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静電浮遊炉実験における Ti6Al4V 中のヘテロ凝固核 TiC の溶解挙動

Dissolution behavior of heterogeneous nuclei TiC in Ti6Al4V in Electrostatic Levitation Furnace

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1. Introduction

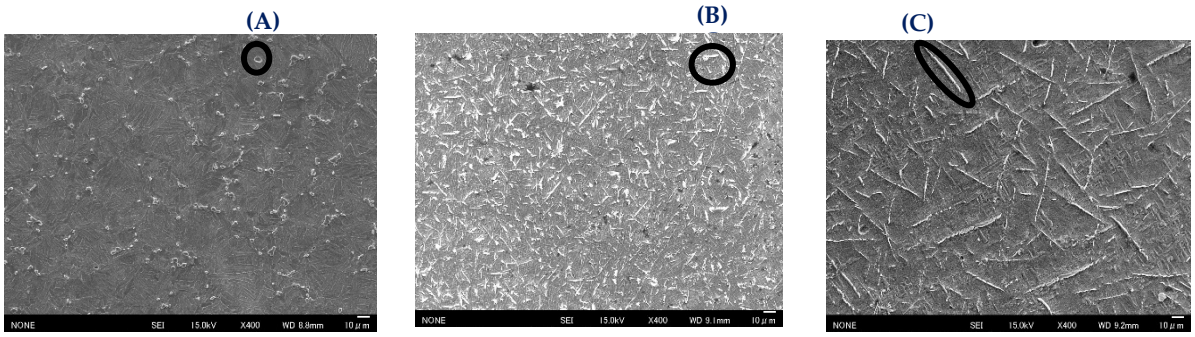
It has been reported that TiC addition to the matrix Ti6Al4V leads to grain refinement in additive manufacturing.[1)] However, it is difficult to focus on grain refinement effect because of many barriers to observe nucleation. Therefore, our research team is planning a space mission *Hetero-3D* to conduct melting experiments under micro-gravity environment with Electrostatic Levitation Furnace on the International Space Station (ISS-ELF) which can eliminate these effects. However, there are few opportunities to conduct experiments with ISS-ELF, therefore, in this study, we used the Electrostatic Levitation (ESL) in order to determine the experimental conditions for ISS-ELF. There is a possibility that added TiC melt during the sample preparation phase or ESL experiments. We aimed to reveal the dissolution behavior of added TiC in each phase.

2. Experimental Procedure

Ti6Al4V powder and 2 vol.% TiC particles of 2-5 μm in diameter were mixed homogeneously. Three kinds of samples, *SPS*, *ARC*, and *ESL* were prepared. Sample-*SPS* was sintered by SPS method and cut into approximately 30 mg to levitate in the ESL. After that, sample-*ARC* was solidified into spherical shapes with plasma arc melting furnace. After arc melting, sample-*ESL* was heated in the ESL, and cooled naturally soon after confirming that the entire sample was melted. Three samples were embedded in resin and polished, and their cross section were etched with Kroll solution for 30 s. Microstructures in the samples were observed with FE-SEM and the composition of sample-*ARC* was obtained with Energy Dispersive X-ray Spectrometer (EDS) and sample-*ESL* was obtained with Auger Electron Spectroscopy (AES). After that, the area ratio of precipitates originated in TiC in the matrix was calculated with an image processing software, and then the ratio of remaining TiC in the matrix was calculated.

3. Results

Figure 1 shows FE-SEM images of the sample-*SPS*, *ARC*, and *ESL*.



(a) Sample-*SPS* **(b)** Sample-*ARC* **(c)** Sample-*ESL*

Fig. 1 FE-SEM images on the cross sections of the samples

As shown in **Fig. 1 (a)**, microstructures appearing spherical in the range of 2-5 μm (**A**) were observed homogeneously after SPS method. As shown in **Fig. 1 (b)**, microstructures appearing white (**B**) could be observed, but the compositions were clarified to be Ti, Al and V without C after arc melting. As shown in **Fig. 1 (c)**, Ti-rich TiC, appearing long, thin, and white (**C**), were observed after the ESL melting. Similar microstructures were also observed in the additive manufacturing experiment.[2)] The composition of Ti-rich TiC was Ti : C ≈ 1 : 0.5. The area ratio of Ti-rich TiC was 2.8-3.2 $area\%$ through measurements in three points in the sample.

4. Discussion

The microstructures of sample-*SPS* (**A**) are considered to be added TiC from their sizes and shapes. The microstructures of sample-*ARC* (**B**) are also not including C. Therefore, TiC did not melt in the sample preparation phase.

By comparing the weight of C before and after ESL melting, the ratio of remaining TiC in the matrix were calculated. To simplify the calculation, we assumed that the sample volume was $V\ m^3$. First, the weight of C before the melting was calculated. Since the density of TiC (ρ_{TiC}) is 4930 kg/m^3 and the molecular weight of Ti (M_{Ti}) and C (M_C) are 47.88 and 12.01, the weight of C in TiC 2 vol.% is

$$(\rho_{TiC}V \times 2) \times \frac{M_C}{M_{Ti} + M_C}\ kg \tag{1}$$

Next, the weight of C after ESL melting was calculated. If the area ratio of Ti-rich TiC is x area% and Ti-rich TiC is assumed to be distributed homogeneously, the volume ratio equals the area ratio: x vol.%. From the density of TiC and the same crystal structures of between TiC and Ti-rich TiC, the density of Ti-rich TiC ($\rho_{Ti-rich\ TiC}$) is calculated to be 5280 kg/m^3. Therefore, Ti-rich TiC is $x\rho_{Ti-rich\ TiC}$V kg, and the amount of C contained in it is

$$x\rho_{Ti-rich\ TiC}V \times \frac{M_C}{2M_{Ti} + M_C}\ kg \tag{2}$$

From the calculation, the remaining ratio of TiC in the matrix is obtained to be

$$100\left\{1 - \frac{x\rho_{Ti-rich\ TiC}(M_{Ti} + M_C)}{2\rho_{TiC}(2M_{Ti} + M_C)}\right\}\% \tag{3}$$

In this study, the area ratio of Ti-rich TiC was 2.8-3.2 $area\%$, so the remaining ratio of TiC was calculated to be 5-17 %.

5. Conclusion

Under the present experimental conditions: Ti6Al4V with 2.0 vol.% TiC, TiC does not melt into the matrix phase Ti6Al4V in the sample preparation phase but in the ESL, and the remaining TiC was 5-17 % of added TiC after ESL melting.

References

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