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ISS-ELF で溶融凝固させた TiC 添加 Ti-6Al-4V 試料表面解析

Surface Analysis of Ti-6Al-4V with TiC Melted and Solidified in the ISS-ELF

上野遥か [1]*, 門井洸衛 [1], 櫛舎祐太 [1], 市川文彩 [1], 上田雄翔 [1], 山田素子 [2], 佐藤尚 [2], 渡辺義見 [2], 小林正和 [3], 鈴木進補 [1]

Haruka UENO[1], Koei KADOI[1], Yuta KUSHIYA[1], Aya ICHIKAWA[1], Yuto UEDA[1], Motoko YAMADA[2], Hisashi SATO[2], Yoshimi WATANABE[2], Masakazu KOBAYASHI[3] and Shinsuke SUZUKI[1]

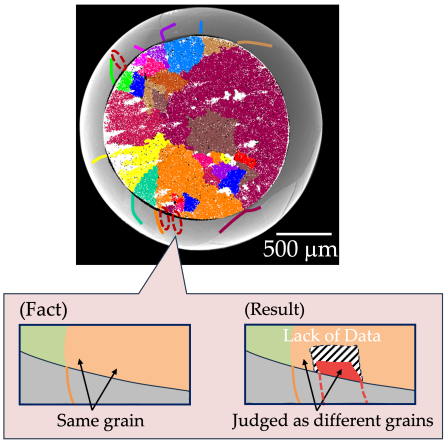
[1] 早稲田大学, Waseda University,

[2] 名古屋工業大学, Nagoya Institute of Technology,

[3] 豊橋技術科学大学, Toyohashi University of Technology

* Correspondence: ueno7712517@akane.waseda.jp

Abstract: Ti-6Al-4V with 5 mass% TiC heterogeneous nucleation site particles was melted and solidified using the electrostatic levitation furnace in the International Space Station. Multiple steps were confirmed at the prior-β grain boundaries on the surface of the sample. Therefore, the objective of this study was to clarify the relationship between the steps on the sample surface and the prior-β grain boundaries. Electron backscatter diffraction was applied to cross section of the sample to obtain the prior-β grain map. Subsequently, the misorientation between the adjacent prior-β grains was calculated. By overlapping the prior-β grain map on the scanning electron microscopy image of the sample cross section, the misorientations of the grain boundaries that could not be confirmed to correspond steps were found to be 1° and 179°. Considering symmetry of a body-centered cubic structure of the β phase, the actual misorientation is less than 1°. If there were no lack of data, the grains without steps at their grain boundaries would be judged as the same grain. Therefore, it was clarified that all steps and prior-β grain boundaries corresponded one-to-one.



Keywords: ISS-ELF, Prior-β grain boundaries, Misorientation, TiC, Heterogeneous nucleation, Ti-6Al-4V, *Hetero-3D*

1. Introduction

Ti-6Al-4V with TiC heterogeneous nucleation site particles was melted and solidified using the electrostatic levitation furnace in the International Space Station (ISS-ELF), which can eliminate nucleation factors from the container and minimize the effects of convection and temperature gradients within the sample[1)]. Multiple steps were confirmed at the prior-β grain boundaries on the surface of the sample[2)]. The objective of this study was to clarify the relationship between the steps on the sample surface and the prior-β grain boundaries.

2. Experimental Procedures

A spherical sample of Ti-6Al-4V with 5 mass% TiC with a diameter of 2.26 mm, that was melted and solidified in the ISS-ELF in 2023 was used. Details are shown in our previous study[3]. After the experiment, the sample was embedded in resin, cut using a diamond wire saw at 0.31 mm from the top of the sample, and polished using a polishing machine. After removing the resin by dipping it in acetone, the distribution of steps on the sample surface was obtained using the scanning electron microscopy (SEM). Subsequently, the electron backscatter diffraction (EBSD) analysis and reconstruction was applied to cross section of the sample to obtain the prior-β grain map. The step size of the EBSD measurements was set to 2 μm. In addition, the area within 5° of misorientation was defined as the same grain[2].

The misorientation between the adjacent prior-β grains was calculated based on the method by Chou *et al.*[4] as follows. First, the Euler angles (ϕ_1, Φ, ϕ_2), which are coordinate transformation variables indicating the crystal orientation relationship, were calculated from the Miller indices of the plane ($h\ k\ l$) and crystal orientations [$u\ v\ w$], respectively, corresponding to the prior-β grains. Then, for all pairs of the adjacent prior-β grains, the orientation tensors A_1 and A_2 were calculated from their respective Euler angles, and the transformation tensor G was obtained using the following equation.

$$G = A_2 A_1^{-1} \tag{1}$$

Subsequently, the rotation angle ω as the misorientation between the adjacent prior-β grains was calculated using the diagonal components g_{11}, g_{22}, and g_{33} of G using the following equation.

$$\omega = cos^{-1}\left(\frac{g_{11} + g_{22} + g_{33} - 1}{2}\right) \tag{2}$$

3. Results

Figure 1 (a) shows an overlapped image of the sample cross section obtained by SEM and the prior-β grain map obtained by EBSD analysis. Then, each prior-β grain boundary connected to the sample surface was assigned a grain boundary number (No.). Steps were observed in all prior-β grain boundaries except for grain boundaries No. 4, 5, and 10.

Figure 1 (b) shows the misorientation distribution in each prior-β grain boundary. The misorientations of grain boundaries where the steps could not be confirmed were 1° for No. 4 and No. 5, and 179° for No. 10.

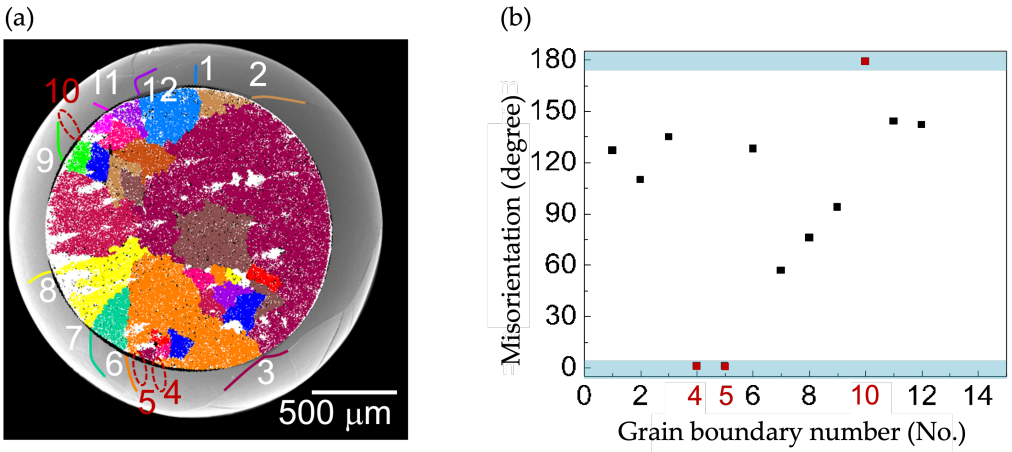


Figure 1. Relationship between prior-β grain boundaries and steps. (a) SEM image of the sample surface and cross section overlapped with the prior-β grain map. The steps were traced with lines for clarification. Each prior-β grain boundary was assigned a grain boundary number (No.). (b) Distribution of misorientation in the prior-β grain boundaries. To clarify, grain boundaries No. 4, 5, and 10, which could not be confirmed to correspond to steps, were plotted in red. The areas where the misorientations are within 5° and grains are judged to be the same are shown in light blue.

4. Discussion

A lack of data was found at grain boundaries No. 4, 5, and 10 shown in **Fig. 1(a)**. Although steps were not confirmed on the sample surface at these grain boundaries, the corresponding misorientations were 1° and 179°. Considering symmetry of a Body-Centered Cubic structure of the β phase, the actual misorientation is less than 1°. Since the misorientation is within 5°, this difference is judged to be the same grain if there is lack of data. Thus, these grain boundaries are not actual grain boundaries but same grains that were judged as different grains due to the lack of data. Therefore, it can be assumed that all steps and prior-β grain boundaries correspond one-to-one in the cross section of this sample.

5. Conclusion

The misorientation of the prior-β grain boundaries, where no steps were generated, was within 5° on the surface of the ISS-ELF sample. If there were no lack of data, the grains without steps at their grain boundaries would be judged as the same grain. Therefore, it was clarified that all steps and prior-β grain boundaries corresponded one-to-one. This enabled the number of areas surrounded by step lines to be counted as the number of prior-β grains on the sample surface.

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Conflicts of Interest

The authors declare no conflict of interest.

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