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# *Hetero-3D* 用 TiC 添加 Ti6Al4V 中の気泡発生を抑制する試料作製

# Preparation of samples to suppress bubble formation in Ti6Al4V with TiC for *Hetero-3D*

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## 1. Introduction

Space mission *Hetero-3D* has a plan to conduct experiments to melt and solidify Ti and Ti6Al4V with TiC in Electrostatic Levitation Furnace on the International Space Station (ISS-ELF) in order to clarify the grain refinement mechanism by TiC heterogeneous nucleation site particles. In the preliminary ground experiments, however, there were some instances in which bubbles were formed in the melted samples with TiC. These bubbles obstruct the understanding of the grain refinement mechanism. Our study aimed to reveal the process of bubble formation in the samples for the ISS-ELF experiments to decide the optimum preparation process.

## 2. Experimental Procedure

TiC particles were prepared in two ways. One was pulverized with a mortar and a pestle from larger TiC particles, and this is called mortar TiC. The other was pulverized with ball mill and crusher mill, and this is called BM+CM TiC. TiC particles with a particle size < 20 μm were then extracted with a sieve. The grain size distributions and Scanning Electron Microscope (SEM) images were obtained for each type of TiC particles.

After that, block samples were prepared for four types of Ti6Al4V particles with mortar TiC of 5 vol.% or BM+CM TiC of 0, 2, or 5 vol.% by Spark Plasma Sintering (SPS) method. Thermal Desorption Spectrometry (TDS) analysis in the room temperature to 1800 °C range, including the melting point of Ti6Al4V, which is 1650 °C, was conducted for the sintered sample with mortar TiC. The densities of three other sintered samples were determined by the Archimedes method, and the porosity for each sample was calculated to determine how the densities varied depending on the amount of TiC content. Additionally, all sintered samples with 5 vol.% TiC were solidified in a sphere shape by using surface tension in the arc furnace. In the Electrostatic Levitation Furnace (ESL) on the ground, samples melted and solidified in a short time. After that, the pore volumes in X-ray Computed Tomography (CT) images were calculated as the bubble volumes in the samples.

## 3. Results

The SEM images and the grain size distributions of TiC particles in **Figures 1(a)** and **(b)** show that the mortar TiC particles contained more angular ones like (A) and were smaller in mean diameter than BM+CM TiC. In the TDS analysis of the sintered sample with mortar TiC, CO gas was detected when the temperature was higher than 1650 °C. The porosities in the sintered samples with 0, 2, and 5 vol.% BM+CM TiC were 0.3, 0.5, and 0.7 % respectively, indicating that they rose in proportion to the amount of TiC.

The results of X-ray CT following ESL experiments in **Fig. 1(c)** show that there was a pore of approximately 0.3 mm$^2$ in the sample with mortar TiC, but none at all in the sample with BM+CM TiC.

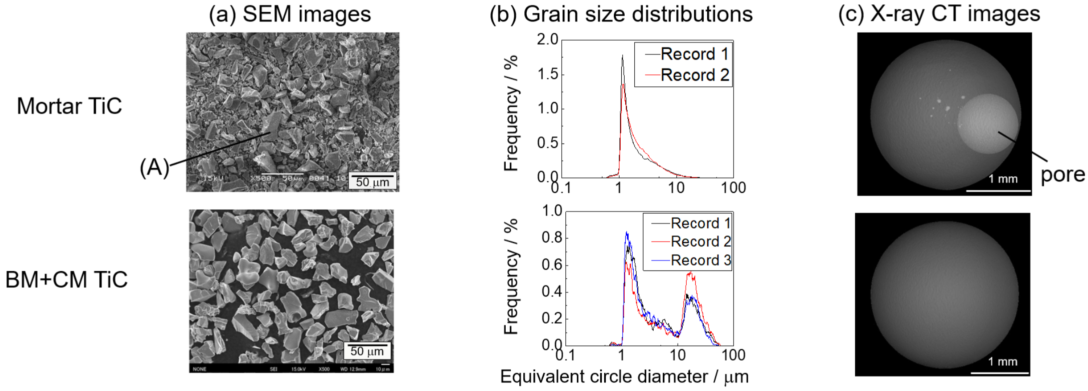


**Figure 1.** The differences between mortar TiC and BM+CM TiC:
(a) SEM images; (b) Grain size distributions; (c) X-ray CT images.

## 4. Discussion

The cause of these results can be attributed to the following mechanism. Since porosities increased as TiC content in the samples increased, TiC can be considered to induce pores between the particles in the samples in the SPS process. Then, $O_2$ in the pores and C in the TiC reacted, generating CO and appearing as bubbles during melting. Therefore, samples with higher TiC content and larger pores between the particles are more likely to form bubbles. When the percentage of TiC in the sample is 5 vol.%, the surface area of TiC with a smaller mean diameter is larger. Furthermore, angular TiC particles exacerbate fluidity and increase the porosities. Due to the larger surface area of TiC which can react with $O_2$ and the increased pores containing $O_2$, the sample with mortar TiC formed more bubbles than one with BM+CM TiC.

## 5. Conclusion

Preparing TiC with the BM+CM instead of the mortar was effective in suppressing bubble formation in the sample for the ISS-ELF experiments. The cause of bubble was the CO gas produced by C in TiC and $O_2$ in pores between particles while the samples of sintered Ti6Al4V and TiC particles melted. Based on that, the following two methods are important for the optimum preparation:

1) Reducing the surface area of TiC by raising the mean diameter to prevent C from reacting with $O_2$;
2) Improving the fluidity of TiC by removing angular TiC and reducing porosities in the sintered samples, as well as decreasing $O_2$ in the samples.