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静電浮遊させた Ti6Al4V のヘテロ凝固核 TiC による結晶粒微細化効果

Grain refinement effect of heterogeneous nucleation site particle TiC on Ti6Al4V levitated by Electrostatic Levitation (ESL)

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1. Introduction

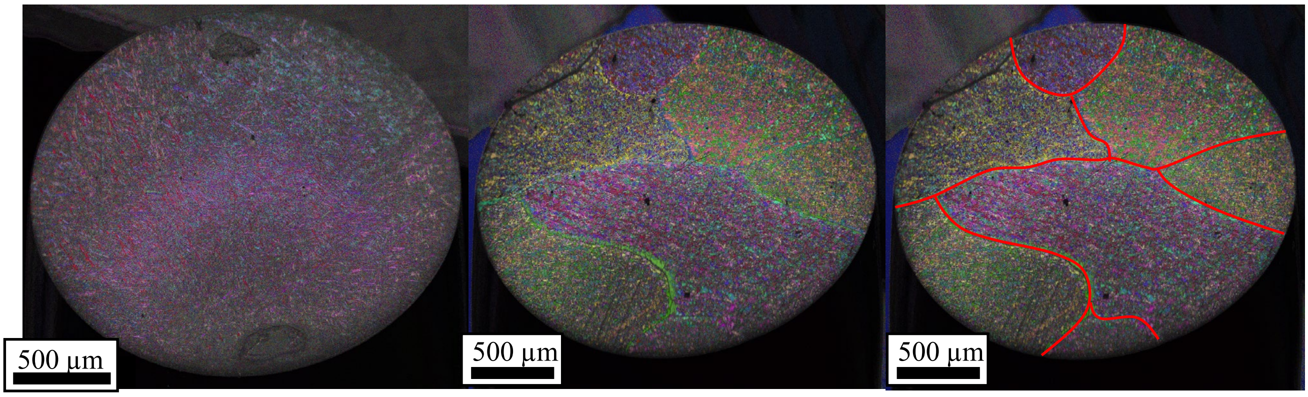
For Ti6Al4V powder during additive manufacturing, adding TiC has been proposed as heterogeneous nucleation site particles, which results in fine-equiaxed microstructure[1)]. In order to reveal the mechanism of this phenomenon, our research group is planning a space mission *Hetero-3D* to conduct experiments with Electrostatic Levitation Furnace on International Space Station (ISS-ELF), which can eliminate factors that affect nucleation caused by other than TiC. However, ISS-ELF experiment has limited opportunity. In this study, as a preliminary experiment for ISS-ELF, we aimed to reveal refinement effect of Ti6Al4V with TiC from undercooling and the number of grains in the cross section of Ti6Al4V samples with and without TiC after melting. Since samples were melted in high vacuum in Electrostatic Levitation (ESL), we also aimed to reveal the effect of this environment.

2. Experimental Procedure

First, Ti6Al4V powder without and with 1 vol.% TiC particles of 2-5 μm in diameter were sintered by Spark Plasma Sintering (SPS) method at 1273 K. After that, these sintered rod were cut into pieces with a mass of about 30 mg so that they could be levitated in ESL. Then, these pieces were melted in an arc furnace for less than 1 s to form spheres, which were prepared as samples. Each sample was heated in ESL under high vacuum. After confirming that the sample was completely melted, heating lasers were turned off immediately. After the values in the cooling curves obtained with the radiation thermometers were adjusted to fit melting point to 1923 K, the undercooling was calculated. The collected samples were polished to hemisphere and their composition was analyzed with Electron Probe Micro Analyzer (EPMA). Then, Electron Backscatter Diffraction (EBSD) measurements were conducted on the cross section to obtain the Inverse Pole Figure (IPF) images, and the number of grains on the cross sections were counted after visually drawing the prior-β grain boundaries on them. In EBSD measurement, the samples were tilted at 70°, and then projection transformation was conducted for the same angle. However, since the cross section was visually leveled, the scale may cannot be applied in the vertical direction.

3. Results

Figure 1(a) and (b) show the IPF images of the samples without and with 1 vol.% TiC, and **(c)** show the prior-β boundaries drawing on **(b)**.



(a)The sample without TiC (b)The sample with 1 vol.% TiC (c)After drawing prior-β boundaries on (b)

Fig. 1 IPF images on the cross section of the samples melted in ESL.

For the sample without TiC, there was mainly one specific crystal plane orientation shown by purple in **Fig.(a)**, though the other orientations shown by blue or yellow were also observed entirely. On the other hand, for the sample with TiC, the cross section was divided into some areas which were consisted of definitely different orientations shown in **Fig.(b)**, and these areas had various orientations as well as the sample without TiC.

The undercooling was 272 K for the sample without TiC and 265 K for the sample with 1 vol.% TiC. The concentration of Al in the sample without TiC was 0.23 mass%, while that in the sample with 1vol.% TiC was 0.21 mass%.

4. Discussion

When it is defined *α*' phase with uniform crystal plane orientations are precipitated from the one prior-β grain during cooling, seven prior-β grains were observed on the cross section shown in **Fig. 1(c)**. On the other hand, since no similar boundaries were observed on the cross section in **Fig. 1(a)**, the entire cross section is considered one large prior-β grain.

The undercooling was decreased by 7 K by adding 1 vol.% TiC compared to the sample without TiC. In theory, adding TiC decreases energy barrier for nucleation and promotes heterogeneous nucleation, which means undercooling required as a driving force becomes smaller. Therefore, the result in this study is reasonable.

From the composition analysis, it was found that the Al in Ti6Al4V almost evaporated and the composition changed regardless of TiC addition. Therefore, ISS-ELF experiments, which can suppress the sample evaporation by melting under Ar atmosphere, are necessary to reveal the effect of TiC addition to Ti6Al4V more strictly.

5. Conclusion

It was revealed that adding TiC to Ti6Al4V tended to refine the prior-β grains and decrease the undercooling of Ti6Al4V melted in ESL by 7 K. Since Al in the Ti6Al4V sample is almost completely evaporated, ISS-ELF experiments are necessary to suppress it.

References

1) Y. Watanabe, M. Sato, T. Chiba, H. Sato, N. Sato, S. Nakano, S. Suzuki, Journal of Japan Laser Processing Society, **26**(2019)46.