IJST (2014) 38A3: 253-256
Iranian Journal of Science & Technology

http://ijsts.shirazu.ac.ir

Applications of microwave oven and microwave furnace heat resources on phase transformation: A research study on red sediment placer ilmenite

S. Sai Srikant[1]*, P. S. Mukherjee[2] and R. Bhima Rao[3]

[1]*Department of Electronics and Communication Engineering SRM University,*
Modinagar, Ghaziabad, 201204, India
[2]*IMMT-CSIR, Bhubaneswar 751013, India*
[3]*Aryan Institute of Engineering and Technology, BPUT University, Bhubaneswar, Odisha, India*
E-mail: satya.srikant@gmail.com

Abstract

Microwave energy has potential for the efficient heating of minerals. Minerals or materials, which couple to microwave energy are called dielectrics and many valuable minerals are found to be dielectric. The mineral ilmenite is one which shows good dielectric heating characteristics. In this paper, the effect of microwave heat energy on the partial reduction of oxidized ilmenite oxidized in a conventional muffle furnace at 900°C for 3 hours is attempted. Carbon as an additive was used for reduction of ilmenite at different time intervals using microwave oven as well as microwave sintering furnace. The results indicate that at one minute, the metallic iron formed contained 0.39% and no observation in mineral phases was observed from microwave oven. At three minutes duration, a partial phase transformation of ilmenite was observed and the metallic iron contained is 1.65%. Interestingly, at six minutes duration a distinct metallic iron phase containing 32% metallic iron is seen in microwave oven. The result of preliminary investigations from microwave sintering furnace indicates that apart from metallic iron phase, the distinct Titania phase was also observed. Further studies are in progress.

Keywords: Microwave heat energy; ilmenite; reduction; oxidization; metallic iron; carbon; XRD

1. Introduction

Microwaves are a form of electromagnetic energy with associated alternating electric and magnetic fields in the range of 300MHz-300 GHz. Microwave oven and microwave sintering furnace are designed to resonate at the microwave frequency thereby producing an equal and even energy distribution.

The heat is produced in the food itself by induced agitation of its molecules during microwave cooking of food (organic matter). When a microwave signal passes through the food, it aligns the polar molecules parallel to the direction of the wave. Within a fraction of a millionth second the signal reverses itself accordingly. This flip flopping results in considerable molecular friction. This friction generates heat and heat cooks the food (organic matter).

Microwave sintering furnace and microwave oven also have a wide range of applications in mineral technology, metallurgy, etc. It is an established fact that microwave energy has potential for the speedy and efficient heating of minerals and in a commercial context may provide savings in both time and energy. Literature review reveals that there are many specific applications of microwave heat energy on minerals for selective liberation of minerals or phase transformation of minerals or enhancement of magnetic properties or comminution properties etc., (Kelly and Rowson, 1998; Bhima Rao and Patnaik, 2004). In microwave heating of minerals, the materials which couple to microwave radiation are termed as dielectrics and contain dipoles (Bhima Rao and Patnaik, 2004; Sandish, and Worner, 1996). These dipoles align themselves in an applied electric field and will flip around in an alternating electric field. As a consequence, the material will be heated as the stored internal energy is lost to friction. This energy mode conversion has the advantage of being selective to individual mineral phases within a mass (Bhima Rao and Patnaik, 2004; Sandish and Worner, 1996).

Many valuable minerals are found to be dielectrics. However, once the ferrous iron is oxidized to ferric iron, its ability to form dipoles is lost and it will not heat without the use of reducing agent (Bhima Rao and Patnaik, 2004; Srikant et al., 2011; Lei et al., 2011; Pickles, 2009; Haque, 1999).

*Corresponding author
Received: 8 July 2013 / Accepted: 20 July 2014

Carbon, conveniently, is a reducing agent and when mixed with fully oxidized ilmenite allows the mixture to dielectrically heat due to extra mobility. At elevated temperature, many dielectrics increasing couple to microwave energy (Sandish and Worner, 1996; Srikant et al., 2011; Lei et al., 2011; Kingman and Rowson, 1998). This factors creates the hotspots in samples and hence a non-uniform temperature distributions.

2. Material and methods

2.1. Raw Material

Low grade ilmenite was recovered from red sediments of badlands topography of Nolia Naugam, Chatrapur, Gangam dist., Odisha. The ilmenite sample is 98% pure and on average contains 47.3% TiO_2. Graphite 99% purity sample was used as an additive for reduction of ilmenite mineral.

2.2. Oxidation of Raw Ilmenite

The raw ilmenite sample obtained from deslimed red sediment sand sample was first heated in a muffle furnace at 900°C in an air atmosphere for 3 hrs and also separately heated in a microwave oven in order to oxidize all the ferrous iron into the ferric state.

2.3. Microwave reduction of Ilmenite

Pre-oxidized ilmenite by using conventional muffle furnace was again oxidized in a microwave oven for a minute. The microwave oven used in the present investigation was IFB model 38SC1, 50 Hz, 850 W. After pre-oxidation, the sample was mixed with a fine graphitic powder (carbon) having stoichiometric amount (i.e. 10 % of graphite) which is needed for reduction process. This graphite act as a reducing agents and it helps in heating the sample as it lacks the dipole formation. The susceptor silicon carbide (SiC) placed near the sample further facilitates the microwave absorption and heating the sample in microwave oven. A small amount of SiC powder was also mixed in the oxidized ilmenite sample in order for it to heat rapidly. This sample was heated totally for 6 minutes with a lapse of every 2 minutes i.e after every two minutes of heating, the microwave oven was switched off for one minute, otherwise the microwave oven may be damaged because of metallic formation inside the oven (Srikant et al., 2011; Lei et al., 2011). Also the pre-oxidized ilmenite sample was simultaneously heated in a microwave sintering furnace for 40 minutes in order to compare the heated sample with that from microwave oven. The schematic sketch for heating ilmenite sample in microwave furnace is shown in Fig. 1.

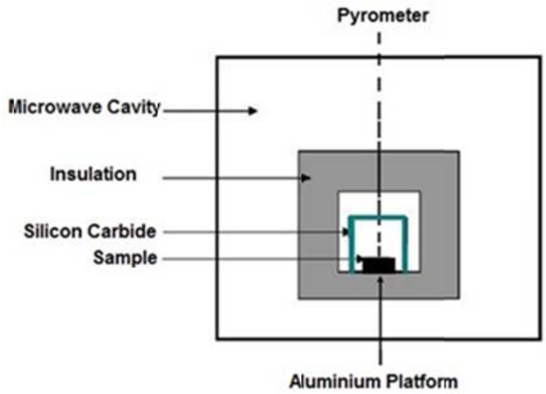

Fig.1. Schematic sketch for heating of ilmenite sample in a microwave furnace

3. Analytical Method

PANalytical X-Pert X-ray powder diffractometer with Cu-Kα radiation , (λ=1.54056 Å) from 5° to 75° scanning angle at a scanning rate of 0.05°/sec was used for phase analysis of red sediment placer ilmenite mineral. Morphological features of reduced ilmenite sample were studied using the FESEM (model: Supra 55; Zeiss, Germany) The FESEM has a resolution of 1 nm at 30 KV which is equipped with 20 mm^2 Oxford's Energy dispersive X-ray spectroscopy (EDS) detector for imaging of conducting as well as non-conducting samples without gold coating. FESEM-EDAX studies were done using Hitachi VP-SEM S-3400N. It has high SE resolution of 10 nm at 3 KV. The magnification of the instrument is 5X-300,00X; alternating voltage is 0.3-30 KV. The grains were mounted on a SEM brass stub. The mounted grains were coated with gold in a vacuum evaporator while the sample was slowly rotated.

4. Results and discussions

The effect of microwave heating on oxidized ilmenite with graphitic carbon on formation of metallic phase is distinctly observed with microwave oven. The general view of megascopic picture of ilmenite mineral grains can be clearly be seen from Fig. (2a) and close view of metallic phase of ilmenite in Fig. (2b). This clearly shows that all the ilmenite grains tend to fuse at first instance, then converts for reduction process.

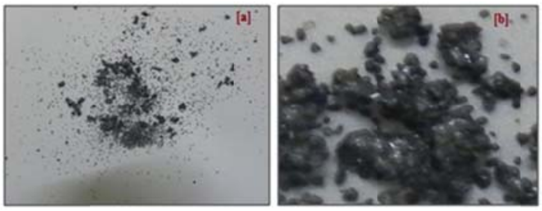

Fig. 2. Microwave heating on oxidized ilmenite with graphite on microwave oven [a] General view of megascopic picture of ilmenite grains and metallic phase, [b] close view of metallic phase of ilmenite

The results of the microwave heating on oxidized ilmenite with graphitic carbon indicate that at one minute of time the metallic iron formed contain 0.39% and no observation in mineral phases was observed. But from the heat treatment of oxidized ilmenite in a microwave sintering furnace for 40 minutes, the preliminary investigations clearly indicates that Titania was formed from XRD analysis as shown in Fig. 3 and its mineral phase shown in Table 1. A metallic iron phase was also observed.

The FESEM-EDAX analysis as shown in Fig. 4 also confirms that there is a formation of 76.5 % of Titania in the form of rich slag. It is observed that as the metallic mineral ilmenite melts at high temperatures, the carbon atoms and SiC powder form the vapour (liquid) helps to form Titania products in the form of rich slag.

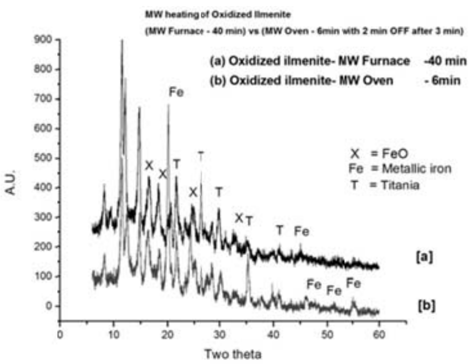

Fig. 3. XRD pattern of sample mixed with oxidized ilmenite and graphitic carbon heated in microwave oven and microwave furnace in presence of susceptor

Table 1. XRD data of placer ilmenite heated in microwave oven and microwave furnace

XRD Mineral Phase	Two Theta (2Θ)
Fe = Metallic Iron	45.23; 46.54; 51.23; 56.43
X= Iron Oxide (FeO)	16.34; 18.88; 24.56; 34.94
T= Titania	20.22; 24.098; 29.87; 41.95

Table 1 and Fig. 3 clearly indicates the various XRD pattern of iron oxide (FeO), metallic iron and Titania phase which has been observed after heating the ilmenite in microwave sintering furnace. The effect of microwave heat treatment of ilmenite minerals as shown in Table 1 with microwave oven in the presence of coupling agent (graphitic carbon) shows only the metallic iron formation.

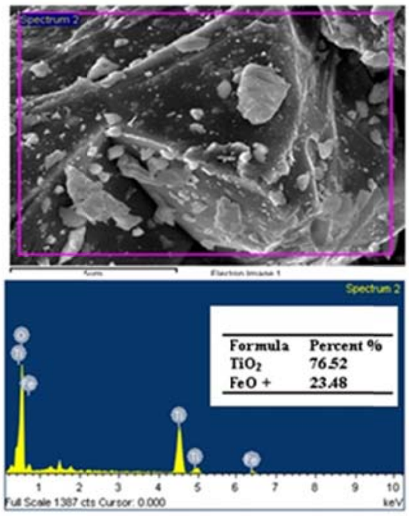

Fig. 4. FESEM-EDAX analysis data confirming Titania rich slag formation

5. Conclusions

The effects of heat treatment on oxidized ilmenite with graphitic carbon were investigated both in microwave oven and microwave sintering furnace. The preliminary investigations from XRD analysis clearly indicate that the formation of Titania (TiO_2), metallic iron (Fe) and iron oxide (FeO) phases were distinctly observed in the microwave sintering furnace whereas only metallic iron (Fe) phase formation was observed in microwave oven. The limitation for microwave oven is that after every two minutes of sample heating, the microwave oven has to be switched off for one minute otherwise the microwave oven may become damaged because of metallic formation inside the oven. The FESEM-EDAX analysis also confirms around 76.5% Titania formation in the form of slag and 23.5 % metallic iron phases from reduced ilmenite by using microwave sintering furnace.

Acknowledgments

Dr. Satya Sai Srikant is thankful to Prof. B. K. Mishra, Director, CSIR-Institute of Minerals and Materials Technology, Bhubaneswar for giving permission to utilize infrastructural facilities of CSIR-IMMT and for his encouragements to do Ph D research work and publication of papers. Thanks are also due to some of the scientists and research scholars of CSIR-IMMT for their suggestions on this research topic.

References

Bhima Rao, R., & Patnaik, N. (2004). Microwave Energy in Mineral Processing A Review. *Journal of Institution of Engineers (India)–Mining*, 84(3), 56–61.

Haque, K. E. (1999). Microwave energy for mineral treatment processes–a brief review. *International Journal of Mineral Processing*, 57, 1–24.

Kelly, R. M., & Rowson, N. A. (1998). Microwave Reduction of oxidised ilmenite concentrates. *Minerals Engineering*, 11(11), 1427-1438.

Kingman, S. W., & Rowson, N. A. (1998). Microwave Treatment of Minerals-A Review. *Minerals Engineering*, 11(11), 1081–1087.

Lei, Y., Li, Y., Peng, J., Guo, S., Li, W., Zhang, L., & Wan, R. (2011). Carbothermic Reduction of Panzhihua Oxidized Ilmenite in a Microwave Field. ISIJ International Journal, 51, 337–343.

Pickles, C. A. (2009). Microwaves in extractive metallurgy: Part 1-A Review of Fundamental. *Minerals Engineering-Elsevier Journal*, 1102–1111.

Sandish, N., & Worner, H. K. (1996). Microwave Application in the Reduction of Mineral Oxides. *Journal of Microwave Power and Electromagnetic Energy*, 25, 177–180.

Srikant, S. S., Mukherjee, P. S., & Bhima Rao, R. (2011). Microwave reduction of placer ilmenite concentrate. *Mineral Processing Transaction,* 11(11), 1055–1062.