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*Hetero-3D*の静電浮遊実験における加熱条件が Ti6Al4V 中のヘテロ凝固核 TiC に及ぼす影響

The effect of heating conditions in electrostatic levitation experiments in *Hetero-3D* on TiC heterogeneous nucleation site particles in Ti6Al4V

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1. Introduction

Our space mission *Hetero-3D* is currently planning solidification experiments in the Electrostatic Levitation Furnace on the International Space Station (ISS-ELF) to confirm the grain refinement effect from TiC heterogeneous nucleation site particles added in Ti6Al4V. In the experiments, it is important to prevent TiC from dissolving during sample heating to remain as *Active-TiC*, which can serve as heterogeneous nucleation sites. Therefore, we hypothesized that the number of *Active-TiC* would increase by heating the sample for as brief a period of time and at as low a temperature as possible. We focused on the shape of precipitates formed by dissolved TiC to assess the amount of remaining TiC in the matrix and the effects of the heating conditions in the Electrostatic Levitation Furnace (ESL) on the ground. The goal of this study was to identify this condition under which there is enough *Active-TiC*.

2. Experimental Procedure

Ti6Al4V powder and TiC (particle size < 20μ m) of 5 vol.% were mixed homogeneously. These samples were sintered by Spark Plasma Sintering (SPS) method and solidified into spherical shapes by using surface tension in the plasma arc melting furnace. Then, each sample was heated in the ESL. The maximum temperature measured by a pyrometer and the heating time at which the sample melted entirely were varied from about 1950 K to 2050 K and from 2 s to 5 s, respectively. A parameter "heating integral value" was introduced, which was calculated by integrating the heating time and temperature. The calculation range was from the sample

first reached the melting point of Ti6Al4V, 1923 K, until it fell below 1923 K after the lasers were turned off. For observations of microstructures, samples were embedded in resin and polished. First, prior- β grain maps were obtained by reconstructed crystal orientation data of the samples measured by Electron Backscatter Diffraction (EBSD) measurements. Next, images of microstructures were observed with Scanning Electron Microscope (SEM).

3. Results

Figure 1 shows needle-shaped precipitates and unrefined grains in the sample in which the heating integral value was above 60,000 Ks. However, when it was less than 60,000 Ks, three types of precipitates were observed: massive (A), ring-shaped (B), and tiny needle-shaped (C). Furthermore, the prior- β grains in the sample were refined.



Figure 1. Changes of precipitates' shapes and prior- β grains

4. Discussion

When the heating integral value was above 60,000 Ks, it was estimated that most TiC was dissolved, failing grain refinement. The dissolved TiC was thought to become needle-shaped precipitates by being precipitated between the martensite matrix α ' phase during solidification.

In contrast, when it was less than 60,000 Ks, (A) is thought to be remaining TiC from its size and shape, and some of (A) caused grain refining as *Active-TiC*. Therefore, evaluating the amount of (A) could be used to estimate the amount of *Active-TiC*. Due to grain refining, a part of dissolved TiC was thought to have been ring-shaped (B) by precipitating from the grain boundary around α' or prior– β phases preferentially. At the same time, the other dissolved TiC was precipitated between the α' phase and produce tiny needle-shaped (C).

5. Conclusion

When the heating integral value is controlled to be less than 60,000 Ks during experiments in ESL, some of TiC (particle size $< 20 \ \mu$ m) of 5 vol.% added in Ti6Al4V remains in the matrix and some functions as heterogeneous nucleation site particles.

