§10. Observation of the Temperature-Jump of the Sample during Microwave Iron Making

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The microwave heating technology accumulated through the nuclear fusion research has contributed widely to the field of microwave processing. The fact that the microwave heating can be applied to powdered metals has paved the way for the development of the field of its science and application. [1] Highly pure pig irons can be produced in a microwave reactor from powdered iron ores with carbon as a reducing agent. [2] A typical chemical equation can be given as $Fe_3O_4 + 2C \rightarrow 3Fe + 2CO_2 - 75.66$ kcal/mol, as magnetite (Fe_3O_4) is employed. This reaction is endothermic, so that microwave energy sustains the reaction. In this report, we present a nature of the microwave heating in the iron making.

The hexagonal cross-section of the microwave furnace shown in Fig 1 reduces the microwave energy concentration to the center. The microwave (2.45 GHz, 2.5 - 5.0 kW) heating was performed in the nitrogen atmosphere. The infrared pyrometer monitored the surface temperature. UV-visible light emissions were observed with the spectrometer. The weight ratio of magnetite and graphite powders (these grain sizes were under 50 μm) was Fe_3O_4 : C = 89 : 11. The amount of carbon was equivalent to the mol concentration for de-oxidation of the magnetite to pig iron. Volume and total weight of the sample were 89 cc and 89.3g respectively.

Temporal evolution of the surface temperature is displayed in the Fig 2. For the period of $t < 380$ s the system is low temperature ($T < 690$ °C) state. Small hot spots due to the discharge spark, less than 1 mm in diameter, blink in the cracks of the sample. For the period of 380 s < t < 390 s, T jumps from 690 °C to 980°C in a few second. Very bright discharge flashes, and then the bright flame bursts up. It should be mentioned that from this moment the temperature of the outside wall of the crucible begins to increase rapidly, indicating the sample-temperature monitored by the pyrometer detects indeed the T-jump without crucial overestimate by IR lines from both the sample and flame. Its UV-visible emission spectra consist mainly of carbon-nitride (CN) and Iron atoms (Fe I) [see Fig.2]. The Fe (I)'s continue, while the CN disappears in a few minutes. No emission lines of CO or CO_2 are detected. After the burst ($t > 390$ s), T rises gradually from 950 °C; at $T \approx 1350$ °C molten metal appears in the crucible. Then, the microwave power is turned off, and the emission and flame vanishes.

The T-jump indicates the change of energy balance of the sample. A possible interpretation is as follows. Before the jump, the sample is heated mainly by the microwave absorption of which channel is said (but still major open question) to be, e.g., Joule, dielectric, eddy-current, spin wave, multi photon, and etc. Just before the jump, the sample temperature reaches ~650 °C which is coincident with the critical value for the spontaneous chemical reaction, according to the comparison of Gibbs' free energies for iron oxide and carbon oxide. Under this condition, a discharge spark can ignite the burst and trigger the T-jump. The hot-spot temperature becomes much higher than the critical value of ~650 °C; so that CO and CO_2 are produced locally. Those particles are heated and excited by electrons that are multiplied with avalanche in the hot spot, resulting in molecule heat flux. The heat flux gives thermal energy to the surround of the hot spot, leads to the chain reaction and develops to the burst.

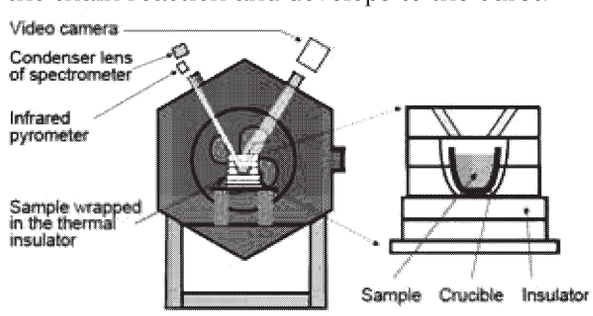

Fig.1. Schematic diagram of the microwave furnace.

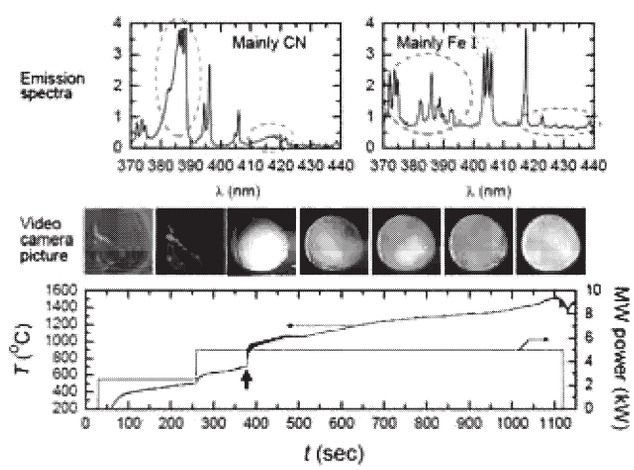

Fig.2. Emission spectra, video camera picture, and variation of the surface temperature of the sample.

[1] Roy, R., et al., Nature **399**, 668 (1999).
[2] Sato, M., et al., 11th *International Conference on Microwave and High Frequency Heating*, O-24, Sep.11-15, (2005).