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TiC 添加 Ti 液滴の静電浮遊炉による地上実験と ISS 実験条件の最適化

Ground based experiments of TiC doped Ti droplets in the Electrostatic Levitation (ESL) and optimization of the experimental conditions on ISS

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1. Introduction

Addition of TiC particles to Ti powder to promote heterogeneous nucleation and fine equalization has been demonstrated during additive manufacturing[1]. The Electrostatic Levitation Furnace on the International Space Station (ISS-ELF) is an effective tool to measure the thermophysical properties in liquid and detailed solidification characteristics which are needed to simulate the solidification mechanism because of the elimination of crucible-derived nucleation and impurities. However, it is essential to optimize the experimental conditions on the ISS by conducting ground experiments because of the limited opportunities for space experiments. The objective of this study was optimization of the heating time of samples and the amount of TiC addition on the ISS. We identified the relationship between heating time during the acquisition of their properties and solidification microstructure and determine the amount of TiC addition which is the most effective to fine equalization of pure Ti microstructure. After melting and solidifying the Ti samples in the ESL, the microstructure of the samples was observed.

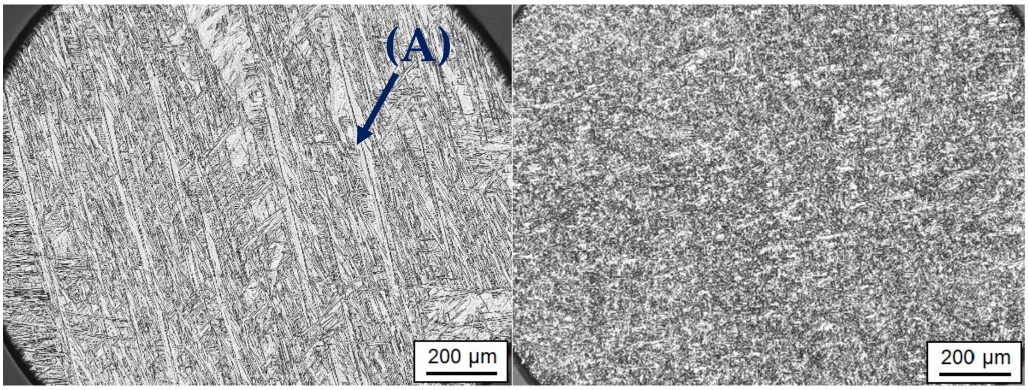
2. Experimental Procedure

The samples for levitation were prepared through sintering pure Ti powder without and with adding from 0.1 to 10.0 vol.% TiC particles of 2-5 μm in size, followed by machining to about 20 mg in weight. The samples were melted by heating with laser irradiation in the ESL in high vacuum. The maximum temperature was 1793 °C, and the heating time was varied from 140 sec. to 600 sec. The surface tension, density and viscosity of the molten sample were measured at various temperatures. The laser was turned off and cooling curve was obtained during natural cooling. And then, the solidified samples were collected. The samples were embedded in resin and polished, and their cross sections were observed with an optical microscope. The mapping of Ti, O and C elements were obtained by EPMA for quantitative analysis.

3. Results

Figures.1(a) and (b) show the microstructure of the samples with 0.1 and 1.0 vol.% TiC, respectively. In the 0.1 vol.% TiC sample (**Fig.1(a)**), as in the pure Ti (without TiC) sample, several elongated structures(A) of several-hundred μm in length aligned in parallel were observed. Similar structures were observed in the 0.2 vol.% and 0.3 vol.% TiC samples. In the 1.0 vol.% TiC sample (**Fig.1(b)**), this kind of elongated structures were not found, but fine needle-like microstructure was observed. The sample was refined equiaxially compared to pure Ti. At 2.0 vol.% addition, the sample was refined equiaxially with fine needle-like microstructures similar to in **Fig1(b)**, but the microstructure was coarser than that of the 1.0 vol.% sample. The 2.0 vol.% sample with a longer heating time (600 sec.) had structures of 50-100 μm in size aligned in L-shape. At 10.0 vol.%, which is an extremely high-level amount, the sample did not become spherical during the heating process, and TiC agglomeration was observed on the surface.

The thermophysical properties of pure Ti droplets were comparable to the reference data of previous ESL measurements[2)]. There was no significant difference between them and the TiC-added sample. However, the thermophysical properties could not be measured at 10.0 vol.%, because of remaining solid particles by TiC agglomeration.



(a)Sample with TiC0.1 vol.% (380 sec. heating)   (b)Sample with TiC1.0 vol.% (140 sec. heating)

Fig. 1 Microscopic images of solidified samples

4. Discussion

Anisotropic microstructure formed at 0.1-0.3 vol.% addition because TiC is considered to have been melted and dispersed into the base Ti during heating, and thus the amount of TiC acting as a hetero-solidification nucleus was reduced[3)]. On the other hand, the refinement at 1.0 vol.% is thought to be due to enough amount of TiC remaining as heterogeneous nucleation site particles. At 2.0 vol.% addition, C was detected in the refined needle-like structure at about 10-20 at.% by EPMA analysis, which was lower than that of TiC. This indicates that TiC was dispersed into the Ti during melting and precipitated out of the solid phase[3)]. The amount of TiC acting as heterogeneous nucleation site particles was reduced and the microstructure was coarsened compared to that of the 1.0 vol.% addition.

5. Conclusion

The microstructure of the sample melted and solidified in the electrostatic levitation furnace was observed, and it was found that the appropriate amount of TiC to act as heterogeneous nucleation site particles without surface segregation and agglomeration is about 1.0 vol.%, which is considered to be the optimal TiC-adding condition for obtaining the physical properties of the droplets in the ISS-ELF experiment.

References

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