§24. Basic Physics on Reduction Reaction of Ferromagnetic Oxides under Microwave Irradiation

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Interaction between microwave and materials had not been cleared because the interaction includes non-equilibrium process. The purpose of the study is an investigation of basic material physics on interaction between microwave electromagnetic field and materials. To clear the interaction between microwave electromagnetic field and materials, the authors focus on a reduction behavior of ferromagnetic materials during microwave irradiation. The reduction behavior depends on non-thermal microwave effect, rate of heating, selective heating effect, etc. In this study, we tried to clear the effect of heating rate on the reduction behavior of $NiMn_2O_4$ using In-situ outgas measurement system.

Experiment was conducted using the device for measuring a mass of gas emitted from a sample during microwave irradiation under high vacuum state. The details of the device have been reported[1)]. Before in-situ gas analysis experiment, $NiMn_2O_4$ was synthesized by electro furnace from mixed powder of NiO (99.9 % up, Kojundo Chem. Lab. Co. Ltd.) and $MnCO_3$ (99.0+ %, Wako Pure Chem. Inc. Ltd.) at 850 °C for 12 h. The synthesis procedure was repeated three times and the authors obtained single-phase $NiMn_2O_4$. This procedure have been reported[2)]. The pre-synthesized nickel manganate powder (0.2 g) was pelletized to 6mmϕ was irradiated with microwave at the maximum point of H-field. The microwave furnace was composed of a single-mode TE103 cavity and a semiconductor oscillator (Nagano Japan Radio Co., Ltd., NJZ-2450). A rate of heating was controlled by changing a microwave power. For comparison, another sample was heated in an electric furnace.

The sample absorbed microwaves well and we could control the rate of heating by changing a microwave power. To conduct experiments under the same condition of microwave irradiation, we controlled a reflected power ratio to 30 dB by using a vector network analyzer before microwave irradiation. In conventional heating, heating rate was set as a maximum value. Figure 1 and 2 show a sample temperature and partial pressure of oxygen dependence of time. In Fig. 1 (a), the microwave power was 120 W. Although the microwave heating process is a rapid heating, we could be obtained quadrupole mass spectrometer (Q-mass) data per 0.5 sec. In microwave heating, we had to switch off the microwave power at 350 °C because a lot of out gas was emitted. On the other hand, in conventional heating, out-gas was not rapidly increased and we can heat the sample over 500 °C. The profiles of partial pressure of oxygen in each heating have three peaks. At lowest temperature, oxygen was emitted around 60 °C in microwave irradiation and between 200 and 300 °C in conventional heating. The peaks was very small and the emission resulted from adsorbed oxygen or oxygen which split adsorbed water at an electrode of Q-mass. Thus second peak (②) was the first reduction reaction of the sample. In previous work[2)], reduction reaction of the sample was occurred in air. Therefore, we focus on the second peak. In microwave heating, the reduction reaction was started around 100 °C, but it was started around 300 °C in conventional heating. In other words, the reduction reaction in microwave was enhanced. The results are similar to our previous works[3, 4)].

We are now analyzing the relationship between reduction rate and heating rate by Arrhenius plot of the results.

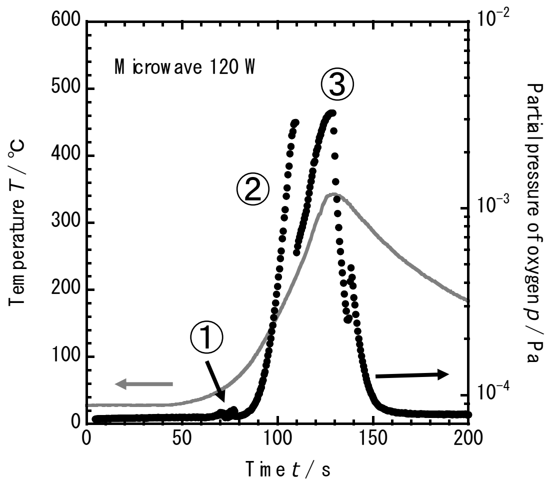

Fig. 1. Sample temperature and partial pressure of oxygen dependence of time in microwave heating

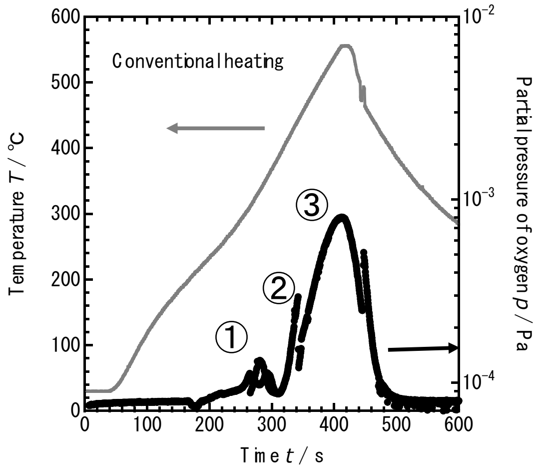

Fig. 2. Sample temperature and partial pressure of oxygen dependence of time in conventional heating

1) Fukushima, J. et al.: *NIFS Ann. Rep.*, (2009) 420.
2) Goto, H. et al.: *Materials* **9** (2016) 1-12.
3) Fukushima, J. et al.: *Mater. Lett.*, **91** (2013) 252-254.
4) Fukushima, J. et al.: *Proc. Mater. Sci. Tech.*, (2010) 2831-2836.