturing signature development

Task 4: Nondestructive high resolution multi-dimensional nuclear signature method for advanced manufacturing technologies
- Nuclear signature-based data analytics and sample attribution in advanced manufacturing characterization
- Nuclear signature optimization
- Nuclear signature-based alarm triggers in advanced manufacturing technology domains
- Formulation of the nuclear signature method for advanced manufacturing technologies

Methods

Two sources of AM stainless steel have been identified for use in the project. Dr. Chen Sun from Idaho National Laboratory was able to provide 316L stainless steel that was made using the direct energy deposition (DED) process. A laser power of 400 W and a scanning speed of 12.7 mm/s were used in the production of the samples. Additional samples were provided by Dr. Dan Thoma from the University of Wisconsin-Madison. Dr. Thoma was able to provide a matrix of samples that vary in laser power, scanning speed, and hatch spacing. Those samples were manufactured using the powder bed fusion (PBF) process.

The Ion Beam Accelerator Laboratory at Texas A&M University is equipped with five ion accelerators of terminal voltages ranging from 10 keV to 3 MV. The lab has several unique capabilities including (1) an ion beam focusing system which is able to focus a beam (i.e. 2 MeV helium ions) from a typical beam spot size of a few millimeters down to a few microns, by using series of magnetic quadrupole lens system; (2) a high resolution RBS detector system which is able to reach an energy resolution of 1 keV, instead of >15 keV in traditional semiconductor solid state detector system. The high resolution is achieved through combination of a strong magnetic field and 2-D detector matrix; and (3) combination of RBS, PIXE, and NRA in one general chamber for a comprehensive analysis. This project will make use of these unique capabilities at later stages.

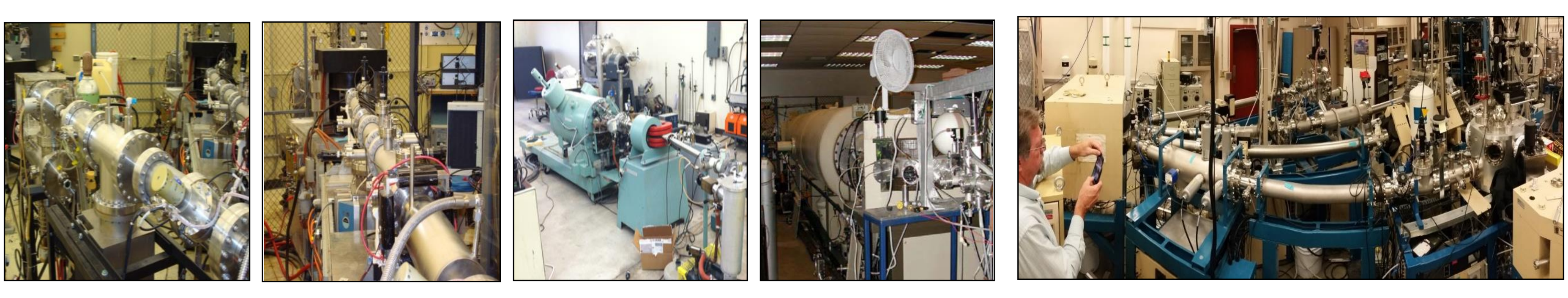

Figure 1. Five ion accelerators of various terminal voltages at Texas A&M University.

The current stage of the project makes use of standard heavy/light ion irradiations in order to better understand radiation responses in additive manufactured alloys. The DED samples received and purchased wrought samples were irradiated using 2 MeV protons at 360°C. The PBF samples, in contrast, have been irradiated under three different conditions. To study void depletion as a function of boundary misorientation angles, the PBF samples were irradiated using 100 keV helium at 300°C. To study boundary segregation as a function of misorientation angles, an irradiation was done with 2 MeV protons at 400°C. To study swelling tolerance at high displacements per atom, an irradiation was done with 5 MeV iron at 575°C.

The microstructures of the samples were characterized using scanning-electron microscopy (SEM) with an electron backscatter diffraction (EBSD) detector transmission electron microscopy (TEM). A focused ion beam (FIB) was used to prepare lamellaes for TEM. The DED samples have finished characterization while the characterization of the PBF samples is currently still in process.

Results

Before irradiation, the microstructure of the DED samples was investigated. The grain structure along the vertical direction was found to not fully follow the melt pool boundary of the alloy, but the grain structure along the horizontal direction was seen to be influenced by the laser path. Small amounts of ferrite were also found in the microstructure of the sample. This was somewhat expected due to the difficulty of uniform cooling during the manufacturing process. Pores observed in the DED samples were found to have a core-shell structure. It was not determined if the formation of the structure was from the FIB redeposition or if it was from the manufacturing process.

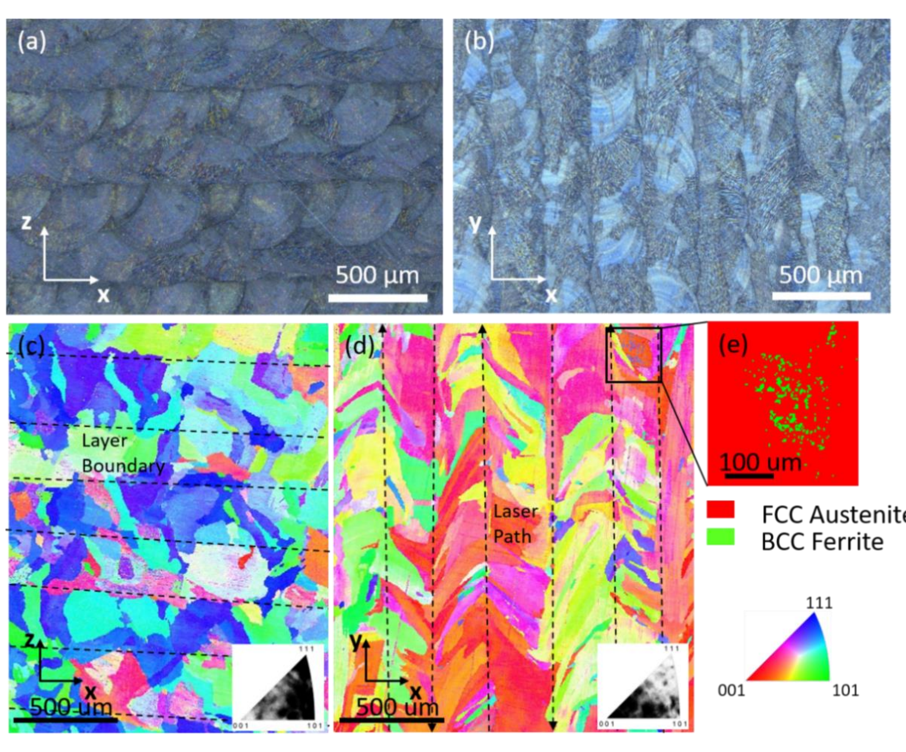

Figure 2. Optical images on (a) vertical and (b) horizontal surface orientation, and EBSD images on (c) vertical and (d) horizontal surface orientations.

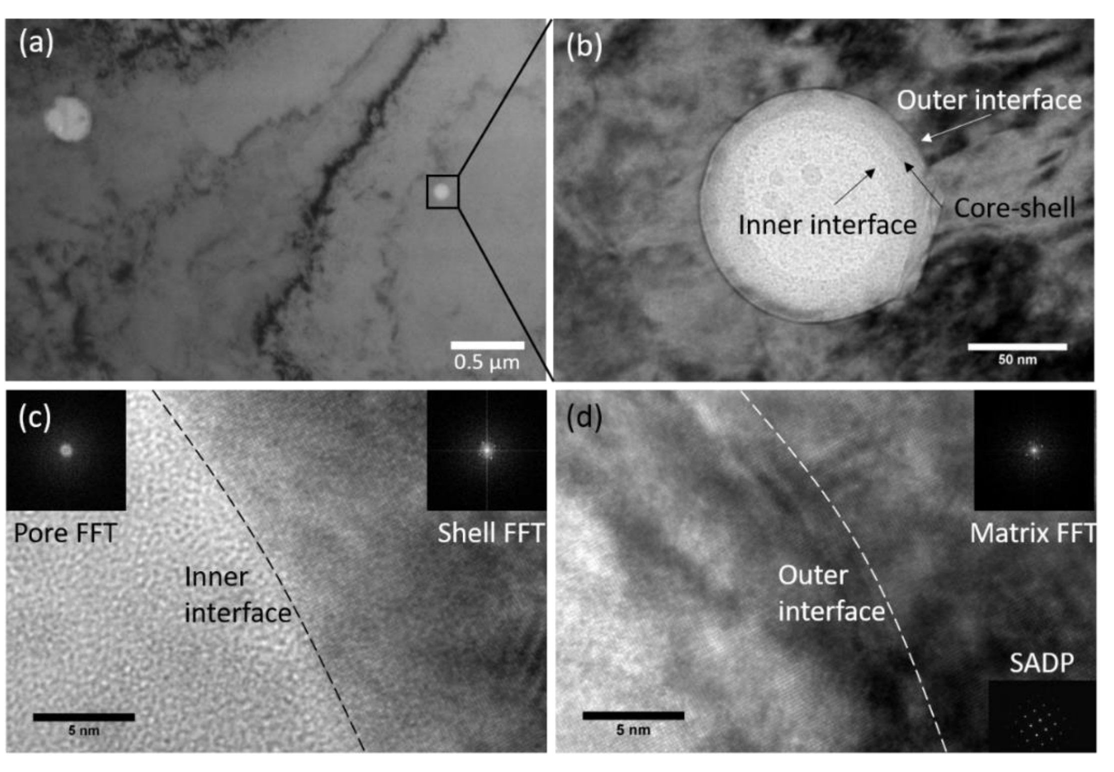

Figure 3. Bright-field TEM micrograph showing core-shell structure at pore.

The swelling rate of both wrought 316L and the DED samples were measured after irradiation. The swelling rate of wrought 316L was 0.09% at 0.35 dpa and 0.47% at 1.80 dpa. On the other hand, the DED samples had a swelling rate of 0.04% at 0.35 dpa and 0.07% at 1.80 dpa. The DED samples showed increased swelling resistance, but when comparing to other published data, it can be seen that more improvement can be made.

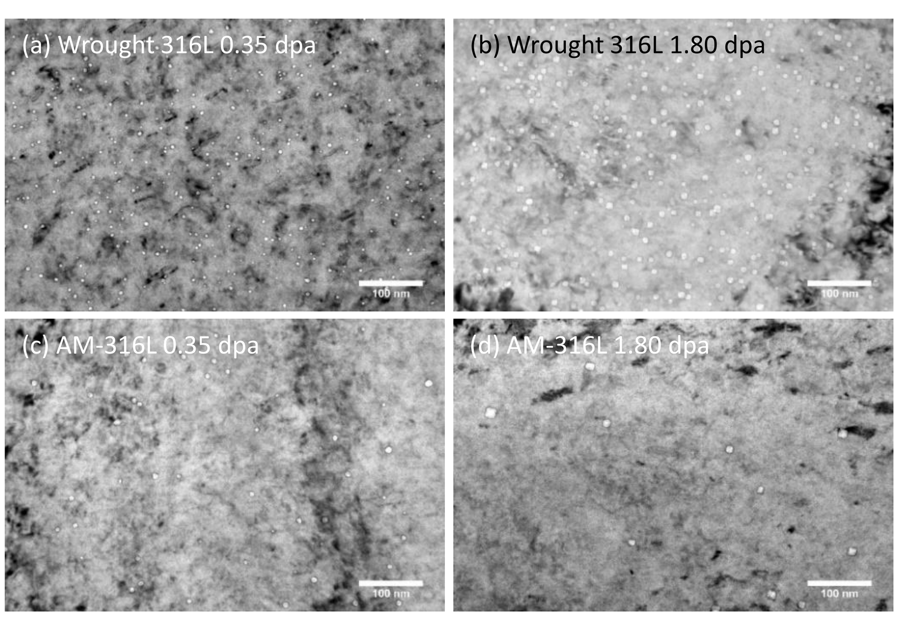

Figure 4. Bright-field TEM micrographs showing voids from proton irradiation. (a) Wrought 316L 0.35 dpa. (b) Wrought 316L 1.80 dpa. (c) AM-316L 0.35 dpa. (d) AM-316L 1.80 dpa

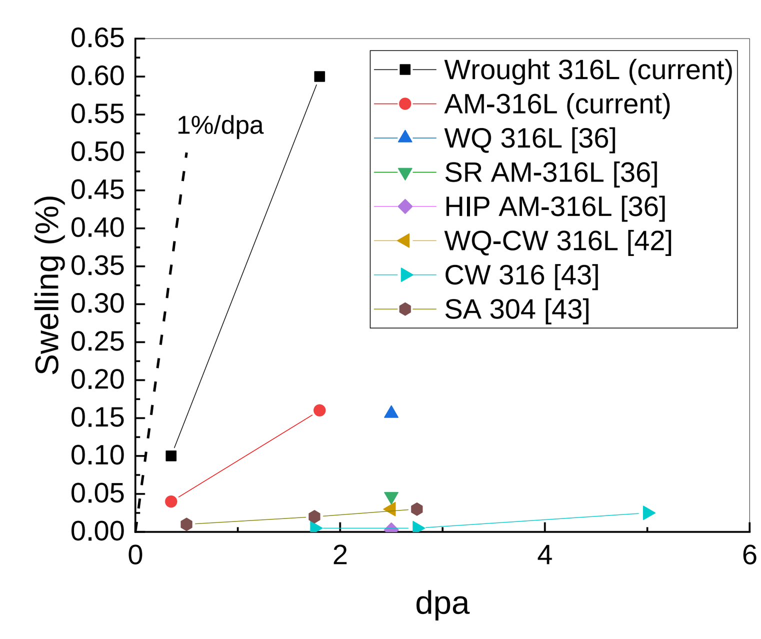

Figure 5. Comparison of AM-316L and wrought 316L proton irradiation swelling rate of current research to published data. WQ: water quenching, SR: stress relieve annealing, HIP: hot-isotropic pressing, CW: cold worked, SA: solution annealing.

The void denuded zone is currently being measured as a function of grain misorientation angle for the PBF samples. At small misorientation angles, there was no void denuded zone found. This finding was expected when comparing to previous published data that measured the void denuded zone as a function in misorientation angle in copperMeasurement of the void denuded zone for larger misorientation angles is currently in progress.

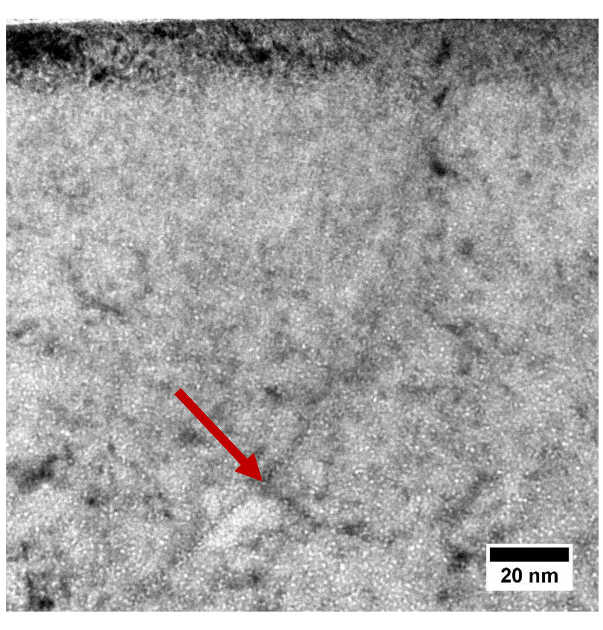

Figure 6. Bright-field TEM micrographs showing voids at small misorientation grain boundary for PBF samples. The arrow indicates the location of a grain boundary

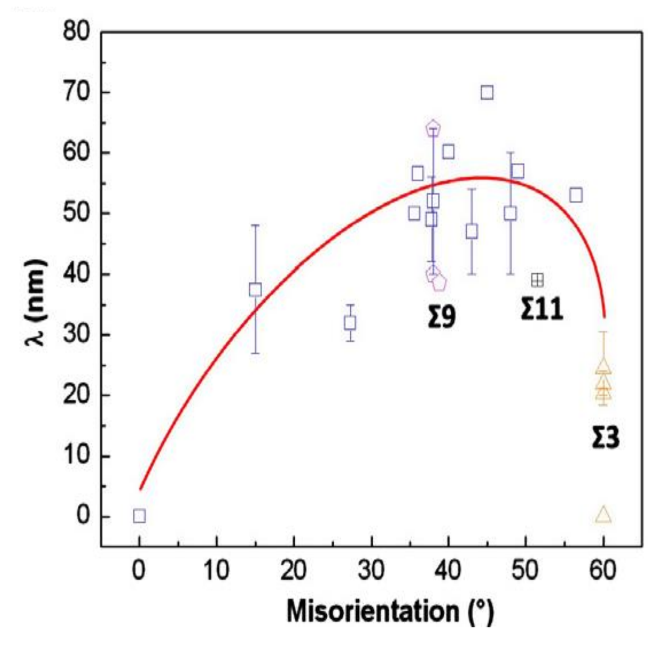

Figure 7. Void denuded zone width as a function of misorientation angle (Han et al., 2012).

References

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