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Scattering-angle Selective STEM Images for a Co-Cr-Mo Alloy Fabricated by Selective Laser Melting

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Cellular structure of a Co-Cr-Mo alloy fabricated by selective laser melting (SLM) has been investigated by scanning transmission electron microscopy (STEM). Angle-selective STEM detector provides diverse information on the microstructures of the alloy. Annular dark field (ADF)-STEM images with medium scattering angle visualize defect distributions at the interfaces and stacking faults crossing the interfaces. Thickness distributions of thin film specimen made by an electro polishing affect the contrasts in the STEM images and then require careful considerations for correct interpretations of the images.

1. Introduction

Additive manufacturing (AM), also known as 3D printing, is a unique technique for fabricating metallic works with complex structures [1]. AM techniques have been energetically studied in several industrial fields such as medical apparatus in the past decade. Scanning laser beam or electron beam give an alternative repetition of partial melt and rapid cooling on the work surface. Thus fabricated metals have complex microstructures such as cellular (columnar) structure [2]. Growth mechanism of the structure has also been discussed [3]. However, there are a few reports on detailed structures, especially about crystal defects. We report microstructures of a Co-Cr-Mo made by a selective laser melting (SLM [1-3]) studied by scanning transmission electron microscopy (STEM). STEM images are presented and discussed in terms of analytical technique and defects in the alloy.

2. Experimental

A Co-Cr-Mo alloy rot with a diameter of about 6 mm was made by a SLM. The alloy powder raw material with an average diameter of 20 μm has the composition of ASTM F75; Cr/26.5 ~ 30.0 mass%, Mo/4.5 ~ 7.0 mass%, C/≦ 0.35 mass%, and N/≦ 0.25 mass% for main elements. Thin films were obtained by an electro-polishing for STEM. The observation plane is normal to the manufacturing direction.

A field emission (FE) STEM instrument, a model Talos F200X (FEI, now Thermo Fisher) equipped 3 types of annular dark field (ADF) detectors was used with the primary electron energy of 200 keV. The surface of the thin film was observed by a FE scanning electron microscope (SEM, a model ULTRA55, Carl Zeiss) with the primary electron energy of 1 keV.

3. Results and discussion

Figure1 shows STEM images recorded for the Co-Cr-Mo alloy. The images drastically changes with the scattering angle ranging from less than 9 mrad (Fig.1 (a) bright field; BF) to up to 200 mrad (Fig.1 (d) high-angle annular dark field; HAADF). Cellular structure which consists of cells with the diameter of about 500 nm and interface regions with a network structure is clearly seen in the HAADF image (Fig.1 (d)). The interface regions contain precipitates as shown in Fig.1 (a) – (c). Takaichi et al. [2] reported Co-Cr-Mo alloys fabricated by SLM had cellular structures consisting of columnar units along the manufacturing direction. One can understand that the

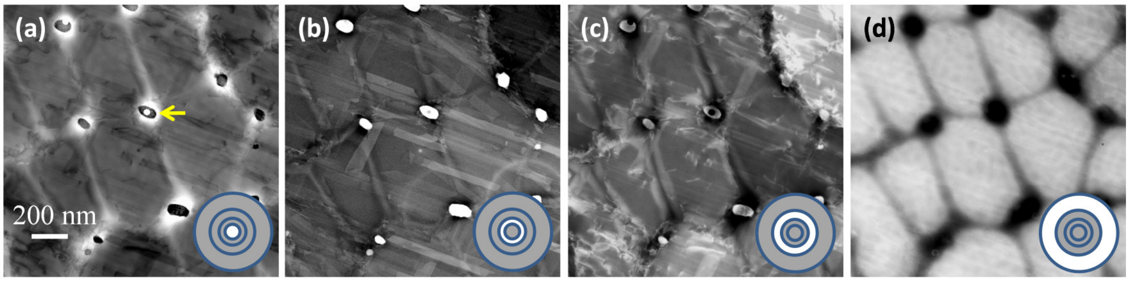

Fig. 1 STEM images for a Co-Cr-Mo alloy fabricated by SLM: (a) bright field (BF) image, (b) annular dark field image with a lower scattering angle range, (c) annular dark field image with a higher scattering angle range, (d) high angle annular dark field (HAADF) image. Schematic drawing showing STEM detector segments are inserted in each figure (Bright segments are active.).

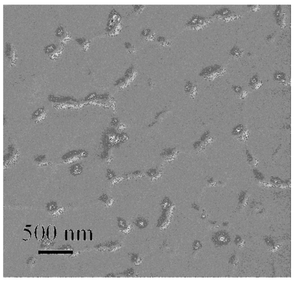

Fig. 2 SEM image for the surface of electro- polished Co-Cr-Mo thin film used for STEM experiments (1 keV, a chamber detector)

cellular structure in Fig.1 corresponds to a cross section of the columns. The interface regions show dark contrast in the HAADF image, suggesting that the average atomic number is smaller than that interior the cells. However this scenario is incorrect. Figure 2 is a SEM image for the surface of a thin film used for the STEM experiments. The precipitates and the interfaces were etched deeper than the cell matrix by the electro-polishing. The dark contrasts in the HAADF image is resulted from the thinner thickness not from smaller average atomic number.

Remarkable contrasts are seen in Fig.1 (b) and (c) obtained by STEM with the medium scattering angles between the BF and HAADF conditions. These images are sensitive to atomic-level crystal arrangements such as defects and dislocations [4]. Linear stripes with bright contrasts in Fig. 1(b) are stacking faults (SFs). Defects introduced around the cell interfaces and dislocations are clearly seen in Fig.1 (c). The brightest dotted contrast is found for the precipitates in Fig.1 (b). The origin of the bright contrast would be the complex crystal structures and/or defects in the precipitates. Our results indicate that adjusting the scattering angle is of important for effective extraction of specific information from the microstructure of the alloys fabricated by SLM.

It is reasonable to think that these SFs and defects are introduced during rapid solidification in SLM process. The defects around the interfaces would be introduced to reduce local distortions such as lattice mismatch between adjacent cells. Some SFs across the cell interfaces, which means that crystal orientations between the adjacent cells are almost identical. This suggests that the SFs were introduced to reduce distortions in wider area over the cells. The knowledges about atomic-level defects certainly contribute to understanding the formation mechanisms and mechanical properties of the alloys fabricated by SLM.

4. Summary

ADF-STEM images obtained with scattering-angle selection highlighted defects around the interface as well as SFs and dislocations in a Co-Cr-Mo alloy fabricated by SLM. These defects are important keys for understanding formation mechanism of the complex microstructure. SEM image showed a nonuniform etching during electro-polishing. The thickness distribution of thin film should be carefully considered for proper interpretation of the STEM images.

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