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# 浮遊法による TiC 添加 Ti6Al4V 合金の凝固組織微細化に及ぼす対流の影響

# Effect of Convection on Microstructure Refinement in Ti6Al4V Alloys with Additive TiC Solidified via Levitation Techniques

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## 1. Introduction

Additive manufacturing (AM) is attracting attention for achieving in-space and in-orbit manufacturing due to its ability to produce intricate three-dimensional products near net shape, without joints. However, the solidification conditions in AM, which include, unidirectional solidification, repeated laser heating, and substantial temperature gradients, often induce a formation of large columnar microstructure, resulting in products with anisotropic strength of the products. For Ti6Al4V alloy, a leading material for aerospace components, the addition of TiC particles serves as a heterogeneous nucleation site, promoting the formation of fine equiaxed grains. This effectively inhibits large columnar structures during the solidification via AM. However, the grain refinement mechanism using TiC on the solidified microstructure of Ti6Al4V alloys has not yet been well clarified due to the presence of container wall and gravitational convection in the terrestrial experiments. Therefore, our group is conducting research on the solidification of Ti6Al4V alloys with added TiC, using levitation techniques involving a space experiment with the electrostatic levitation furnace in the International Space Station (ISS-ELF), called Hetero-3D mission.

In the present work, we examined the microstructural differences in Ti6Al4V alloys solidified under container-free conditions using electrostatic levitation (ESL) with minimal convection, and electromagnetic levitation (EML)[1] with large convection due to electromagnetic agitation on the ground. The aim of this study was to examine the influence of melt convection during solidification on the microstructure of Ti6Al4V alloys as well as the effect of added TiC, as preliminary ground work for the Hetero-3D mission.

## 2. Experiment procedure

Gas-atomized Ti6Al4V powders, with nominal diameters of less than 45 μm, were thoroughly mixed with 5 mass % TiC powders, with nominal diameters less than 20 μm, using a three-dimensional motion mixer. This mixture was then sintered into a cylindrical compact, with 20 mm in diameter and 10 mm in thickness, using spark plasma sintering (SPS). The sintered sample was cut into cubes, with sides of 2 mm for containerless solidification experiments using ESL and 6 mm for those using EML.

For the ESL experiment, a cube cut from the sintered compact was melted and solidified into a nearly spherical shape using an arc furnace, under a high-purity argon gas atmosphere. The spherical sample was electrostatically levitated and subsequently melted by irradiating it with $CO_2$ laser beams from three directions. Upon shutting off the laser, the sample was cooled and solidified under a containerless state. The temperature history of the sample was monitored by monochromatic pyrometers. Details of the ESL facility and experiments can be found elsewhere[2].

In the EML experiment, a piece of the cube sample was placed on a quartz holder and positioned in the center of the EML coil. The sample was then electromagnetically levitated and then inductively melted under high purity argon gas flowing at 2 L/min. To enhance the sample heating, irradiation from a semiconductor laser beam was superimposed on the levitated droplet. After turning off the semiconductor laser heating, the levitated droplet was cooled and solidified by blowing high-purity helium gas over it. The cross-sectional microstructures of the solidified samples obtained from both the ESL and EML experiments were examined using a scanning electron microscopy (SEM) equipped with an electron backscattered diffraction detector (EBSD).

## 3. Results and Discussion

**Figure 1** shows typical cooling curves of the levitated sample solidified by using ESL and EML. When the heating time of the levitated melt was prolonged or its maximum temperature was high, the melt experienced a large undercooling followed by recalescence, as shown in (a) and (c), regardless of the levitation techniques used. It has been reported that TiC dissolves in the molten Ti6Al4V at elevated temperatures. As a result, the added TiC particles would dissolve before solidification and would serve as heterogeneous nucleation sites in these samples.

Conversely, when the heating duration of the sample was minimized and was immediately cooled after melting, solidification began at the equilibrium liquidus temperature, regardless of whether the experiment was conducted using ESL or EML, as indicated in **Figure 1** (b) and (d). This confirms that excessive heating causes the TiC particles to dissolve into the molten Ti6Al4V. For the samples subjected to minimal heating, the release of latent heat during solidification led to a lower cooling rate post nucleation at the equilibrium liquidus temperature.

**Figure 2** exhibits the typical microstructures of the solidified samples, corresponding to the cooling curves of (a) to (d) of **Figure 1** The microstructures of the samples that experienced a large undercooling before solidification consisted of relatively large grains as displayed in (a) and (c). In contrast, the microstructures of the samples that nucleated at the equilibrium liquidus temperature presented smaller grains, as observed in (b) and (d). This result confirms that the TiC particles acted as a heterogeneous nucleation site.

A higher cooling rate of the sample usually results in small grains in the solidified microstructure[3-5]. The cooling rate of the levitated droplet was slower in the EML than in the ESL, because the electromagnetically levitated sample inevitable undergoes inductive heating, even when it is forcibly cooled by blowing helium gas. However, the grain size of the sample solidified by the EML was smaller than that of the sample solidified by the ESL. Strong electromagnetic stirring of the melt in the EML technique may cause fragmentation or separation of the crystal growing on the melt surface[6-8]. In addition, the larger number of TiC particles contained in the sample for the EML experiment may promote nucleation. Although the sample composition is the same for both the ESL and EML experiments, the number of TiC particles in the sample is greater in the EML experiment than in the ESL experiment due to the larger sample size. It is difficult to distinguish the effects of cooling rate, convection and number of TiC particles on the solidified microstructure as long as we use the ESL and EML on the ground. The results of the Hetero-3D project under microgravity conditions on the ISS will provide further insight into the grain refinement effects of added TiC on solidified Ti6Al4V.

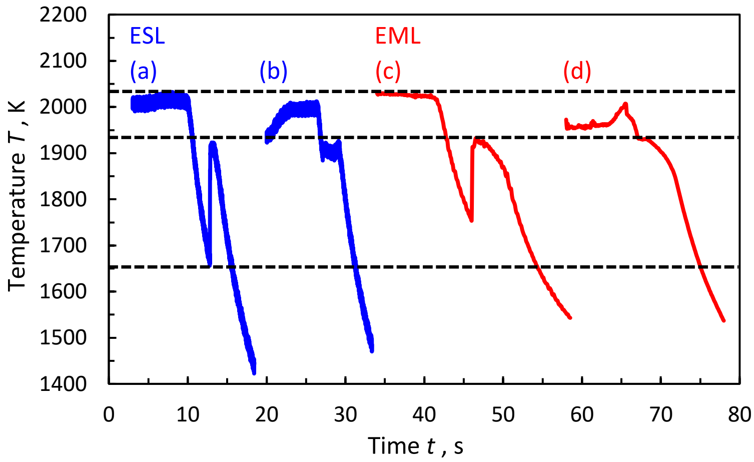


**Figure 1.** Typical cooling curves of levitated molten samples by ESL and EML

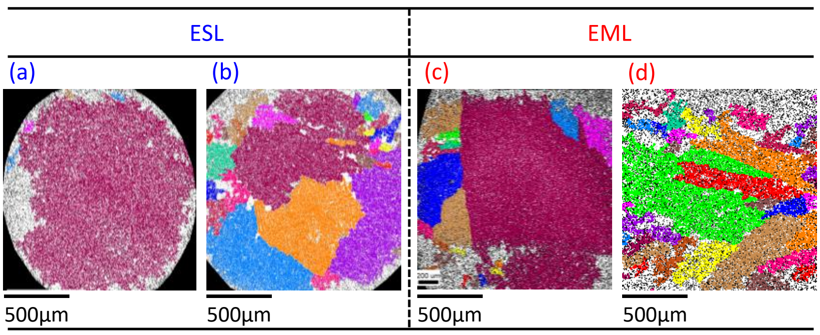


**Figure 2**. Typical microstructures of solidified samples by ESL and EML

## 4. Summary

Ti6Al4V samples with 5 mass% TiC, prepared from their respective powders by SPS, were melted and solidified under levitated state using ESL and EML. Due to prolonged heating and reaching higher maximum temperatures, TiC particles dissolved in the melt. As a result, the molten sample undercooled deeply prior to solidification. The microstructures of the solidified samples exhibited relatively large grains. In contrast, when the sample was subjected to minimal heating and was immediately cooled after melting, nucleation occurred at the equilibrium liquidus temperature, due to heterogeneous nucleation from residual TiC particles. This resulted in a microstructure with smaller grains in the solidified sample. Even though the cooling rate at post nucleation in the sample solidified using EML is lower than that using ESL, the resulting grain size was smaller.

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