§26. Development of the High Vacuum Device to In-Situ Measurement of Partial Pressure of Oxygen on Microwave/Conventional Heating

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Effects of microwave heating have recently been receiving much attention. One example of the effect of microwave heating is the microscopically thermally non-equilibrium state. During microwave heating, many "local hot spots", that has large temperature gradients of 200~400 degrees Celsius in the order of 10~100 μm region, are generated. For the other example, nano-particles with random orientation of the axis are observed after microwave heating of Fe_3O_4 powder[1).

Though many phenomena of microwave heating are reported, there is little information for explanation to microwave effects. This is because many experimental factors in previous studies to reveal microwave effects. For instance, when thinking about the reductive reaction, there were a lot of parameters (inert gas atmosphere, reductant, etc.), and the unique effect of reduction of the microwave was not able to be revealed. Moreover, frequency stability was not good either. Then, the authors have developed the high vacuum device for simple system and high precision microwave system.

<u>i). High vacuum device to measure partial pressure of oxygen</u>

Fig. 1 shows schematic view of the high vacuum device to measure partial pressure of oxygen. It is made from the stainless steel, so baking above 300 ℃ were possible. A turbo molecular pump(TG50F,OSAKA VACUUM Ltd., Osaka, Japan) was used. It can generate many degrees of vacuum up to ultra-high vacuum levels ($\sim10^{-6}$ Pa). Specimen temperature was measured by radiation thermometer(FTZ6-R220, Japansensor, Tokyo, Japan) and two color thermometer(IGAR12-LO/MB13, IMPAC, Germany). Total pressure of the system was measured by full range vacuum gage(PKR251, HAKUTO Co., Ltd., Tokyo, Japan). And we can analyze partial pressure of outgas by the quadruple mass spectrometer system(QMG220, Pfeiffer Vacuum Inc.).

We are able to calculate the partial pressure of oxygen of outgas with data of quadruple mass spectrometer and exhaust velocity of turbo molecular pump.

<u>ii). High precision microwave system</u>

Fig. 2 shows the schematic diagram of the microwave heating apparatus. In this applicator, magnetron which outputs 1.5kW within the range of the frequency of 2.455GHz±10MHz(PMJ-1000-L, EWIG Co. Ltd., Tokyo) was used. And a single-mode cavity was utilized, which have a waveguide of TE_{103} mode. Plunger is set at the end of the waveguide, which act as a metal wall and reflects electromagnetic waves. It slides back and forth by linear motor CPL28T08B-06, Orientalmotor Co., Ltd., Tokyo. Japan). This plunger is high precision, thus we are able to control temperature of sample easily.

A test tube was placed in the waveguide at the maximum point of electric or magnetic field. Heating was performed with tuning the power of the reverse wave to be minimized by manipulating the three stub tuners and the plunger. Input power and reverse power are monitored by a dual directional coupler. Isolator which assembled next to a magnetron is used to prevent the reflected wave from returning. In order to control oscillating frequency, a novel method called "mode-lock" was applied. A low power microwave, generating by a semiconductor oscillator(NJZ-2450A, Nagano Japan Radio Co., Ltd., Tokyo, Japan), is intentionally put into the magnetron through an isolator which assembled next to the other isolator. This semiconductor oscillator is able to change an oscillating frequency with 0.1MHz increments in between. As a result, we can control and fix the oscillating frequency of a magnetron the range of ±0.1MHz.

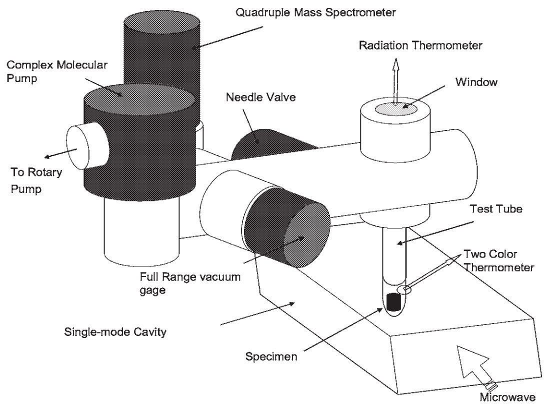

Fig. 1. Schematic view of the high vacuum device to measure partial pressure of oxygen

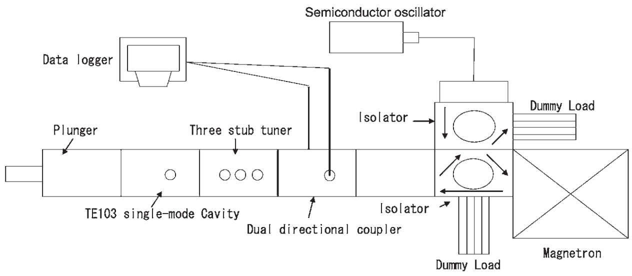

Fig. 2. Schematic diagram of Microwave heating apparatus. This system can divide into electric and magnetic fields by single-mode cavity. Mode-locked microwave is oscillated incorporating semiconductor oscillator and isolators.

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