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静電浮遊炉で溶融凝固した Ti-6Al-4V に添加した TiC ヘテロ凝固核の解析

Analysis of TiC Heterogeneous Nucleation Site Particles Added to Ti-6Al-4V Solidified in the Electrostatic Levitation Furnace

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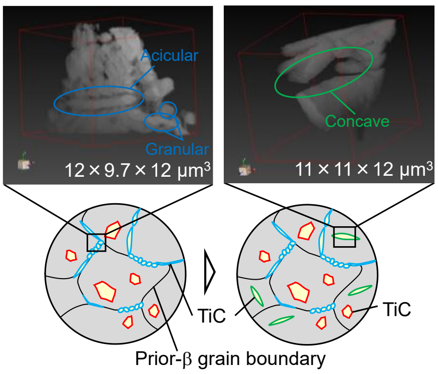
Abstract: Adding TiC heterogeneous nucleation site particles to Ti-6Al-4V results in forming refine grains. It is necessary to clarify the characteristics of TiC that contribute to the refinement. Ti-6Al-4V with 5 mass% TiC, solidified in the electrostatic levitation furnace, has been reported to contain residual TiC and precipitated TiC; however, previous evaluations have been limited to the morphology and composition observed in a cross section. Therefore, the objective of this study was to classify TiC based on there three-dimensional morphology and location toward elucidating TiC that contribute to nucleation. To obtain their three-dimensional morphology, TiC located either within prior-β grains or at grain boundaries were subjected to serial sectioning using a focused ion beam and visualized by scanning electron microscopy. TiC in the prior-β grain was a concave/convex morphology, whereas TiC at the prior-β grain boundaries was acicular. The concave/convex TiC within a prior-β grain interior was smaller than an α grain, suggesting possibility of incorporation into α grains. The acicular TiC at prior-β grain boundaries is considered morphologically similar to acicular precipitates observed in overheated samples.



Keywords: Precipitate, Prior-β grain boundary, Heterogeneous nucleation site particles, *Hetero-3D*, ESL

1. Introduction

Watanabe *et al*. reported that adding TiC heterogeneous nucleation site particles to Ti-6Al-4V results in forming equiaxed grain structure and improved tensile strength[1)]. On the other hand, not all TiC particles added to the sample act as nucleation sites[2)]. Therefore, it is essential to clarify the characteristics of TiC that are effective in nucleation. To this end, an experiment using an electrostatic levitation furnace (ESL) was conducted to investigate the role of TiC as nucleation sites. Mabuchi *et al*. reported that residual and precipitated TiC exist in the cross section of samples after the ESL experiment[2)], but their evaluation was limited to the morphology and composition observed in the cross section, and no clear definitions were provided. The objective of this study was to classify TiC based on its three-dimensional morphology and location, in order to elucidate the role of TiC in nucleation.

2. Experimental Procedures

A powder mixture of Ti-6Al-4V with 5 mass% TiC (nominal particle size < 20 μm) was sintered using spark plasma sintering machine and cut into cubes weighing approximately 30 mg. The cube was pre-melted in an arc melting furnace to stabilize levitation. The sample was then levitated, melted and solidified by laser beams in the ESL. After the ESL experiment, the sample was embedded in resin and mechanically polished to observe the cross section. Electron back scattered diffraction (EBSD) was performed to obtain α grain and prior-β grain maps. TiC identified within grains and at grain boundaries in the prior-β grain map were subjected to the serial sectioning in a 10 × 10 μm area using focused ion beam- scanning electron microscopy (FIB-SEM) milling with Ga ions. To obtain their three-dimensional morphology, this polishing process and scanning electron microscopy (SEM) imaging were conducted alternatively at every 100 nm.

3. Results

The α and prior-β grain maps of the cross section of the sample obtained by EBSD are shown in **Figs. 1(a)** and **(b)**, respectively. In addition, the three-dimensional reconstruction of TiC obtained by the serial sectioning within the prior-β grain interior and at the grain boundary are shown in **Figs. 1(c)** and **(d)**, respectively.

Within the prior-β grain interior, concave/convex TiC with sizes of approximately 10 μm was observed. At the prior-β grain boundaries, two kinds of morphologies of TiC were identified: acicular TiC approximately 10 μm in length and granular TiC approximately 1 μm in diameter. The α grain surrounding the concave/convex TiC within the prior-β grain interior measured approximately 20 μm. At the prior-β grain boundaries, multiple elongated TiC were observed intersecting the grains each other.

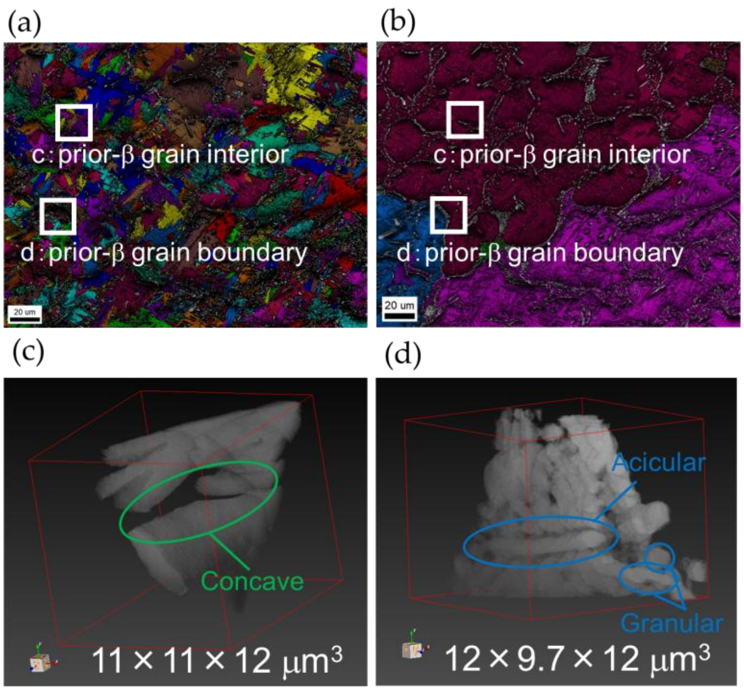


Figure 1. Three-dimensional morphology of TiC inside and boundary of prior-β grains. (a) α grain map on the cross section of the sample. (b) Prior-β grain map on the cross section of the sample. (c) Perspective view of TiC observed within the prior-β grain interior. The size of the rectangular prism enclosed by the red frame is 11 × 11 × 12 μm^3. (d) Perspective view of TiC at the prior-β grain boundary. The size of the rectangular prism enclosed the red frame is 12 × 9.7 × 12 μm^3.

4. Discussion

The TiC within the prior-β grain interior exhibited concave/convex/ morphology and was entirely enclosed by an α grain. This results suggested the incorporation into α grains during solidification. The TiC at the prior-β grain boundaries exhibited both acicular and granular morphologies. Because the diameter of the granular TiC was comparable to the short-axis diameter of the acicular TiC, the granular morphology can be attributed to fragmentation of the acicular TiC. Furthermore, the acicular TiC at prior-β grain boundaries were arranged in an intersecting configuration, such as TiC precipitates observed in the cross section of overheated samples[2)].

5. Conclusions

TiC were characterized as concave/convex, multiple intersecting acicular and granular morphologies. The concave/convex TiC of 10 μm located within the interior of the prior-β grain indicates it was incorporated into α grains with 20 μm. The acicular TiC located at the prior-β grain boundaries can be the same as ones found in overheated samples. The granular TiC of 1 μm located at the boundaries can be a fragment of the acicular TiC of the short-axis diameter of 1 μm.

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Conflicts of Interest

The authors declare no conflict of interest.

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