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ガス浮遊炉で溶解・凝固した W 粒子分散アルミニウム合金の内部構造の評価

Microstructural Evaluation of W-Dispersed Aluminum Alloys Solidified via Gas Levitation Melting

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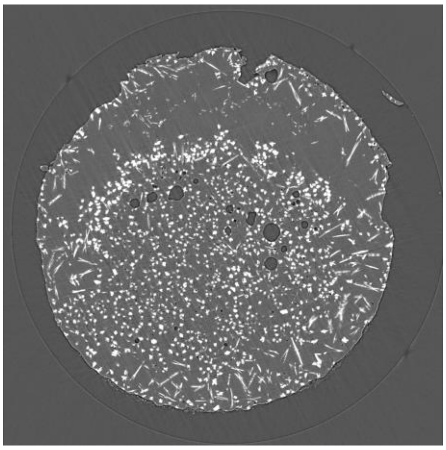

Abstract: Al-10 mass% W-based alloys were prepared by means of powder metallurgy methods. As raw powder materials, Al of 99.99 % purity with an average diameter of 20 μm, W of 99.99 % purity with an average diameter of 50 nm or 1 μm were used. After mixing these powders, round-shaped pre-formed materials (ϕ 8 x 5mm) were made by hot-pressing, 200MPa at 400˚C for 10 min. Using the pre-formed materials, spherical-shaped specimens (ϕ 2 mm) for Ar gas levitation melting were prepared. The test materials were melted by floating by using CO_2 laser, holding at temperatures ranging from 5 to 60 seconds. Internal microstructures of the materials after the Ar levitation melting were analyzed with synchrotron X-ray micro-computed tomography at SPring-8 with an X-ray energy of 40 keV. It was shown that internal microstructures were changing depending on the maximum temperature and holding time. When the Al- 10% W alloys were melted at 670 ˚C for 30 sec, W-based particles were un-uniformly dispersed. Two types of W-based particles, with needle or block-shaped ones, were observed.

Keywords: Nanoparticle, Tungsten, Aluminum, Gas Levitation Melting, Alloying, Gravity Effect, Synchrotron X-ray Computed Tomography

1. Introduction

It is widely known that alloying elements contained in aluminum alloys produced on Earth tend to become segregated within the bulk material due to differences in specific gravity between aluminum and the alloying elements (gravity segregation). Alloying elements with a higher density than aluminum tend to accumulate in the lower part of the ingot, while lighter elements (including gaseous elements) tend to concentrate near the surface. In conventional melting processes, the molten metal is stirred just before casting to homogenize the solute distribution; however, during solidification, the influence of gravity causes the distribution of strengthening phases and porosity to become non-uniform again. Uniform dispersion of hard nanoparticles within aluminum makes it possible to produce high-strength alloys. Meanwhile, advances in synchrotron X-ray imaging techniques have achieved spatial resolution at the nanometer scale, enabling highly precise evaluation of the three-dimensional distribution of nanoscale strengthening phases and porosity that could not previously be characterized. In this study, the morphology and spatial distribution of dispersed phases and porosity inherent in hard nano-tungsten particle–dispersed aluminum alloys, melted and solidified in a ground-based gas levitation furnace, will be quantitatively investigated using synchrotron X-ray tomography. The influence of gravity on the distribution of strengthening dispersed particles and porosity within the alloy will be clarified. The findings will be applied to the development of high-strength aluminum alloys in which nano-strengthening particles and porosity are uniformly dispersed and controlled.

2. Experimental

2.1. Material preparation

Aluminum powder (purity 99.99 %, particle size 20 μm, Toyo Aluminium K.K.) and tungsten powder (purity 99.9%, particle size 1 μm (micro-W) or 50 nm (nano-W), EM Japan) were used as raw materials. Each powder was mixed to obtain an Al–10 mass% W alloy composition and loaded into a die with a diameter of 8 mm and height of 5 mm. A preform was then fabricated by hot pressing at 400 °C under a pressure of 200 MPa for 10 minutes.

2.2. Levitation Melting

From the obtained preform, samples with a diameter of 2 mm were cut out and levitated in an argon gas levitation furnace. The samples were heated with a semiconductor laser, melted, held at the molten state for a certain period, and then solidified by switching off the laser. The laser heating current was set to either 2 A or 4 A, with a maximum temperature of approximately 1000 °C. The holding time was adjusted in the range of 5 to 60 s.

2.3. Synchrotron X-ray analysis

The recovered samples (ϕ 2 mm) after levitation melting were examined for their internal structure using X-ray micro-CT at the X-ray imaging beamline BL20B2 of the large synchrotron radiation facility SPring-8 [1] as shown in **Figure 1**. The X-ray energy was 40 keV, and the voxel size was 0.72 μm. Two-dimensional CT images obtained from the observations were reconstructed into three-dimensional images using the rendering software Amira-3D. Images of W particles observed inside the samples were extracted through binarization processing using ImageJ. A program developed in MATLAB was then used to measure the size and number of W particles contained within the inspection volume (2.26 mm^3) inside the samples.

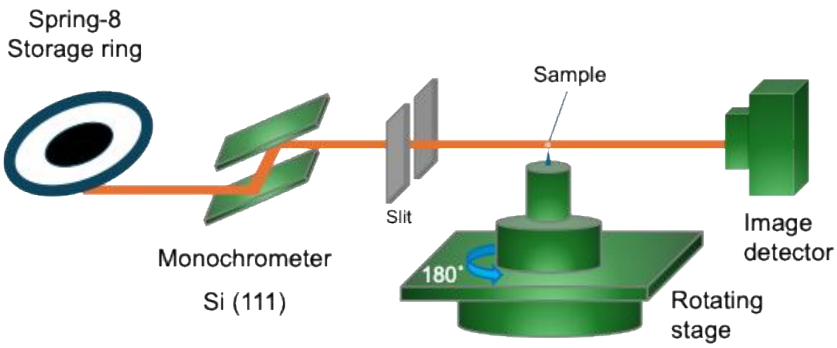

Figure 1. Setup of synchrotron X-ray microtomography in SPring-8 (Beamline: BL20B2).

3. Results

Figure 2 shows the CT image of the central cross-section and the corresponding temperature history of an Al–10% W alloy (particle size: 1 μm) levitation-melted in argon at 940 °C for 5 s with a heating current of 2 A. The observed W particles were distributed throughout the entire sample, and two distinct morphologies—blocky and needle-like—were identified depending on the observation location. Based on thermodynamic analysis using the Thermo-Calc software, the blocky particles were estimated to be $Al_{12}W$, while the needle-like particles were estimated to be Al_4W. This variation in particle structure with location is considered to reflect differences in thermal history during the solidification process following levitation melting.

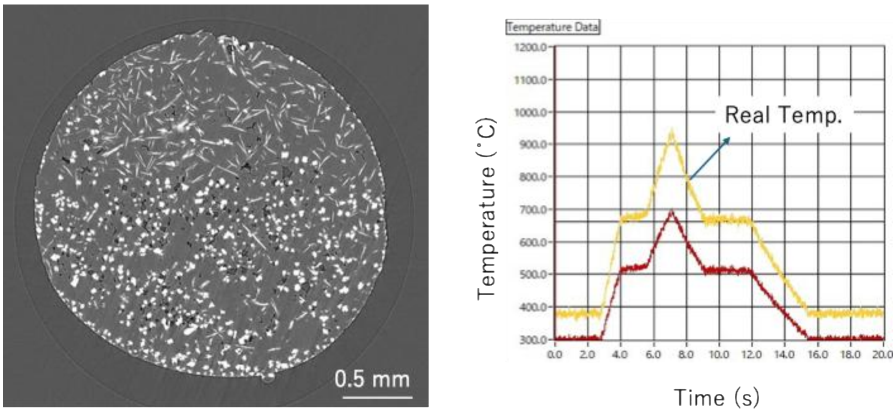

Figure 2. X-ray microtomographic image of Al-10%W (micro-W) after levitation melting at 940˚C for 5s.

Figure 3 shows the CT image of the central cross-section and the corresponding temperature history of an Al–10% W alloy (particle size 50 nm) levitation-melted in argon at 1050 °C for 10 s with a heating current of 2 A. The W particles were distributed throughout the entire sample, and compared with the case in Figure 2, they were found to be slightly finer. As in Figure 2, two distinct morphologies—blocky ($Al_{12}W$) and needle-like (Al_4W)—were observed depending on the location. Quantitative analysis of the particles with different compositions (**Table 1**) revealed that both the average size and number density of $Al_{12}W$ particles were greater than those of Al_4W.

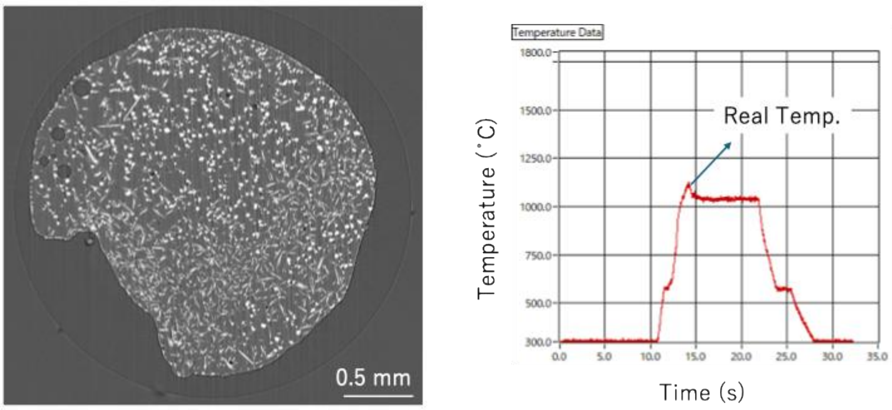

Figure 3. X-ray microtomographic image of Al-10%W (nano-W) after levitation melting at 1050˚C for 10s.

Table 1. Number density and average diameter of W-based particles in Al-10%W alloy in Fig.3

Particles	Density (1/mm^3)	Average diameter (μm)
Al_4W (Needle)	1381	3.35
$Al_{12}W$ (Block)	53871	5.63

Figure 4 shows the CT image of the central cross-section and the corresponding temperature history of an Al–10% W alloy (particle size 50 nm) levitation-melted in argon at 670 °C for 30 s with a heating current of 4 A. At the lower melting temperature, the observed particles were found to consist almost entirely of blocky $Al_{12}W$. Comparison with Figure 3 revealed that increasing the holding time from 10 s to 30 s resulted in a tendency for the observed $Al_{12}W$ particles to concentrate in the lower region. This suggests that during melting, the distribution of $Al_{12}W$ particles may have been influenced by gravity, leading to their segregation.

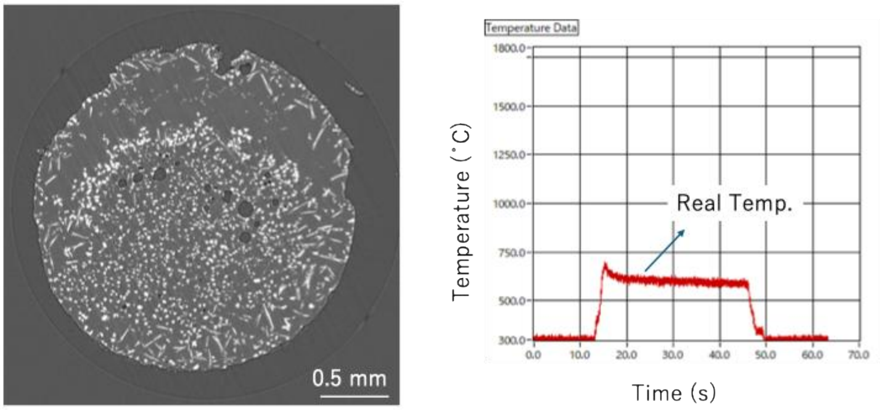

Figure 4. X-ray microtomographic image of Al-10%W (nano-W) after levitation melting at 670 ˚C for 30s.

4. Summary

Observation of the internal structure of Al–10% W alloys containing dispersed W particles, fabricated by powder metallurgy and subsequently melted and solidified in a ground-based argon gas levitation furnace, revealed the following: (1) Reducing the size of the W particles in the preform results in an overall decrease in the W particle size after levitation melting, (2) At higher melting temperatures, needle-like Al_4W forms, whereas at lower temperatures, blocky $Al_{12}W$ is formed. (3) Prolonged holding at low temperatures during levitation melting resulted in a tendency for the dispersed W particles to segregate toward the lower region.

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Conflicts of Interest

The authors declare no conflict of interest.

References

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