

## Neutron diffraction measurement of residual stress in high melting point metal 3D printing

Dong-Kyu Kim<sup>a\*</sup> and Wanchuck Woo<sup>a</sup>  
<sup>a</sup>Neutron Science Center, KAERI, South Korea  
\*Corresponding author: kimdk@kaeri.re.kr

### 1. Introduction

Recently, metal 3D printing technology has been considered as one of the most innovative manufacturing technology due to its various advantages. In particular, there have been urgent needs for technology using high melting point metal (HMPM) for the extreme environmental applications such as defense weapon, aerospace rocket, power plant. In this study, we conducted feasibility test for application of Mo and W alloys in the direct energy deposition (DED), which is one of the major additive manufacturing (AM) technology. Nominal melting temperature of pure W and Mo is 3422 °C and 2617 °C, respectively, which ranks at the first and sixth places among all materials on earth. So as-processed HMPMs are expected to have strong residual stress due to the use of high laser power in the 3D printing process. We measured the residual stress and microstructure of Mo and W alloys processed by DED using non-destructive neutron diffraction method in conjunction with cutting-based contour method.

### 2. Methods and Results

#### 2.1 Sample Preparation

Two DED specimens were processed using Mo and W power alloys, of which chemical compositions are C 0.25, N 0.002, O 0.03, Fe 0.01, Ni 0.005, Si 0.005, Ti 0.05, Zr 0.09, W 0.012, Mo balance and Re 0.05, O 0.003, W balance. Power particles were in spherical shape and their sizes were approximately 45-145  $\mu\text{m}$  in diameter. The specimens were processed by AM machine in INSSTTEK using orthogonal laser scanning in-between each layer, the powder layer thickness of 150  $\mu\text{m}$  under Ar atmosphere. Constant scanning speed of 0.85 m/min was used and layer powers of 800 W and 1200 W were used for Mo and W alloys, respectively. The powder was deposited on the ferritic steel base plate with thickness of 4 mm. The sample dimension was 15 mm (x)  $\times$  15 mm (y)  $\times$  40 mm (z) in the shape of rectangular bar. Note that the x, y, z axes correspond to the transverse (TD), normal (ND, thickness), longitudinal (LD) directions, respectively.

#### 2.2 Neutron diffraction experiment

Neutron diffraction measurements were performed on the KOWARI diffractometer at the Bragg's Institute at the Australian Neutron Science and Technology

Organization (ANSTO). All strain components were measured using the (211) diffraction plane. A wavelength of 1.81 Å was used. The gauge volume was 2 (x)  $\times$  2 (y)  $\times$  2 (z) mm<sup>3</sup>. The measurements were carried out on the plane located in the middle of the longitudinal direction.

For determination of lattice strain and residual stress, we made calculations under constant  $d_0$  assumption. The stress-free lattice spacing  $d_0$  was measured in the corner of the specimens.

#### 2.3 Residual stress distribution

Fig. 1 shows residual stress distribution in the cross-section in the middle of LD in the Mo alloy specimen processed by DED. General findings in this study are consistent with the fact that residual stresses tend to be compressive inside the AM specimens and tensile near surfaces [1]. In addition, it is noticeable that there were apparently different distributions of residual stress among the residual stress components. While LD and TD residual stress can be referred as the in-plane components. ND residual stress can be referred as out-of-plane components when it comes to the building layer. While LD and TD components shows relatively high tensile residual stress in the top and bottom regions, ND component exhibits strong tensile residual stress both lateral edges (max. +230 MPa).

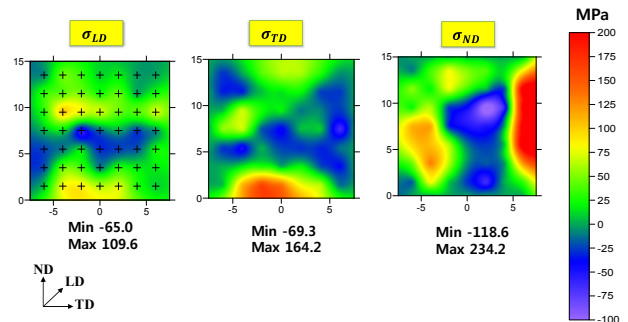


Fig. 1 Residual stress distribution in the cross-section of the middle of the longitudinal direction in the Mo alloy specimen processed by DED.

Fig. 2 shows residual stress distribution in the cross-section in the middle of LD in the W alloy specimen processed by DED. In general, magnitude of residual stress in W alloy is much less than in Mo alloy: approximately 50 MPa in W alloy and -125~250 MPa in Mo alloy. In the W alloy specimen, discontinuous distribution of compressive residual stress was found in the central region of the specimen. In the Mo alloy specimen, however, continuous distribution of

compressive residual stress was found in the central region and tensile residual stress was formed near the surface regions to satisfy force equilibrium over the whole area.

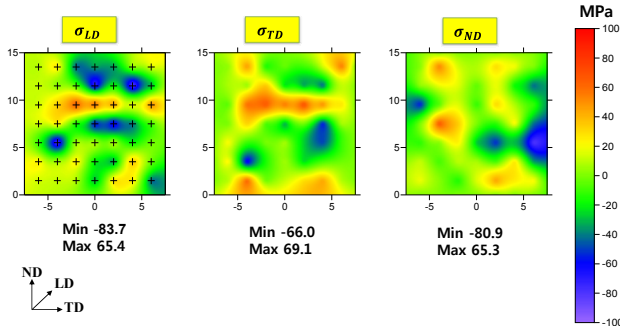


Fig. 2 Residual stress distribution in the cross-section of the middle of the longitudinal direction in the W alloy specimen processed by DED.

There are three conceivable reasons why the much less intensity of residual stress in the W alloy specimen: i) coefficients of thermal expansion (CTEs), ii) intergranular micro-cracks, and iii) annealing effect. First, CTEs of HMPMs are very low:  $5.2 \times 10^{-6}/K$  and  $4.5 \times 10^{-6}/K$  for Mo and W alloys, respectively. So the thermal deformation and the resultant residual stress will be obviously small based on the fact that the CTEs of the conventional materials like Al and steel alloys are  $23.6 \times 10^{-6}/K$  and  $12.2 \times 10^{-6}/K$ . Second, the original residual stress may be relaxed due to the occurrence of significant amount of micro-cracks as shown in Fig. 3. Due to the use of high power laser beam, the microstructure revealed very coarse and columnar grain structure resulting from the grain growth. In addition, there were many intergranular micro-cracks formed along the degraded grain boundary as indicated by dotted circles. Third, annealing effect may contribute to the relief of residual stress. It is attributed to the high melting pool temperature, slow scanning speed of 0.85 m/min (14 mm/s), and large melting pool size of  $\sim 500 \mu m$ . So the cooling speed of the melt pool was considerably slow in contrary to the conventional alloys by DED and the AM specimens processed by the power bed fusion.

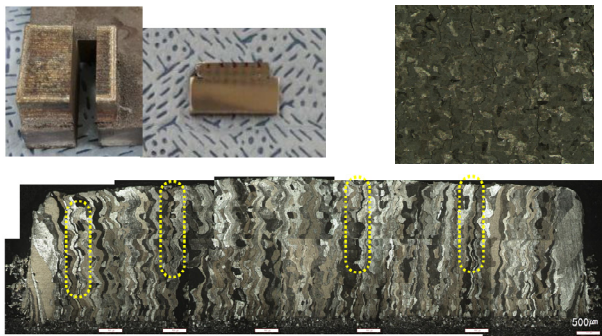


Fig. 3 micro cracks found in the W alloy specimen processed by DED.

### 3. Conclusions

In this study, we conducted feasibility test for application of Mo and W alloys in the direct energy deposition (DED) by measuring the residual stress using non-destructive neutron diffraction method. In contrary to our expectations, the residual stress in HMPMs was found to be relatively low compared to neither the conventional materials nor the powder bed fusion AM. There are three conceivable reasons why the much less intensity of residual stress in the W alloy specimen: i) coefficients of thermal expansion (CTEs), ii) intergranular micro-cracks, and iii) annealing effect.

### 4. Acknowledgment

The authors are thankful for the support and DED sample fabrication to Mr. Dae-Jung Kim at INSSTK metal 3D printing company.

### REFERENCES

[1] P. Mercelis and J.-P. Kruth, Residual stresses in selective laser sintering and selective laser melting, Rapid Prototyping Journal, Vol. 12, pp.254-226, 2006.