Microstructure characterization and micro-hardness of Fe-9Cr ODS alloy produced by laser powder bed fusion

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Abstract. Oxide dispersion strengthened (ODS) alloys have been considered as the promising candidates for advance Generation-IV fission reactor application due to excellent radiation tolerance and high temperature mechanical properties but traditional manufacturing process is complex. A 9Cr-ODS alloy with nominal composition of Fe-9Cr-1.5W-0.3Ti-0.3Y2O3 (wt %) was produced by laser powder bed fusion (LPBF) which is a laser additive manufacturing technique. The microstructure and nanoscale oxide particles were characterized by using optical microscope (OM), electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). The micro-hardness was tested by hardness tester. The results showed that the equiaxed grains with an average size of 3.8 μm were obtained. The dispersed oxide particles with 10-20 nm were observed in LPBFed 9Cr-ODS alloy. The micro-hardness of built sample was about 460 HV.

1. Introduction

The harsh work environments of the advanced Generation-IV fusion reactors require structural materials with outstanding performances. Nanostructured oxide dispersion strengthened (ODS) alloys present excellent irradiation resistance and mechanical properties because of the high-density of nano-sized oxides, which is considered as one of the most promising candidate materials for fuel cladding in fast reactors [1-3]. At present, powder metallurgy (PM, such as mechanical alloy + high temperature consolidation) is a conventional process to produce ODS alloys [4-7]. Although the powder metallurgy is a ripe and effective method to produce ODS alloys with high-density of nano-sized oxide particles and outstanding performances, the long manufacturing period, high cost and less flexibility are still insurmountable.

Recently, developments in laser additive manufacturing (LAM) have offered a potential method for structural materials application in the future advanced reactors. The LAM has unique advantages compared to the conventional PM such as, short manufacturing cycle, low cost and great flexibility. The ODS alloys with high chromium content produced by LAM have been explored in recent years. T. Boegelein et al have successfully manufactured the high chromium content and Al-containing ODS alloy named PM2000 by selective laser melting (SLM) process [8, 9]. The fine precipitates with high-density were obtained. A Fe-18Cr Al-free ODS alloy which was manufactured by electron beam selective melting (EBSM) presented high-density precipitates and tensile strength of 1226 MPa [10].
However, the aging embrittlement and irradiation embrittlement are inevitable for the ODS alloys with high chromium content [11]. Therefore, the low-Cr (9-12Cr) ODS alloys are considered for application in reactors with higher temperature and irradiation dose.

In this study, a 9Cr-ODS alloy was produced by laser powder bed fusion (LPBF) which is a powder-bed metal printing system. The microstructure and nano-sized particles were characterized and examined carefully.

2. Experimental

The high-purity elemental powders (Fe, Cr, W, Ti) and nano-scale Y$_2$O$_3$ powder were mixed according to the ratio of Fe-9Cr-1.5W-0.3Ti-0.3Y$_2$O$_3$ (wt%). The mechanical alloying (MA) was carried out in Fritsch P-5 planetary mill under high purity argon (99.99%) atmosphere for 50 hours. Detailed MA parameters for producing the supersaturated alloy powder are detailed in a previous publication [12].

The LPBF machine was a self-assembly apparatus and mainly composed of CO$_2$ laser, numerical control system, coaxial powder feeder and inert gas protection system. Scanning strategy was zigzag scan vectors. The substrate was AISI 1045 steel. The schematic of process is shown in Fig.1. In this process a thin layer of powder is first deposited onto the substrate. The laser beam is scanned onto the powder bed to form solidified layers. Scanning path is generated by the CAD and sliced software. The parts can be built up layer-by-layer into a net-shape. The process parameters used in this study are listed in Table 1. The direction of deposition is along the Z direction, X direction is the scanning direction.

![Figure 1. The schematic of process](image)

Table 1. The process parameters in this study

| Laser power | Scanning speed | Layer thickness | Hatch distance |
|-------------|----------------|-----------------|---------------|
| 1500 W      | 6 mm/s         | 0.8 mm          | 1.5 mm        |

The microstructure of the YOZ section was characterized by an optical microscope (OM, OLYMPUS GX71) and a JSM-7001F scanning electron microscope (SEM) with an electron backscattered diffraction (EBSD). The sample was etched by using 5% nital solution for observation of OM. The EBSD specimens were prepared by mechanical ground and electrolytic polishing. The electrolytic polishing was performed in 10% HClO$_4$+90% C$_2$H$_5$OH solution with 20 V voltage at room temperature. The microstructure was further investigated using a Titan G$^{+}$60-300 transmission electron microscope (TEM). The TEM thin foil specimens were prepared by a JIB-4600F focused ion beam (FIB). The Vickers micro-hardness tests were carried out by a 41MVDtm digital micro Vickers hardness tester. 5 indentation tests were made for the built sample to obtain the average value of hardness. The load was 0.98 N and loading time was 10 s.
3. Results and discussion

The Fig.2 (a) shows the OM image of LPBFed 9Cr-ODS alloy in vertical section (YOZ). The equiaxed grains are observed. The EBSD also presents the similar feature (see the Fig.2 (b)) and there is a fine grain size distribution (Fig. 2 (c)). More than 90% of grains are less 5μm and a main peak at ~0.5-1.0μm is obvious. The fine grains are attribute to the higher cooling rate. The cooling rate can reach up about $10^5$-$10^6$ K/s during the laser additive manufacturing [13]. The high cooling rate can result in the rapid solidification as well as inhibits the growth of grains. The average grain size of 3.8 μm is smaller than the SLMed PM2000 (~20μm) and EBSMed ODS alloy (~5-10μm) [8, 10]. This could be associated with the differences of alloy composition and LAM process.

![Figure 2. The (a) OM and (b) EBSD images of LPBFed 9Cr-ODS alloy in YOZ section, (c) the corresponding size distribution of grains.](image)

The lath martensites are observed in the LPBF built 9Cr-ODS alloy (see the Fig.3). A typical feature of 9Cr-ODS alloy is that the phase transformation from ferrite/martensite ($\alpha$) to austenite ($\gamma$) can be controlled by heat treatment [14]. Therefore, some lath martensites formed during the LPBF because of the higher cooling rate.

In order to clarify the morphology of oxide particles, the high angle annular dark field (HAADF) images of STEM analysis were conducted (Fig. 4). These oxide particles exhibit darker contrast than the matrix in the HAADF image, which are attribute to lower mas compared to the matrix. A large amount of oxide particles with ~10-20 nm are found and these particles are homogeneously dispersed in the matrix.

Cr, Y and Ti et al elements were supersaturated solution in Fe matrix after mechanical alloying. During the laser additive manufacturing, the loss of Y and Ti was found due to some micron-sized oxides formed in the melting pool [9, 15]. And these oxides in melting pool would float and agglomerate driven due to continuous stirring and lower mass density than ODS alloy matrix. However, Y, Ti et al elements diffuse slowly in the $\alpha$-Fe matrix [16]. Thus, a certain amount of alloy elements appears to still be atomic supersaturated solution in matrix. The finer precipitates (seen in the Fig. 4) may form in the solidified matrix after deposition during the repeated heating cycles caused by laser powder bed fusion.
Figure 3. The TEM micrograph of as LPBF built 9Cr-ODS alloy

Figure 4. The STEM HAADF images of LPBF built 9Cr-ODS alloy

The Vickers micro-hardness of built sample is about 463 ± 15 HV which is similar with the 9Cr-ODS Fe-matrix alloy produced by conventional process [17].

4. Conclusion

The 9Cr-ODS alloy was successfully produced by laser powder bed fusion. The characterization of grain morphology and nano-sized oxide particles of LPBFed 9Cr-ODS alloy was studied. The fine grains (average size about 3.8 μm) are formed in built 9Cr-ODS alloy. Some lath martensites are introduced due to the high cooling rate. Dispersed nanoscale particles (~10-20 μm) are obtained in the LPBFed 9Cr-ODS alloy. The micro-hardness of this sample is up ~460 HV.

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References

[1] G. R. Odette, M. J. Alinger, B. D. Wirth, Recent Developments in Irradiation-Resistant Steels, Annu. Rev. Mater. Res. 38 (2008) 471-503.
[2] S. Ukai, T. Nishida, H. Okada, et al, Development of Oxide Dispersion Strengthened Ferritic Steels for FBR Core Application, J. Nucl. Sci. Technol. 34 (1997) 256-263.
[3] R. L. Klueh, J. P. Shingledecker, R. W. Swindeman, et al, Oxide dispersion-strengthened steels: A comparison of some commercial and experimental alloys, J. Nucl. Mater. 341 (2005) 103-114.
[4] M. J. Alinger, G. R. Odette, D. T. Hoelzer, On the role of alloy composition and processing
parameters in nanocluster formation and dispersion strengthening in nanostructured ferritic alloys, Acta. Mater. 57 (2009) 392-406.

[5] S. Ukai, S. Mizuta, T. Yoshitake, et al, Tube manufacturing and characterization of oxide dispersion strengthened ferritic steels, J. Nucl. Mater. 283-287 (2000) 702-706.

[6] C. Y. Lu, Z. Lu, C. M. Liu, Microstructure of nano-structured ODS CLAM steel by mechanical alloying and hot isostatic pressing, J. Nucl. Mater. 442 (2013) S148-S152.

[7] Z. Y. Li, Z. Lu, R. Xie, et al, Effect of spark plasma sintering temperature on microstructure and mechanical properties of 14Cr-ODS ferritic steels, Mater. Sci. Eng. A 660 (2016) 52-60.

[8] T. Boegelein, S. N. Dryepondt, A. Pandey, et al, Mechanical response and deformation mechanisms of ferritic oxide dispersion strengthened steel structures produced by selective laser melting, Acta. Mater. 87 (2015) 201-215.

[9] T. Boegelein, E. Louvis, K. Dawson, et al, Characterisation of a complex thin walled structure fabricated by selective laser melting using a ferritic oxide dispersion strengthened steel, Mater. Char. 112 (2016) 30-40.

[10] R. Gao, L. Zeng, Q. F. Fang, et al, Characterization of oxide dispersion strengthened ferritic steel fabricated by electron beam selective melting, Mater. Des. 89 (2016) 1171-1180.

[11] A. Kimura, R. Kasada, N. Iwata, et al, Development of Al added high-Cr ODS steels for fuel cladding of next generation nuclear systems, J. Nucl. Mater. 417 (2011) 176-179.

[12] R. Xie, Z. Lu, C. Lu, et al, Microstructure and mechanical properties of 9Cr oxide dispersion strengthened steel produced by spark plasma sintering, Fusion. Eng. Des. 115 (2017) 67-73.

[13] K. Yang, P. Rometsch, C. Davies, et al, Effect of heat treatment on the microstructure and anisotropy in mechanical properties of A357 alloy produced by selective laser melting, Mater. Des. 154 (2018) 275-290.

[14] S. Ukai, S. Mizuta, M. Fujiwara, et al, Development of 9Cr-ODS martensitic steel claddings for fuel pins by means of ferritic to austenite phase transformation, J. Nucl. Sci. Technol. 39 (2002) 778-788.

[15] Y. Shi, Z. Lu, H. Xu, et al, Microstructure characterization and mechanical properties of laser additive manufactured oxide dispersion strengthened Fe-9Cr alloy, J. Alloys. Compd. 791 (2019) 121-133.

[16] C. Hin, B. Wirth, J. Neaton, Formation of Y2O3 nanoclusters in nanostructured ferritic alloys during isothermal and anisothermal heat treatment: A Kinetic Monte Carlo study, Phys. Rev. B. 80 (2009) 134118.

[17] H. R. Z. Sandim, R. A. Renzetti, A. F. Padilha, et al, Annealing behavior of ferritic-martensitic 9%Cr-ODS-Eurofer steel, Mater. Sci. Eng. A. 527 (2010) 3602-3608.