

## Effect of Cellular structure of the additively manufactured 316 stainless steels on mechanical property and molten salt corrosion in NaCl-MgCl<sub>2</sub>

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### 1. Introduction

Additive manufacturing (AM) is defined as a process of joining materials to make objects from 3D model data, layer by layer, as opposed to conventional subtractive manufacturing methodologies [1]. Additively manufactured stainless steel has been actively researched for applications in nuclear power plant components and has already been applied in several nuclear power plants. In this study, we consider the application of additively manufactured stainless steel to the internal components of the molten salt reactor. 316L stainless steel is the most used structural material, especially in nuclear power plant industries because of its excellent resistance to general corrosion, good high-temperature mechanical properties, and extensive weldability. The microstructure of additively manufactured stainless steel has a cellular structure with element segregation and dislocations at cell boundaries as a unique microstructural feature, and these microstructural features result in high yield strength and high hardness [2]. Previous studies have reported that cellular structure can be decomposed and dissolved by heat treatment. In this study, 316 stainless steel was additively manufactured using the directed energy deposition (DED) method, and the specimens were heat treated at various temperatures to observe changes in microstructure and hardness. To determine the effect of the cellular structure on molten salt corrosion, as-built DED 316 specimens and heat-treated DED 316 specimens are exposed to NaCl-MgCl<sub>2</sub> molten salt at 700°C for 500 hours. This study demonstrates the influence of microstructure on molten salt corrosion of additively manufactured stainless steel.

### 2. Methods

316 stainless steels were additively manufactured by DED (directed energy deposition) method. The additive manufacturing parameters are as follows; laser power of 1800 W, scan speed of 1000 mm/min, feed rate of 13g/min, and layer thickness of 0.9mm. The specimens for microstructural observation were prepared for microstructural observation by polishing with SiC papers and Diamond Suspensions and finished with Colloidal Silica. The Vickers hardness test was performed using an HM-122 micro hardness tester (AKASHI, Japan) with a load of 500 gf. To observe

changes in microstructure by temperature, as-built specimens were heat treated for 1 hour at each temperature from 400°C to 1100°C at 100°C intervals to obtain eight heat treated specimens. The specimens were etched with a 10% oxalic acid solution, and the cellular structure was observed using SEM. SEM images were obtained using an EDS detector mounted on a JSM-7200F scanning electron microscope (SEM) operating at an accelerating voltage of 20 kV. Schematic illustration of the static immersion corrosion test designed for molten salt corrosion testing is shown in Figure 1. The inside of the crucible was filled with Ar. Operations were performed in the glove box and oxygen and moisture were maintained below 10 ppm. NaCl-MgCl<sub>2</sub> eutectic salts (43% NaCl-57% MgCl<sub>2</sub>) were prepared by physical mixing. NaCl-MgCl<sub>2</sub> eutectic salts were purified by heat treatment at 300 °C for 48 hours and adding Mg to remove impurities such as moisture and oxygen. The molten salt corrosion samples were produced with a size of 10mm x 20mm x 1mm, and each surface was polished with 1000# of SiC paper. The corrosion specimen was hung on an alumina tube and prevented from galvanic corrosion by an alumina spacer. Corrosion tests were performed at 700°C for 500 hours.

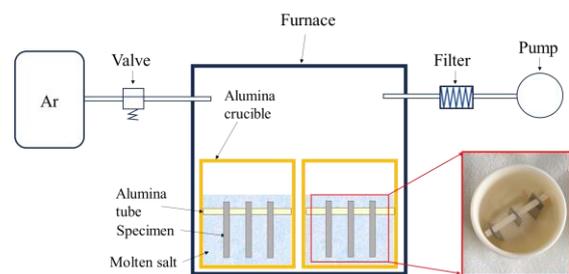


Fig. 1. Schematic of molten salt corrosion test

### 2. Results

#### 2.3 Sample preparation

As shown in Figure 2, EDS analysis of as-built specimens revealed elemental segregation at cell boundaries with enrichment of Mo and depletion of Mn and Fe. To observe the cellular structure, the specimens were chemically etched and observed by SEM. The cellular structure of each heat-treated specimen is shown in Figure 3. The cell structure was found to have

a diameter of approximately 4 $\mu$ m to 5 $\mu$ m. As the heat treatment temperature increased, the cell size increased. The elemental segregation gradually dissolved due to diffusion and redistribution of elements, and the cell structure completely disappeared in the 1100 $^{\circ}$ C heat-treated specimen. On the other hand, as the heat treatment temperature increased, the hardness of the specimen decreased. The relationship between cell size and Vickers hardness is shown graphically in Figure 4. Depending on the heat treatment temperature, the bubble size tended to increase slightly, while the hardness decreased.

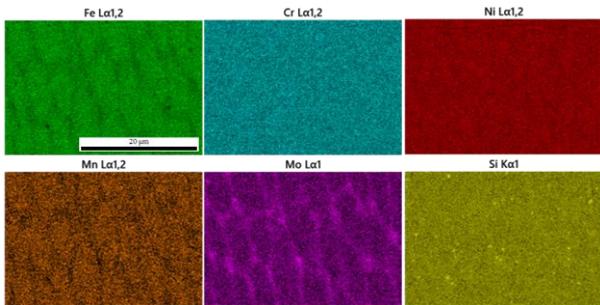


Fig. 2. Element segregation on the cellular structure

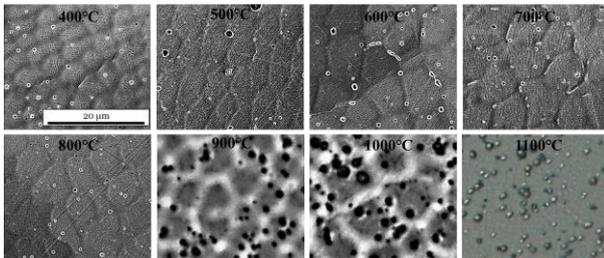


Fig. 3. Cellular structure by heat treatment temperature

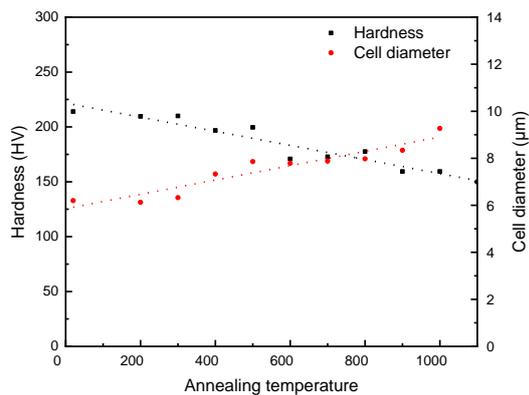


Fig. 4. Relationship between cell size and Vickers hardness

To confirm the effect of element segregation on molten salt corrosion, the corrosion mass of as-built specimens and 1100  $^{\circ}$ C heat treatment specimens were compared. The mass loss of each sample after a corrosion test in NaCl-MgCl<sub>2</sub> molten salt is shown in

Figure 5. The 1100 $^{\circ}$ C heat treatment specimen, in which the cell structure and element segregation disappeared, showed less corrosion than the as-built specimen. The cross-sections of the two corrosion specimens are shown in Figure 6. Similar to the mass loss results, it was observed that the as-built specimen had a deeper corrosion depth than the heat treatment specimen.

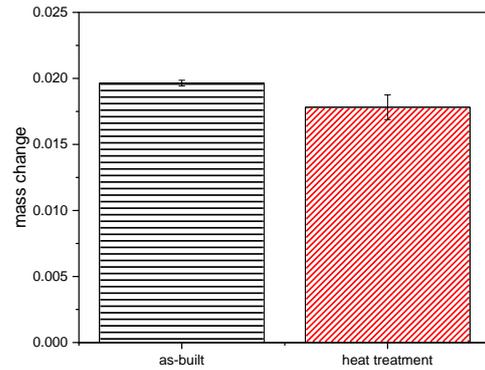


Fig. 5. Mass loss after molten salt corrosion test

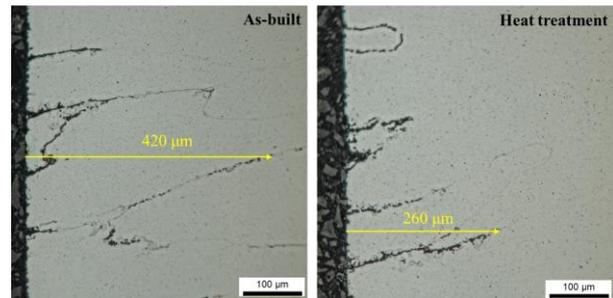


Fig. 6. Corrosion depth after molten salt corrosion test

### 3. Conclusions

The additively manufactured 316 stainless steels revealed a cellular structure with Mo segregation occurring at the cell boundaries. This cellular structure exhibited remarkable hardness and contributed significantly to enhancing mechanical strength. However, the segregation of elements within these cellular structures was observed to accelerate molten salt corrosion in NaCl-MgCl<sub>2</sub> salt environments. To confirm this, further investigation into microstructural analysis of corrosion specimens is required.

### REFERENCES

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- [2] T.R. Smith, J.D. Sugar, C.S. Marchi, J.M. Schoenung. Strengthening mechanisms in directed energy deposited austenitic stainless steel, Acta Mater., 164, 2019.