Extremally low oxidation Al and Ti metal fine powder prepared by a developed induction thermal plasma system

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Abstract: A new system to prepare submicron-sized oxidation resist metal powder by induction thermal plasma process was established. In this work, we focus on Ti and Al metal fine powder which are known to be one of the easily oxidable metals. The shape of processed powders was spherical with single crystal. The particle sizes of 205 ± 88 and 55 ± 30 nm and the thickness of oxide layer on the surface of 0.6 and 0.5 nm were obtained for Al and Ti, respectively.

Keywords: Fine metal powder, Al, Ti, Thermal plasma, Low oxygen system

1. Introduction

The process to prepare metal fine powder with low oxygen content is limited, especially for the ductile metals which cannot be refined by mechanical top-down method, such as ball milling, mechanical alloying, jet milling and so on, and/or the metals which cannot be reduced by hydrogen. In case of metals which can be reduced by hydrogen, we can use fine oxides powder which is prepared through a chemical route as a starting material and then after obtain fine metal powder by hydrogen reduction. While, in case of metals which cannot be reduced by hydrogen, we have a choice to use Ca with higher reduction ability as a reducing agent[1], for example. It should be pointed out that remaining CaO in the powder is washed out by water normally, indicating that the surface oxidation is inevitable.

The induction thermal plasma process is one of the promising physical bottom-up processes for the preparation of submicron-sized powder directly, due to its dynamic high temperature process[2] with a controllability of the particle size (ideally monodispersed) [3, 4] and an inherently contamination-free. For easily oxidizable metals, however, this process has some difficulties to correct the processed fine powder in the air. Therefore, the additive technique to prevent the oxidation should be considered using a thin protective layer[5].

Recently, we developed a new set up to prepare the submicron-sized oxide resist metal powder using low oxygen induction thermal plasma (LO-ITP) system[6]. In this work, we focused on Al and Ti which were known to be one of the most easily oxidizable metals and prepare metal particles with extremely low oxygen content.

2. Experiment

The detail of LO-ITP system we developed is shown in ref. [6]. The starting materials were the coarse metallic aluminum powder (particle size <20 μm, purity 99.9 %, Kojundo Chemical Lab. Co., Ltd., Japan) and the coarse metallic titanium powder (particle size <45 μm, purity 99.9 %, Kojundo Chemical Lab. Co., Ltd., Japan). A process pressure of 100 kPa was used in the thermal plasma process. The raw metal powder was then introduced from the top of the plasma torch at a feed rate of ~0.011 mol/min. Here, G1 grade Ar was used as the plasma and carrier gas, and their flow rates were 35 and 3 L/min., respectively. The input power of 6 kW was used.

The phase was confirmed by XRD (PANalytical, Empyrean, Co-Kα. The particle morphology and size distribution were estimated from images taken with a scanning electron microscope (SEM; JEOL, JSM-7800F). The microsample for STEM and EBSD measurement was prepared by a FEI Scios (FIB-SEM; FEI, Scios). The oxygen concentration of the powders was measured by EMGA-620W (HORIBA, Ltd) without atmospheric exposure.

3. Result and Discussion

Figure 1 shows SEM images of Al and Ti. The particle sizes of 205 ± 88 and 55 ± 30 nm were estimated for Al and Ti, respectively. This difference is related to the intrinsic properties of a surface tension and a vapour pressure and/or quenching rate which is originated from the temperature distribution in the chamber[7]. A single phase of Al and Ti is obtained as shown in Figure 2(c). Note that no oxide phase was detected from the conventional XRD facility for both Al and Ti submicron powders as it is known to exist in the amorphous form [8]. Since we use Co source for XRD measurement, the peaks from Co-Kβ is also detected.

In order to find out whether the particle is single crystal or not, the diffraction pattern obtained from TEM measurement is suitable. However, we do not have a transport setup without atmospheric exposer to the TEM facility so far, after micro sampling by FIB. In this situation, there is possibility that the sample is oxidized during the transportation in the air and the crystal structure might be changed. Therefore, in this work, an EBSD in FIB-SEM machine was used. Figure 2 shows STEM and EBSD image of Al. We found that each Al particle is single crystal. For Ti, unfortunately, a clear EBSD map was not obtained since several Kikuchi patterns from several particles were overlapped due to a smaller particle.

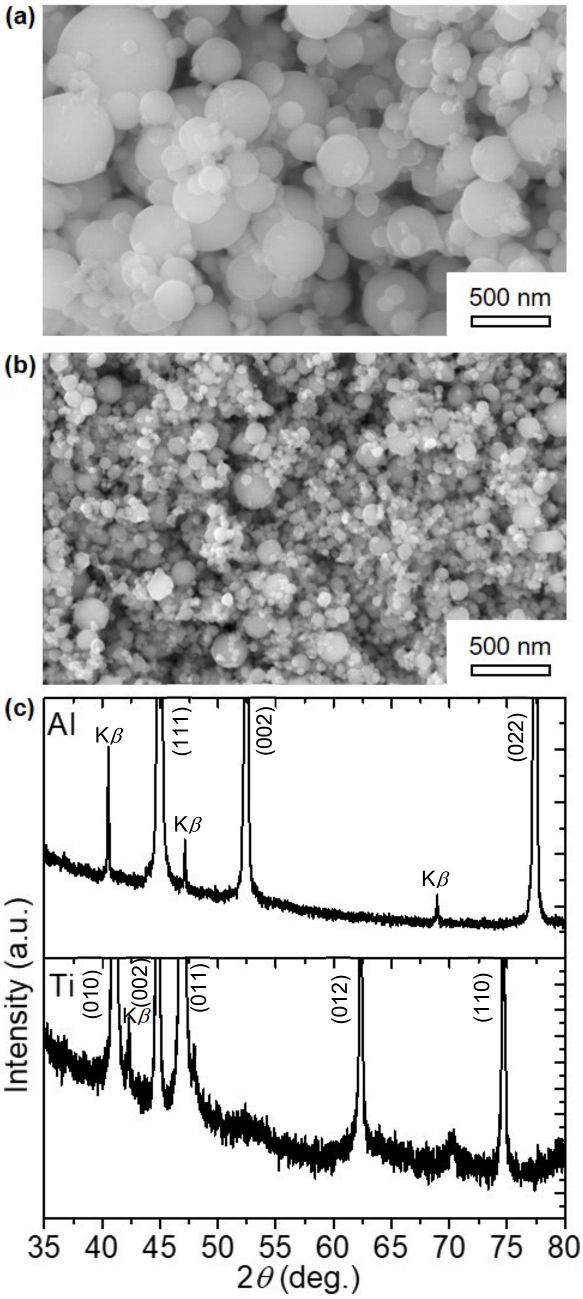

Figure 1 SEM images of (a)Al and (b)Ti powder and XRD profile of Al(upper) and Ti(lower). The sample for XRD was sealed by Kapton film properly in order to avoid the oxidation in the glove box.

The oxygen concentration of Al and Ti were 1.01 wt.% and 1.42 wt.%, respectively, indicating that the thickness of surface oxide layer of around 0.6 and 0.5 nm were estimated, respectively, when the surface oxide layers consist of $Al(OH)_3$ and TiO. This thickness of oxide layer was quite thin. Thus, this induction thermal plasma system we developed realizes the preparation of oxidation resist submicron metal powder.

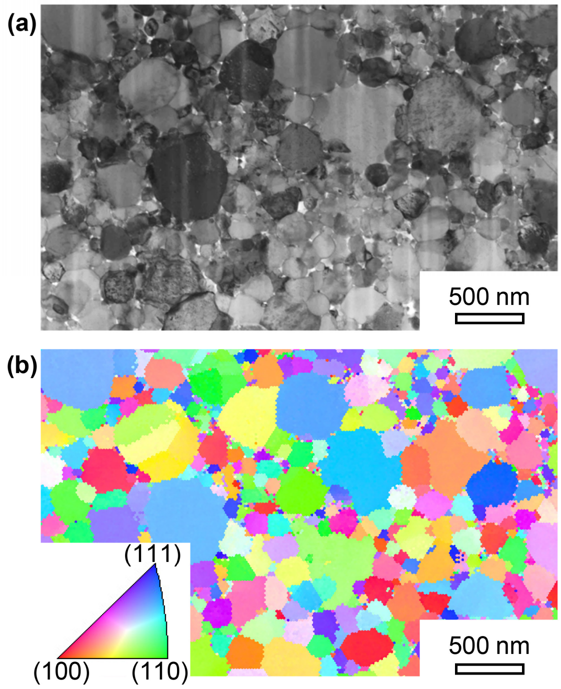

Figure 2 (a)STEM image of Al film cut off from Al green compact by FIB and (b)EBSD image in the same area of (a), which is collected at the acceleration voltage of 30 kV.

4. Summary

We prepared extremally low oxidation Al and Ti metal fine powder prepared by a low oxygen induction thermal plasma system. The particle size is around 205 nm for Al and 55 nm for Ti. The oxygen concentration of Al and Ti were 1.01 wt.% and 1.42 wt.%, respectively, indicating that the oxides on the surface of the particle were quite thin. Therefore, this induction thermal plasma system we developed realizes the preparation of oxidation resist submicron metal powder.

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6. References

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