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Changing the World's Energy Future

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Introduction

Additive manufacturing (AM) is the process of adding layers of material to build a part and can be built from a 3D CAD file. This allows for rapid prototyping of parts, thus decreasing the cost and time for deployment of new products. AM metals have shown similar or better performance than traditionally manufactured metals and can be fabricated into complex geometries, reducing the cost and weight parts without sacrificing strength. [1]

AM has great potential for fabricating parts for advanced nuclear reactors, including molten salt reactors (MSR), chiefly in the designs of advanced heat exchangers, which utilize intricate and complex structures to greatly enhance heat transfer.

Further studies are needed for the United States Nuclear Regulatory Commission (NRC) to qualify AM materials for extreme environments that are not addressed under current ASME codes. Namely, the corrosion performance of AM materials in these environments and its relation to characteristics including surface roughness, microstructure, porosity, and composition needs further study. Processing, including post-build heat treatments which alter the microstructure, and surface treatments also need to be understood for the ways in which they affect the corrosion behavior of the material [1].

AM 316L steel shows homogenous corrosion in molten salts due to the cellular microstructure being rich in Cr, different from the intergranular corrosion in wrought 316L due to Cr dealloying and oxygen/moisture impurities in the salt (Fig. 1). [2]

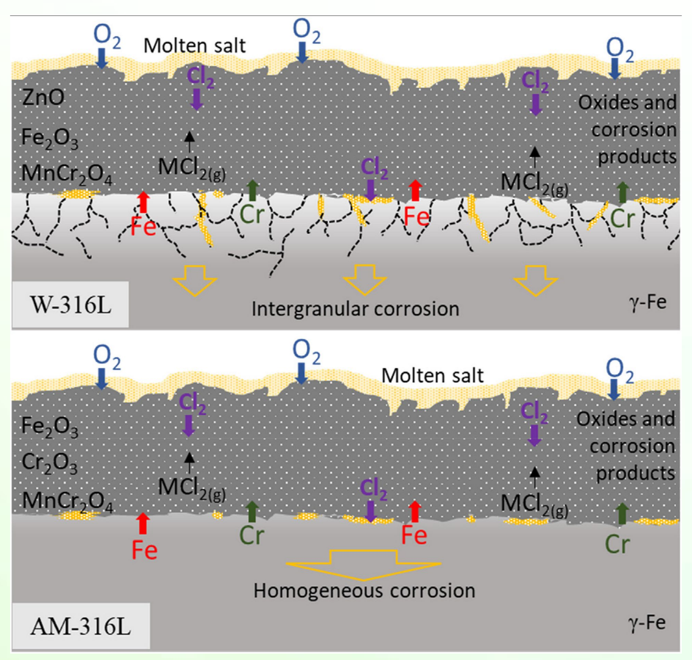


Figure 1. Diagram of MS corrosion od AM and Wrought 316L SS

Surface treatments, such as pickling in acid, can be used to remove the surface oxide layer and other contaminants, thus mitigating sources of corrosion in molten salts. If it is demonstrated to be successful, pickling could be used as a post-processing step to mitigate surface corrosion in molten salts for AM materials.

Sample Preparation

The 316H SS Specimens (loaves) in this investigation were printed at Los Alamos National Laboratory (LANL) using the laser powder bed fusion AM process (Fig. 2). The parameters used in the fabrication of the AM loaves were:
13-OW - Power (W): 275 - Speed (mm/s): 688 - LED (J/mm3): 95
Dimensions: 126 x 25 x 25 mm.

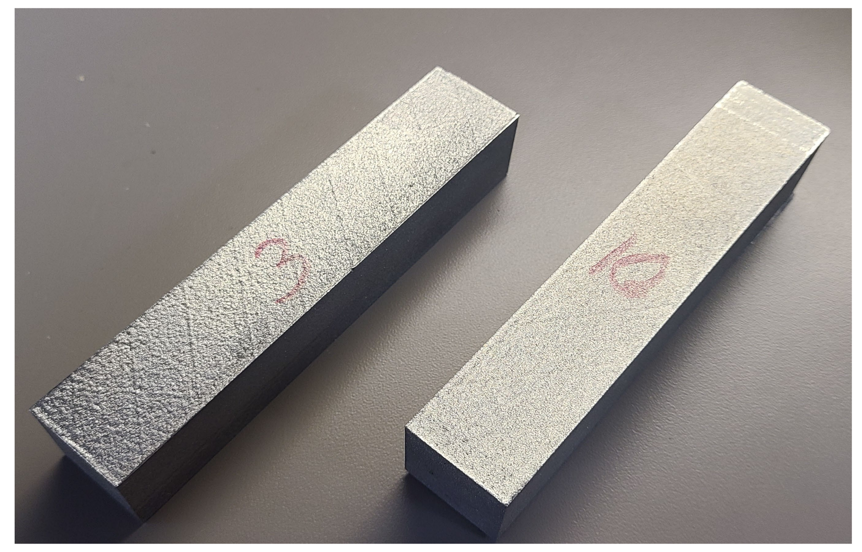


Figure 2. LANL LPBF 316H SS square bars (i.e. "loaves"), featuring loaves #3 and #10

Heat Treatment:

The loaves 3 and 10 were heat-treated at 650 C for 94 hours and then air-cooled to mitigate warping during the sectioning process.

Sectioning Process:

Samples were sectioned using electrical discharge machining (EDM) with unique Identifiers engraved on the EDM cut side (Fig. 3). The sample slices from 3 & 10 will be used to investigate post treatment microstructure.

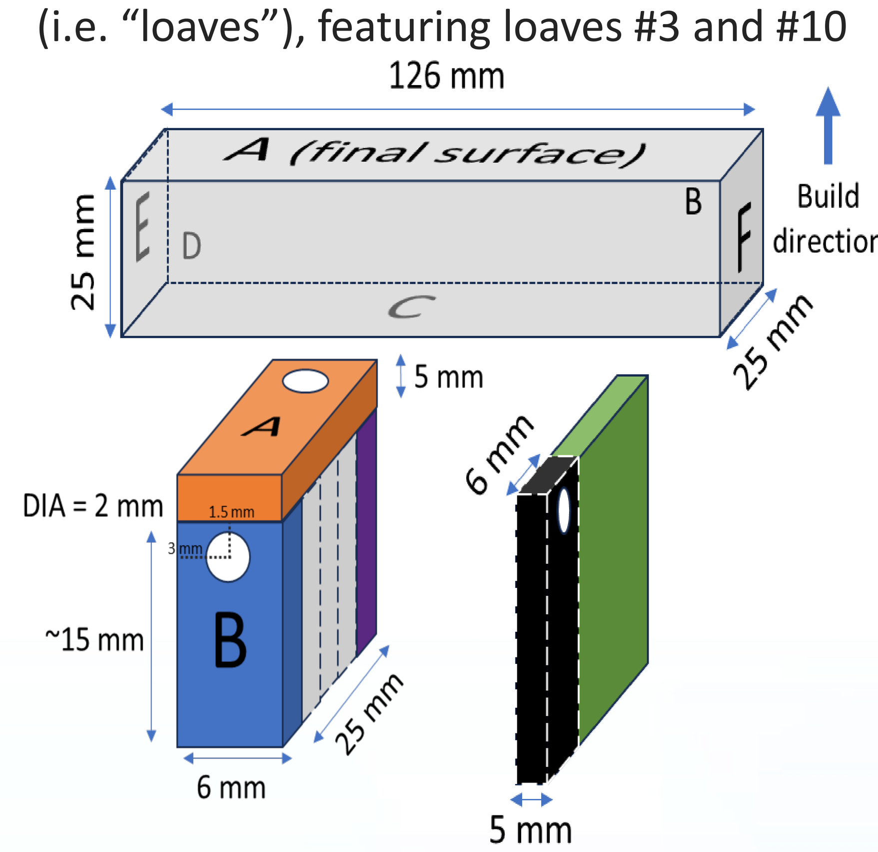
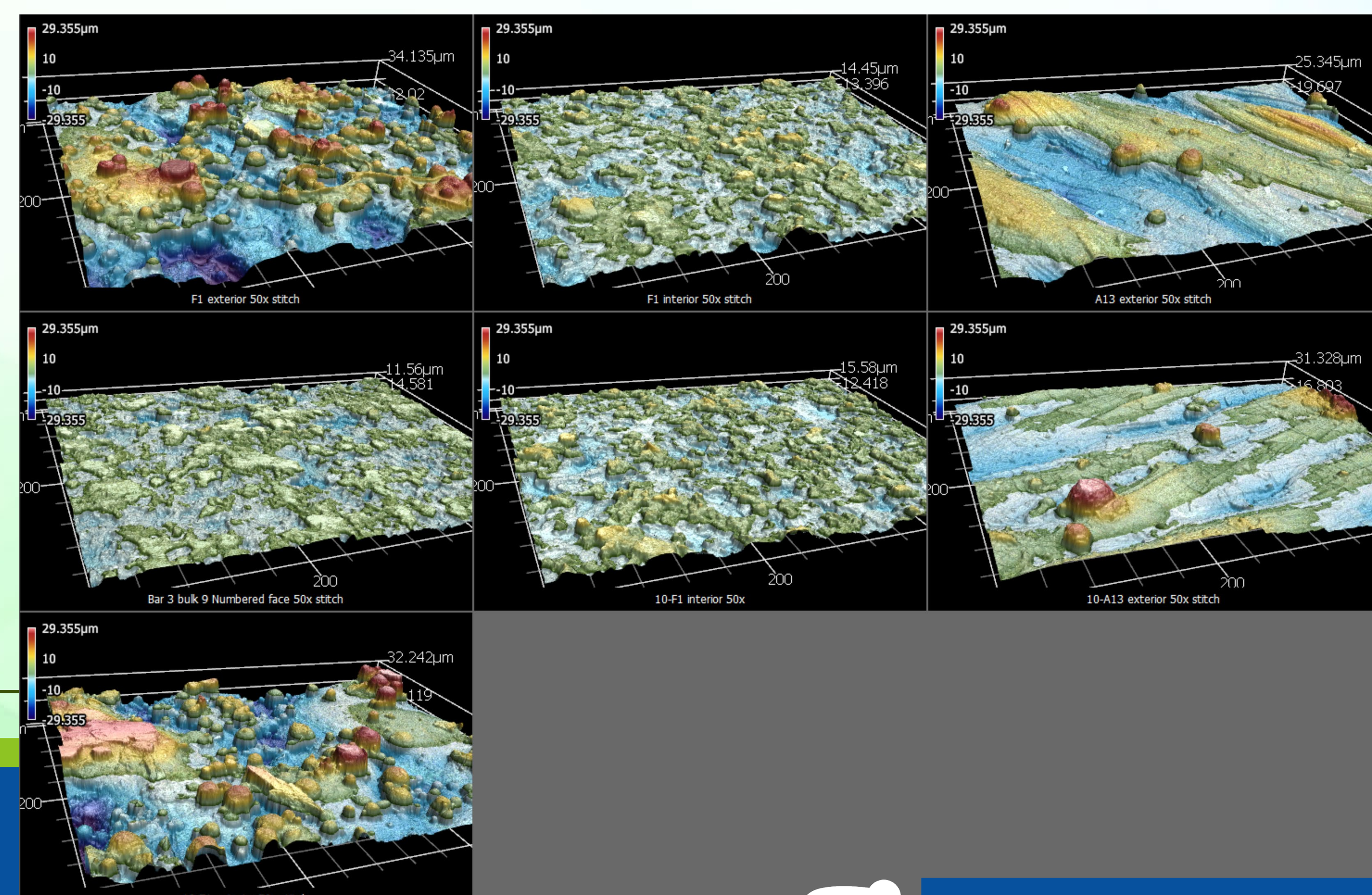


Figure 3. Sectioning diagram LANL LPBF 316H SS square bars (i.e. "loaves").

Surface Analysis:

Samples from surfaces A, E, and F were analyzed using a Keyence 3D Surface Profiler after heat treatment to see the roughness of the material (Fig. 4)



...ure 4. 3D map of surface roughness of loa... Bulk.

Pickling Tests

ASTM A380/A380M-17 provides the procedures for descaling, pickling, and passivation of stainless steels. The procedure using a mixture of HF and HNO_3 acids, from Table A2.1, was chosen and studied. Samples were exposed to a 15% HNO_3 – 1.5% HF mixture at temperatures of 20–60°C and times of 5–10 min.

A successful pickling protocol was found by immersing the sample for 5 min at 60°C, brushing the scale with a brass brush, and immersing the sample again for 5 min at 60°C. This left a visually clean and "white-pickled" finish surface on the sample (Fig. 5).

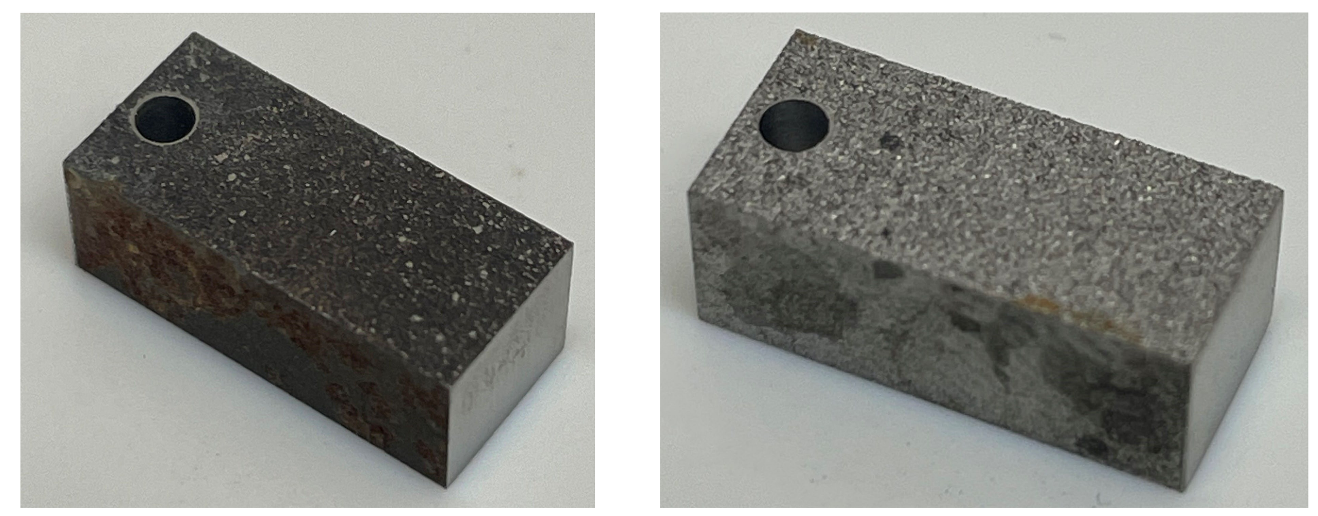


Figure 5. AM 316H sample before (left) and after (right) pickling.

Colleagues at Argonne National Laboratory have found success pickling similar AM 316H materials using a mixture of H_2SO_4, HCl, and HNO_3 also provided in ASTM **. This pickling protocol, along with other testing of the heat treatment and solution-annealing steps, will be studied to establish the most robust and effective way to prepare AM fabricated 316H materials for high temperature molten salt environments.

Future Work

Samples which have undergone varied heat treatments and pickling protocols will be exposed to molten chloride environments and their corrosion characterized. This will provide greater insight into the proper pickling techniques for AM materials deployed in molten salt environments, with follow-on irradiation-corrosion experiments necessary for NRC license applications using AM 316H parts in molten salt reactors.

References: [1]Jokisaari A., *et al.*; *Progress in Nuclear energy* 2024, 174, 105296. [2] Abu-warda, N., *et al.*; *Journal of Materials Research and Technology* 2022, 20, 3949–3961.

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