International Journal of Applied Science and Engineering
2013. 11, 3: 245-250

Effect of Heat Treatment in Microwave Furnace for Placer Ilmenite

Satya Sai Srikant[a,*], P. S. Mukherjee[b], and R. Bhima Rao[c]

[a] *Assistant Professor, Department of Electronics and Communication, SRM University, Modinagar, Ghaziabad, India*
[b] *Chief Scientist, Advance Material Technology, IMMT (CSIR Group) Bhubaneswar, Odisha, India*
[c] *Chief Scientist, Mineral Processing Department, IMMT (CSIR Group) Bhubaneswar, Odisha, India*

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 1000°C for 3 hour is also attempted. Carbon as low ash coke with 87.5% purity act as an additive was used for reduction of ilmenite at different time intervals using microwave sintering furnace. The results of these investigations indicate that at one minute of time the metallic iron formed contain 0.39% and of course no observation in mineral phases was noticed. At three minutes of duration of time, a partial phase transformation of ilmenite was observed and the metallic iron contain is 1.65%. Interestingly at above ten minutes duration of time a distinct metallic iron phase containing 35% metallic iron is seen in microwave furnace. Further studies are in progress.

Keywords: Microwave heating; electromagnetic; reductant; susceptor; ilmenite.

1. Introduction

Microwave radiation is electromagnetic radiation with frequencies in the range of 0.3-300 GHz and free space wavelengths of 1 m to 1 mm. Since microwaves obey the laws of optics, they can be transmitted, absorbed, refracted and reflected. The internationally recognized frequency bands, which are used for domestic and industrial applications are 2450 MHz and 915 MHz, respectively. Microwaves are the most complex heating source used in metal extraction since the heating process depends upon the multifaceted properties of electromagnetic fields of the microwave radiation with both the chemical and physical properties of the material. The factors which influence the interaction are the real and the imaginary permittivities (which are frequency dependent), the thermal conductivity, the heat capacity, the temperature, the geometry of the sample and the microwave cavity, the bulk density, the power level, the particle size, the sample mass or sample size, the presence of susceptors or coupling agents [1, 2] and the occurrence of chemical reactions or phase changes. Microwaves generate heat instantaneously

* Corresponding author; e-mail: satya.srikant@gmail.com

Received 22 June 2012
Revised 23 December 2012
Accepted 19 February 2013

and efficiently inside some materials, so that the thermal phenomena of conduction and convection play only indirect roles in the heating process, influencing only the heat loss factors of the temperature distribution calculation. In conventional heating, the heat is generated outside of the material (e.g. by a flame or a resistance heater) and is transferred into the material by conduction or convection. The surface of the object is heated first, and then the heat flows to the inside by conduction and thus the temperature decreases from the surface to the interior. On the other hand, microwave radiation penetrates into the object, where it is transformed into heat, which is conducted through the material back to the surface, leaving the outside cooler than the interior. The detail of the methods of heat generation is very occur through two mechanisms, namely (1) dipolar rotation, i.e. the rotations like motion of dipolar species in the presence of a high frequency electromagnetic field and (2) electron or ion AC resistivity i.e the resistive losses associated with the drift of ionic species under the same field. The former loss mechanism is associated with dielectric relaxation, while the latter is associated with the dielectric conductivity. At radio frequencies, ionic loss dominates, while at microwave frequencies it is the dipolar loss mechanism, which is more important. When the external fields are applied to atoms and molecules, the bound charges shift slightly relative to each other, creating electric dipoles. The short-range displacement of the charge results in a polarization phenomenon. In addition, as the electromagnetic fields change direction rapidly, all the polar molecules oscillate at the same rate, and as a result, the intermolecular "friction" generates heat. Any unbound charges (primarily electrons, but sometimes-free ions) produce resistive (ohmic) heating because the microwave field induces AC currents. Carbon particles (commonly act as the principal heat source when used as a reducing agent) have conduction electron loss as their primary heating mechanism. So the carbon being as a reducing or coupling agent, act as a susceptors in microwave oven where heat rapidly in an electric field. They conduct energy into the bulk sample so that electrons in the non-dielectric material can become more mobile. This extra mobility allows the formation of dipoles in the microwave field and the material can heat on its own [3, 4, 5].

2. Materials and methods

2.1. Raw materials

Beach placer Low grade ilmenite was recovered from red sediments of badland topography, Odisha. The ilmenite sample is 98% purity and on an average contains 47.3% TiO_2. Graphite 98.5% purity sample was used as an additive for reduction of ilmenite mineral.

2.2. Oxidization of ilmenite

The beach placer ilmenite sample from badland topography, Odisha, was first heated in a muffle furnace at 1000°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. Both the samples were kept separately for characterization and reduction. The microwave oven used was IFB model 38SC1, 50 Hz 850W.

2.3. Microwave heat treatment of ilmenite

Pre oxidized ilmenite by using furnace was again oxidized in a microwave oven for a minute.

After pre-oxidation, the ilmenite sample was mixed with a fine carbon powder (low ash fine coke carbon) having stoichiometric amount (i.e. 13 % of coke carbon) which needed for reduction process in microwave furnace. The coke carbon act as a reducing agents and it helps in heating the sample as it lost the dipole formation. The susceptor SiC placed near to sample further facilitate the microwave absorption and heating the sample in the microwave furnace. The schematic sketch for ilmenite sample in the microwave sintering furnace for heat treatment is shown in Figure 1. The microwave sintering furnace used for the present investigation was G N Tech, 6 KW, 2.45 GHz. A small amount of SiC powder was also mixed in the oxidized ilmenite sample in order to heat rapidly. The close photographs of heated sample were taken from four high magnified cameras interfaced with microwave furnace and personal computer. This sample was heated totally for 10 minutes and observed the metallic formation inside the furnace.

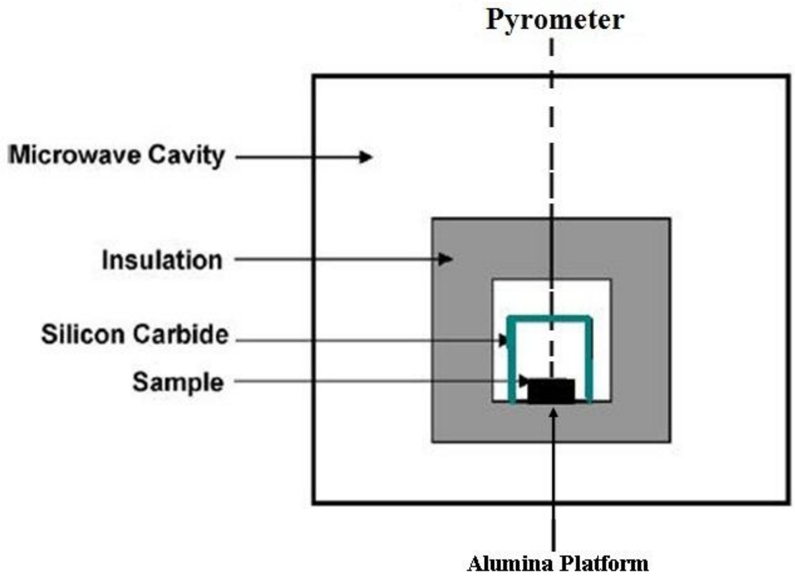

Figure 1. Schematic sketch for heating of ilmenite sample in a microwave furnace

2.4. Analytical methods

PANalytical X-Pert X-ray powder diffractometer (XRD) with Mo-Kα radiation (λ=0.709Ǻ) from 6° to 40° scanning angle at a scanning rate of 0.02°/sec was used for phase analysis of minerals and metals. XRD was used for complete chemical analysis of ilmenite and metallic iron analysis was carried out.

3. Results and Discussion

Effect of microwave heating on oxidized ilmenite with low grade carbon (coke) on formation of metallic phase is distinctly observed. The general megascopic pictures of microwave heated ilmenite grains are shown in Figure 2 and its close view of metallic phase is shown in Figure 3. This clearly shows that all the ilmenite grains are tending to fuse at first instance then converts for reduction process. 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 of course no observation in mineral phases was noticed. At three minutes of duration of time, a partial phase transformation of ilmenite was observed and the metallic iron contain is 1.65%. Interestingly at above ten minutes duration of time a distinct metallic iron phase containing 35% metallic iron is seen as in Figure 3.

Figure 2. General megascopic view of ilmenite mineral grains after microwave heating on oxidized ilmenite with coke carbon

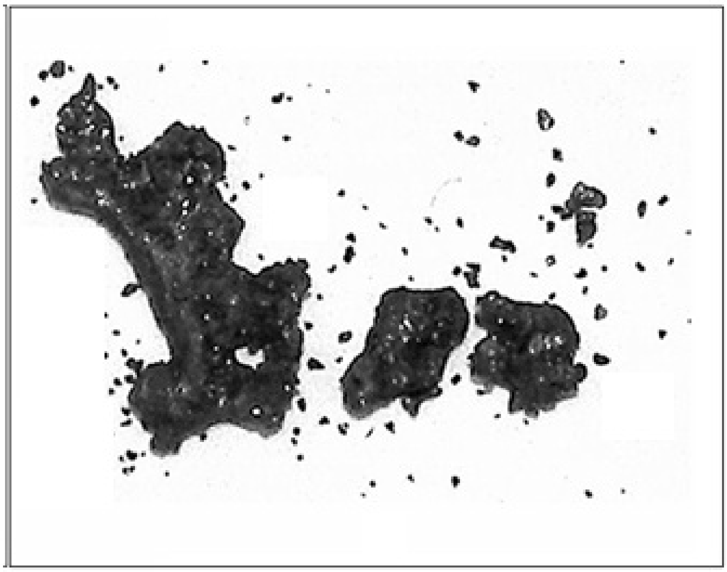

Figure 3. Close view of metallic phase ilmenite

The X-ray diffraction on oxidized ilmenite by using muffle furnace and in a microwave furnace are shown in Figure 4. The effect of microwave heating on oxidized ilmenite with carbon is also shown in Figure 5. The data indicate that the oxidization of ilmenite in both muffle furnace and in a microwave sintering furnace is similar with reference to mineral phase concern.

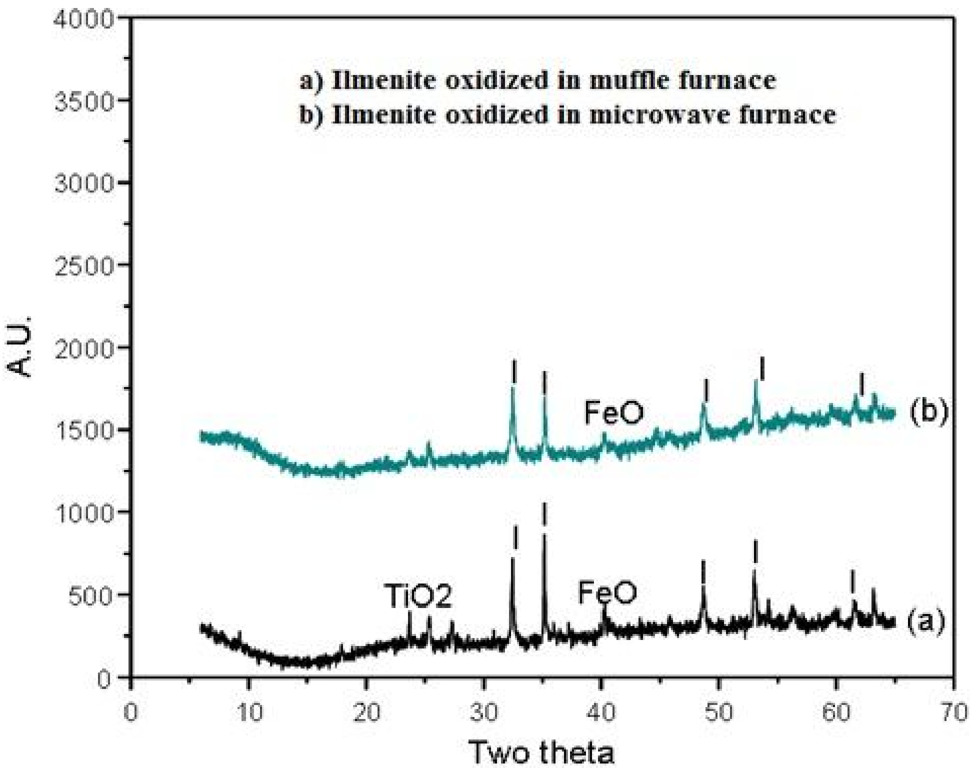

Figure 4. XRD patterns of ilmenite oxidized in muffle furnace and microwave furnace

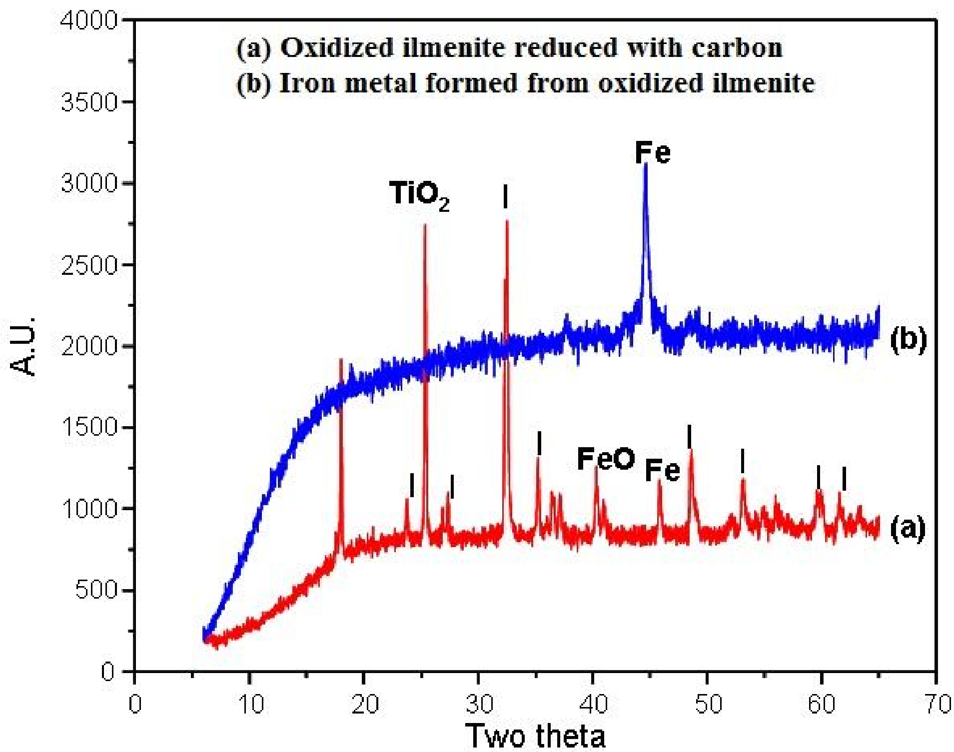

Figure 5. XRD patterns show the effect of microwave heating on oxidized ilmenite with C

4. Conclusion

Many factors have to be considered before employing microwave treatment for processing the placer ilmenite. The oxidization of ilmenite in a microwave oven and microwave sintering furnace gives the similar results as compared to muffle furnace with reference to mineral phase concern. The effect of microwave heat treatment with carbon seen on ilmenite mineral shows a clear metallic formation from ilmenite mineral. It is observed from our experimental results that the ilmenite is good in absorbs heat energy from the microwave heat energy and this effect is more significant in presence of susceptors and reductant. The microwave treatment of the placer ilmenite has more benefits as compared to conventional processing in terms of time saving, greater rapid with selective heating, thermal shock, eco-friendly, uniformity of heating process, process efficiency, environmental compatibility based on unique properties and characteristics.

Acknowledgements

Er. Satya Sai Srikant, is thankful to Prof. B.K. Mishra, Director of CSIR-IMMT for giving permission to utilize the laboratory facilities to carry out the research work. Thanks are also due to some of the scientists and research scholars of IMMT for their suggestions on the research work.

References

[1] Chen, T. T., Dutrizac, J. E., Haque K. E., Wyslouzil, W., and Kashyap, S. 1984. The Relative Transparency of Minerals to Microwave Radiation. *Canadian metallurgical quarterly*, 23, 1: 349-351.

[2] Bhima Rao, R. and Patnaik, N. 2004. Microwave Energy in Mineral Processing-a Review. *IE(I) Mineral Journal*, 84, 1: 56-61.

[3] Pickles, C. A. 2009. Microwaves in extractive metallurgy: Part 1-A Review of Fundamental. *Elsevier Journal*: doi:10.1016/j.mineng.2009.02.015, 1: 1102-1111.

[4] Pickles, C. A. 2009. Microwaves in extractive metallurgy: Part 1-A Review of Applications. *Elsevier Journal*: doi:10.1016/j.mineng.2009.02.014, 1: 1112-1118.

[5] Kelly, R. M. and Rawson, N. A. 1998. Microwave Reduction of oxidised ilmenite concentrates. *Minerals engineering Jr*, 11, 1: 1427-1438.

[6] Omer, Y. 2004. The effect of heat treatment on colemanite processing: a ceramics application. *Powder Technology Jr*, 142, 1: 7 -12.

[7] Haque, K. E.1999. Microwave energy for mineral treatment processes-a brief review. *International Journal of Mineral Processing*, 57, 1: 1-24.

[8] Xia, D. K. and Pickles, C. A. 1997. Applications of microwave energy in extractive metallurgy-a review, *CIM Bull*. 90,1: 96-107.