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Localized Heating during Carbothermic Reduction of Hematite Powders using Microwaves

Abstract:

Microwave heating has recently received attention as an innovative means of applying energy in material processing, and shows potential for CO₂ reduction and energy conservation, due to selective and volumetric heating effects, and the ability to apply heat by non-fossil energy. In order to examine the applicability of microwave heating to iron making, experimental processes and small scale pilot projects have been conducted. Most commonly, magnetite is utilized as the raw material for microwave pig iron making due to its excellent microwave heating properties, however, it would be beneficial if highly abundant hematite ores could be utilized as well. It has been observed that hematite can be used at the cost of a much lower heating rate at low temperatures, but at higher temperatures microwave absorption increases and reaches temperatures similar to magnetite samples. It is unclear whether this increase in absorption is a property of hematite, or whether it is the result of a phase transition. To gain clarity on the matter, the heating properties at elevated temperatures of hematite while observing its chemical structure need to be examined.

Also, many researchers believe there is a possibility that the differences in microwave absorbance among mixed powder components may result in what is referred to as *Localized Heating*, where the different components are of different temperatures. There are only limited research on whether or not this is possible, and there are many unanswered questions regarding the thermal conditions that occur during microwave irradiation.

In this research, *in situ* XRD experiments were conducted to elucidate the microwave heating properties of common raw materials in such processes, the occurrence of micrometer scale thermal gradients within samples, and to observe the carbothermic reduction of magnetite and hematite iron ores.

The contents of respective chapters are summarized below:

Chapter 1: Introduction

The motives of using microwave heating in iron making are introduced, and previous research on microwave pig iron making are presented. The preferred usage of powdery hematite iron ores in such processes is explained, and ambiguities regarding temperatures during reduction experiments are illustrated. Finally, the objectives and structure of the following chapters are outlined.

Chapter 2: Development of *In Situ* XRD and Microwave Heating Combined System Capable of Local Temperature Measurements and Stable Irradiation of Poor Absorbers

To enable the measurements required by the subsequent chapters, an *in situ* X-Ray Diffraction(XRD) microwave heating system was devised. The concept of this measurement technique is explained and test experiments to confirm the applicability of the method were conducted. For the high stability requirements during microwave irradiation experiments of low absorbers to be met, further control systems were developed.

It was shown that the method could provide relevant data for temperature evaluation, and that stability of irradiation could be achieved with the developed system.

Chapter 3: In Situ XRD Evaluation of Specific Temperatures of Microwave Irradiated Unmixed Powders for Pig Iron Making

In chapter 3, the microwave heating properties of powders used in pig iron making are investigated using the developed *in situ* XRD system to provide basis for discussion regarding the heating behavior of mixed powders.

To enable accurate temperature evaluation the thermal expansion properties of the respective powders required by the *in situ* XRD technique were investigated using conventional High Temperature XRD(HTXRD).

Heating properties of the unmixed powders were successfully investigated from the accurately evaluated temperatures at controlled irradiation levels, even for poor absorbers. It was seen that hematite exhibits thermal runaway above 170°C.

Chapter 4: Local Temperature Measurements of Mixtures of Good and Poor Absorbing Powders during Microwave Heating using In Situ XRD

In this chapter, the feasibility of the localized phenomena is investigated using the system developed in chapter 2 was used to attempt observation of localized heating in mixed powders. This phenomena has previously been theorized as a possible cause of increased reaction kinetics during microwave processes, however, no experimental evidence for its occurrence is available. To maximize the possibility of observing said phenomena within the time resolution of the developed technique, mixed powders of non-reacting absorbers and non-absorbers were used.

It was found that a temperature difference of at least 100°C could be observed between alumina and graphite particles and that no localized heating among magnesia and graphite particles could be observed. However, since localized heating was possible in one of the cases, it is likely that localized heating occurs for most cases at a shorter time scale than the time resolution of the used technique.

Chapter 5: Evaluation of Microwave Heating Properties of Mixed Powders during Carbothermic Reduction of Magnetite using *In Situ* XRD

In chapter 5, the carbothermic reduction of magnetite is observed using the *in situ* XRD technique to provide a reference for the following hematite experiments. Temperatures were evaluated from the obtained XRD scans using literature data.

It was observed that samples could be rapidly heated and reduction of magnetite occurred gradually into iron. However, above heating properties rapidly decrease at temperatures above 1000°C.

Chapter 6: Evaluation of Microwave Heating Properties of Mixed Powders during Carbothermic Reduction of Hematite using *In Situ* XRD

Highly abundant hematite powdery ores is preferred as a prospect raw material for microwave pig iron making. In previous research, it has been shown that hematite mixed powders are heated at a lower initial rate compared to magnetite, followed by a rapid increase in temperature. However, ambiguities regarding the thermal conditions and possible phase transition occurring are unresolved.

In chapter 6, thermal conditions and the heating properties of hematite and graphite mixed powders during carbothermic reduction using the *in situ* XRD microwave furnace are investigated. The temperatures of graphite and hematite were evaluated using the HTXRD data obtained in chapter 3. For product phases, the literature data used in chapter 5 were used.

It was observed that hematite mixed with graphite can be rapidly heated without any phase transition of hematite, reaching even higher temperatures than magnetite followed by a temperature decrease. This decrease in temperature occurs along with reduction of the hematite phase, and it is possible that hematite greatly assists the heating prior to its reduction since a similar temperature profile could not be observed for magnetite. Iron phase was predominantly generated during the temporary increase in temperature at temperatures ranging between 1200~1270°C. From the results of chapter 3 and 4, it is possible that hematite reaches a higher temperature than graphite below the time resolution of the method due to thermal runaway.

Chapter 7: Final Conclusions

As was seen from the results of chapter 6, the applicability of hematite in microwave pig iron making was confirmed. If the initial temperature of hematite can be effectively increased using heat from graphite microwave absorption, hematite should aid in the absorption of microwaves and possibly attain temperatures higher than those possible in magnetite mixed powder experiments.