§18. Reduction Behavior of Titanium Oxides during Microwave Heating Using High Vacuum Devices

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We study reduction behavior of TiO2 during microwave heating using high vacuum devices. Details of this device are shown in the annual report[1-2]. The sample was heated by electric furnace at about 1200 ℃.and kept 10000 seconds. And we heated the sample by microwave H-field at about 1600 ℃ and kept about 1000 seconds. We measured the temperature, total pressure of the system, and ion current of outgas. The rutile powder which purity is 99.9% (purchased from Kojundo chemical lab., Co., Ltd., Japan) is molded like the column of 8 mm in the diameter and 2 mm in height. The amount of the sample is about 0.01 mol. Any reductant agent has not been added at all.

Fig.1 shows that time dependence of temperature and partial pressure of oxygen in conventional heating. Open circles indicate temperature of the sample. Solid circles indicate partial pressure of oxygen. With temperature keeping at 1200℃, the value of partial pressure of oxygen was decreasing gradually.

Fig.2 shows that time dependence of temperature and partial pressure of oxygen in microwave H-field heating. At first, the sample was heated to 1600℃. While increasing in temperature, the value of partial pressure of oxygen is increasing rapidly to the order of 5×10^{-2} Pa. The partial pressure of oxygen rose to 2×10^{-2} Pa when lowering the sample temperature up to 800℃ and heating the sample up to 1600 ℃ again once. With temperature keeping at 1300℃, the value of partial pressure of oxygen is also keeping during 150 seconds. But the value of partial pressure of oxygen was decreasing rapidly with temperature keeping at 1400, 1450 and 1500℃.

Fig.3 shows that temperature dependence of partial pressure of oxygen in microwave H-field heating. Data enclosed in solid line are partial pressure of oxygen data of 200 - 400 seconds in Fig.2(we call them First-heated data). Data enclosed in dot-line are partial pressure of oxygen data of 600 - 1200 seconds in Fig.2(we call them Re-heated data). In 1400℃, First-heated data is 100 times larger than Re-heated data. And these data draw the hysteresis loop. And then, data that heated after microwave power putted off are show the same behavior as Re-heated data. From Fig. 1, partial pressure of oxygen by electric furnace is almost the same as Re-heated data and after microwave heating. Therefore, when first heating the sample, reduction reaction promoted, but when re-heating the sample or after microwave heating and cooling naturally, reduction reaction is not promoted.

In summary, it is guessed that the microwave heating promotes reduction reaction only when the sample is heated by microwave h-field at first. Meanwhile, partial pressure of oxygen during re-heated by microwave does not promote reduction reaction. In future, we would like to reveal the mechanism of reduction process during microwave heating.

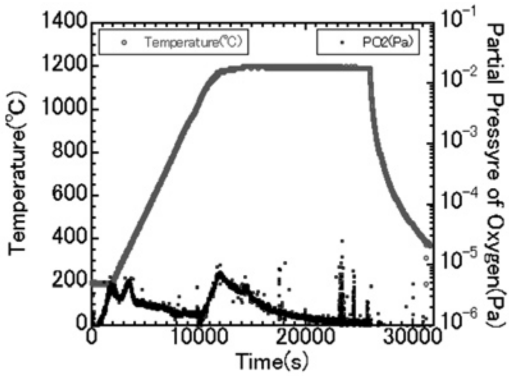

Fig. 1. Time dependence of temperature and partial pressure of oxygen in conventional heating.

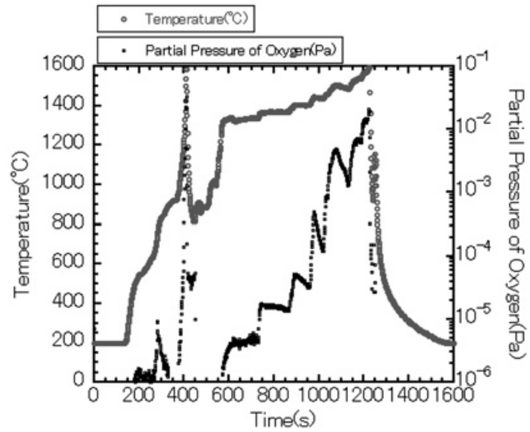

Fig. 2. Time dependence of temperature and partial pressure of oxygen in microwave H-field heating.

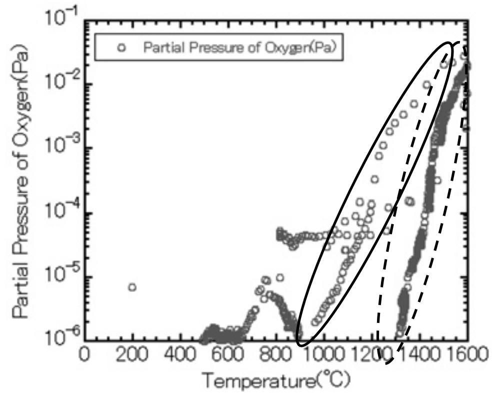

Fig. 3. Temperature dependence of partial pressure of oxygen in microwave H-field heating.

1) Fukushima J. et al. Annual report of national institute for fusion science (2009), 420, ISSN 0917-1185
2) Fukushima J. et al. Annual report of national institute for fusion science (2010).