JASMAC

Conference of the Japan Society of Microgravity Appllication

OS2-8

# Hetero-3D ミッションにおいて ISS-ELF により溶融凝固させた Ti 合金試料の飛行後解析

# Postflight Analyses of Ti Alloy Samples Melted and Solidified by ISS-ELF in Hetero-3D Mission

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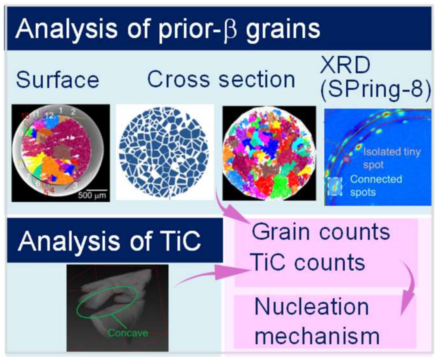


**Abstract:** The Hetero-3D mission aimed to clarify the effects of heterogeneous nucleation site particles on the solidification behavior of metals for 3D printing. Ti and Ti-6Al-4V samples, with and without 5 mass% TiC, were melted and solidified using the electrostatic levitation furnace on the International Space Station, which offers contamination-free, convection-free conditions. Postflight analyses focused on estimating the grain counts and understanding TiC behavior during solidification. A flight sample of Ti-6Al-4V with TiC was analyzed using electron backscatter diffraction, Voronoi tessellation, and X-ray diffraction. The estimated grain count was approximately 400 with good agreement between methods. Grain boundaries aligned well with surface steps. This finding is also effective for estimating the number of internal particles. Additionally, TiC particles were observed in different morphologies depending on their location: concave/convex inside grains and acicular at grain boundaries. These features help identify whether the TiC acted as nucleation sites or formed during or after solidification. This work provides insights into controlling microstructures in 3D printed metals and contributes to optimizing process parameters for grain refinement in additive manufacturing.

**Keywords:** International Space Station, electrostatic levitation, Hetero-3D, SPring-8, XRD, EBSD, Ti-6Al-4V, TiC, heterogeneous nucleation site

## 1. Introduction

Melting and solidification experiments were conducted as part of the mission of the JAXA's science experimental theme "Heterogeneous solidification behavior of powder metals for 3D printer" (Abbreviation: Hetero-3D, PI: S.Suzuki, Waseda University)[1-3] in April and May 2023. The objective of this mission is to clarify the effects of heterogeneous nucleation site particles on solidification behavior of metals for 3D printing. Samples of Ti and Ti-6Al-4V with and without heterogeneous nucleation site particles TiC were melted and solidified with electrostatic levitation furnace on International Space Station (ISS-ELF), which provides ideal conditions to exclude the effects of contamination from the container, evaporation and convection. The results obtained will be applied to improve the process conditions of 3D printer for refinement of crystal grains[4]. **Figure 1** shows the overview of Hetero-3D mission. and simulations, important results were obtained, such as, appropriate sample preparation methods[5], heating conditions[6], solidification behavior of the sample[7], melting behavior of TiC[8], recording conditions[9] and analysis method of movies[10] by high-speed camera by preflight experiments and telemetry data analyses, and temperature and velocity fields in liquid samples[11], nucleation[12] and crystal growth[13] during cooling by numerical simulations.

This presentation introduces the results obtained through postflight analyses of the samples, which returned to the ground in June 2023 [2]. The main objectives of the preflight analyses are estimation of the number of crystal gains and existing TiC just before solidification in the samples. Now we are attempting to estimate the grain counts from cross-sectional observation and Voronoi tessellation[14,15], X-ray diffraction (XRD) and characterization of TiC morphologies in the samples.

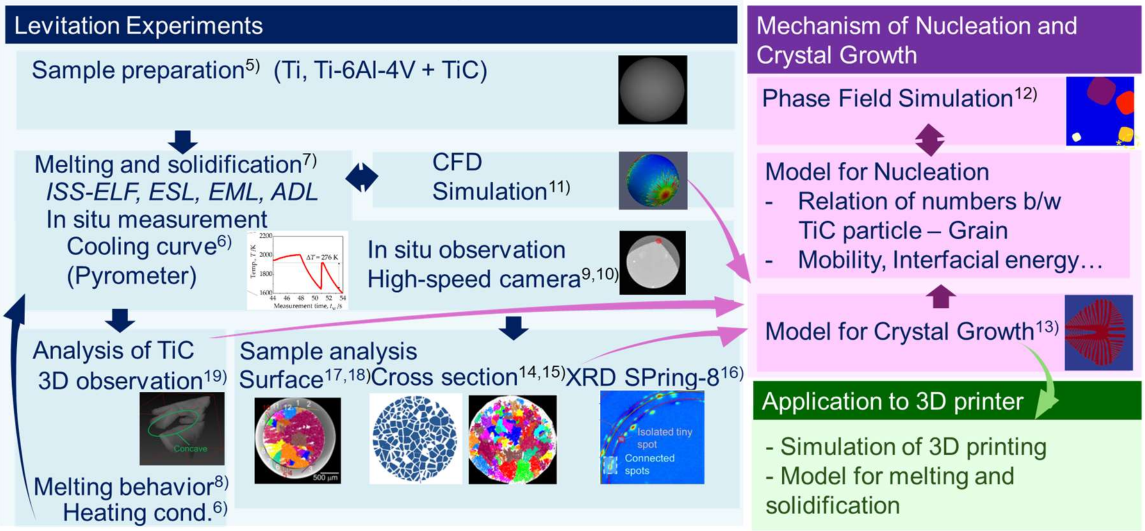


**Figure 1.** Overview of mission Hetero-3D.

## 2. Postflight Analysis

A flight sample of Ti-6Al-4V with 5 mass% TiC was analyzed by cross-sectional and surface observation and X-ray diffraction to estimate the grain count. Ti-6Al-4V solidifies with only the β phase crystallization. After solidification is complete, during the subsequent cooling process, when the temperature falls below the β transus temperature, the α phase precipitates. Therefore, a microstructure in which both α and β coexist can be observed at room temperature. This group examines methods to determine the number of crystallographic grains immediately after solidification when it was in a β single phase, which is called prior-β grains.

### 2.1. *Cross-sectional observation and Voronoi tessellation*[15]

The flight sample was cut and polished. The grain distribution on the cross section of the flight sample was obtained by electron backscatter diffraction. The grains were categorized into three groups according to grain area, because smaller grain has higher coefficient of variation, which means lower reliability.

The grain count in the flight sample was estimated from the count per unit volume, which was obtained using Voronoi tessellation for the three classified groups. As a result, the grain count in the ISS-ELF sample was estimated to be 365±68.

### 2.2. *Analysis of X-ray diffractometer by Spring-8*[16]

The flight sample was analyzed using XRD patterns obtained at SPring-8 BL20XU. The $\{10\bar{1}0\}$ ring of α phase was selected for analysis due to its high-intensity and non-overlapping with other rings. Connected spots across frames were grouped. The maximum projected area of each group was measured and converted to the sphere-equivalent volume. Then the number of β grains was estimated to be $4.0\times10^2$ by dividing the sample volume by the sphere-equivalent volume. This result agreed well with the grain count obtained by Voronoi tessellation, explained in *2.1*.

### *2.3. Analysis of steps on surface*[17)]

The prior-β grain map explained in 2.1 was overlapped on the scanning electron microscopy image of cross section of the flight sample. As a result, most of the lines of steps, which were already reported[18)], matched with the grain boundaries of prior-β grains. The misorientations of the grain boundaries that could not be confirmed to correspond steps were 1° and 179° (=-1°), which is considered small enough to be ignored. These results showed that all steps aligned well with the prior-β grain boundaries. Therefore, the number of regions surrounded by these lines of steps can be counted as the grain counts exposed on the sample surface.

## 3. Analysis of TiC[19)]

The added TiC partially dissolves during the heating process[8)], and only some of the remaining portion functions as nucleation sites. When observing the sample after solidification at room temperature, remaining TiC, crystalized TiC during solidification, and precipitated TiC from the solid phase can be found. To investigate the count of TiC particles that remained just before solidification, a method to distinguish which kind of TiC it is. A sample of Ti-6Al-4V with 5 mass% melted and solidified in electrostatic levitation furnace on the ground at JAXA was analyzed to obtain the three-dimensional morphologies. Two parts of TiC location was selected, that is, inside a prior-β grain and at a prior-β grain boundary. TiC in the prior-β grain had concave/convex shapes. On the other hand, TiC at the prior-β grain boundaries was acicular. These morphological features provide clues for considering the history of TiC.


## Acknowledgments

This study was conducted as the experiments for Hetero-3D mission, using the Japanese Experiment Module, "Kibo", on the ISS. The synchrotron radiation experiments were performed at the BL20XU at SPring-8 with the approval of JASRI (proposal nos. 2024B1125). This study was supported by Waseda University Special Research Project 2024C-70B and by JST SPRING, Grant Number JPMJSP2128.work. This work was the result of using research equipment (G1001, G1011, G1017, G1021, and G1033) shared in MEXT Project for promoting public utilization of advanced research infrastructure Grant Number JPMXS0440500025 with the help of the Joint Research Center for Environmentally Conscious Technologies in Materials Science, Kagami Memorial Research Institute for Materials Science and Technology, Waseda University. We would like to thank Kimura Foundry Co., Ltd. for their financial support.


## Conflicts of Interest

The authors declare no conflict of interest.

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