



Effect of Microwave Heat Treatment for Zircon Placer Mineral

Satya Sai Srikant^a, P S Mukherjee^b, R Bhima Rao^{c,*}

^aDepartment of Electronics and Communication Engineering SRM University, Modinagar, Ghaziabad, 201204, India

^bIMMT-CSIR, Bhubaneswar 751013, India

^cAryan Institute of Engineering and Technology, BPUT University, Bhubaneswar, Odisha, India

Article Info	Abstract
<p><i>Article history:</i> Received November 14, 2013 Accepted December 24, 2013 Available online February 13, 2014</p> <p><i>Keywords:</i> Zircon, Reducing agent, Susceptor Microwave energy, Ceramic material.</p>	<p>India is well-known for its higher zircon reserve in the world. The world trend obtaining the zircon products in form of zirconium metal and zirflor are direct interests through a process consisting with low power consumption, less time, clean and environmental friendly process. Microwave energy is one of the novel methods which satisfy the above conditions. This paper deals with an overall review of microwave applications on zircon minerals to produce value added products.</p> <p>This paper briefly overviews about the effect of microwave heat treatment on absorbance of heat energy of zircon mineral with and without susceptor. The effect of microwave heat treatment followed by thermal shock treatment of zircon mineral are also been reviewed.</p>

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1. Introduction

India, endowed with a coastline of over 6000 km, blessed with some of the largest and richest shoreline placer resources for zircon, ilmenite, sillimanite and with other minerals, which are confined to coastal stretch along the states of Odisha, Andhra Pradesh, Tamil Nadu, Kerala and Maharashtra. However, the concentration of these minerals varies from coast to coast which depend on the geological environmental sources. The high grade beach sand deposits which mostly consists of zircon, sillimanite and ilmenite are being exhausted and at the same time the lean and off grade deposits are also being exploited because of rapid growth of electric, electronic and mineral industries and high demand for industrialization. Hence, the demand on new resources has been increased for exploitation of placer heavy minerals specially ilmenite, sillimanite and zircon. Zircon is one of the important minerals that can be recovered from Red sediment of badlands topography which is the perennial resource to beach sand concentration [1- 6]. Researchers have been continuously attempting the recovery of zircon from beach placer and red sediments of badlands topography at various places across the world as it is very much useful applications in ceramics (54%) and refractory industries (14%), which account in India for 68% of zircon's total world consumption of 1.2 million tonnes. The rest (32%) is consumed in foundry, TV glass, zirconia chemicals and other applications [1]. Zircon chemicals and oxides are used in a wide range of environment friendly applications, as it provides a safe alternative to the use dangerous chemicals, such as formaldehyde resins in the paper industry, chrome in the leather industry and lead in the paint industry. The mineral finds its main applications as zirflor and opacifier. Many of the linings of smelter, chemical vats and heat treatment units in power plants employ zircon sands as an essential component. Coarser zircon sands are used as polishing agents at metal foundries and glass works. Zircon sands also serve as ammunition for some sandblasting machines used to strip paint and mould from building. Zircon is also used for welding purpose. This mineral is also used as gemstone and for radiographic dating, for orthopaedic

application etc. Zircon is mainly used as production of zirconium metal, other ferro alloys. Zirconium metal is used in nuclear reactors.

Microwave energy is relatively a new energy source and novel heating method, which plays an important role in materials and minerals processing because it has properties like selective heating, volumetric heating, rapid heating, environment-friendly and easy to control. However, the strengths of the interaction of the microwave heat treatment with the materials are more interest to Researchers as they found the interaction of microwave effects varied from material to material [2].

The present paper shows how the various researchers across the different parts of the world including the present authors had attempted / experimented and concluded the microwave heat treatment of different zircon samples. They also investigated to determine the advantages of microwave over the conventional heating process.

Microwave Heat Treatment on Zircon

Zircon is used for production of zirconia and zirconium chemicals. In all these industrial application, the heating process is required for zircon. The heat treatment through conventional muffle furnace method is well known process but at the same time, it has been noticed that there are lots of waste generated, capital investment, cost of production and huge power loss with adopting the conventional method. Researchers also attempted plasma process for heating as an alternative to the use of conventional high temperature furnace. However this process could not be scaled up yet for various reasons. However the research developments have suggested that microwave technology could provide a step change in value addition. Microwave energy has gained worldwide acceptance as a novel method for heating, sintering and phase transformation of minerals and materials, as it offers specific advantages in terms of speed, energy efficiency, process simplicity, novel and improved properties, finer microstructures, lower environmental hazards and eco-friendly method.

Several publications and patents are available in the literature on preparation of zirconia from zircon from conventional furnaces. Literature pertaining on this aspect using microwave heat source is very much restricted.

It was in the year 1984, Chen et al [7] published the publications of the most pioneering paper related to relative transparency of minerals to microwave heat treatment. He found that zircon (shown in Table 1) is transparent to microwave heat treatment when exposed to microwave frequency 2.45 GHz, power 150 W for 300 seconds in silicates class.

Table 1 Minerals transparent to microwave heat treatment, microwave frequency 2.45 GHz, power 150 W, exposure 300 sec (Chen, et al, 1984)

Mineral Class	Mineral / Compounds
Carbonates	Aragonite, calcite, dolomite, siderite
Jarosite-type	Argentojarosite, Zinc plant residues - synthetic natrojarosite/ plumbojarosite
Silicates	Almandine, allanite, anorthite, gadolinite, muscovite, potassium feldspar, quartz, titanite, zircon
Sulfates	Barite, gypsum
Others	Fergusonite, monazite, sphalerite (low-Fe), stibnite

The experiment carried out by Lasri and Schachter in 1998 shows the energy conversion in theoretical model during the ceramics sintering in the presence of susceptor. Zirconia (ZrO_2) as other low-loss materials couples very poorly to microwaves at low temperatures but since the loss increases with the temperature significantly, the coupling improves dramatically at high temperatures. In order to improve the heat transfer at low temperature SiC susceptor is introduced; its relatively high losses facilitate good coupling with the microwave field (2.45GHz). The authors experimented on consideration with three mechanisms in energy flow process: microwave absorption due to losses, black body radiation and heat convection. They showed that silicon carbide is heated rapidly when compared to zirconia, whereas zirconia reaches higher temperatures after a prolonged period of microwave exposure [8].

Ebadzadeh and Valefi in 2007 carried out the experiments for Zircon samples with microwave heating (2.45 GHz and 900 W) in a domestic oven between 4 and 60 min. They compared the microwave results compared to conventional heating (1300–1600 °C, holding for 120 min). They found that the microwave heating of zircon with susceptor showed a higher temperature for any heating time compared to those heated in the absence of susceptor. To reach almost the same

densification, microwave heating reduced the sintering time and temperature (16 minutes from room temperature to 1280 °C) compared to conventional heating (130 minutes from room temperature to 1300 °C and holding for 120 minutes at this temperature). The results also show that a long microwave heating (higher than 30 minutes) accelerates the decomposition of zircon. The extra phase formed acts a glassy phase during sintering of the samples; however, a lower average particle size (38.5%) was observed for the microwave-sintered samples.

However Rigopoulos et al. (2009) investigated about the carbothermal reduction of zircon to zirconia and silicon carbide over a range of reaction temperatures and dwell times with electric furnace. They found that zircon, was carbothermally reduced by using the reducing agent to produce ZrO₂ and SiC reaction products. They also found that with the increased yield of SiC and limited ZrC formation suggests that a 5hr dwell at 1500°C is a good compromise reaction condition conventionally for the cost effective production of ZrO₂/SiC particulate material [9].

Satya Sai Srikant et al. (2012) observed while carrying out the experiment for the studies on grinding of zircon in a planetary ball mill for production of zirflor and comparing the same with conventional methods [10]. They used the domestic microwave oven (IFB model, 2.45 GHz, 900 W) and microwave sintering furnace (GN Tech mwsinter, 6kW) for microwave heat treatment of heavy placer zircon minerals. They found that zircon mineral absorbs the heat from the conventional muffle furnace as well as from domestic microwave oven and microwave sintering furnace heat sources. They observed that the microwave heating of zircon with a susceptor showed a higher temperature for any heating time compared to zircon samples heated in the absence of the susceptor. The XRD data from their experiments indicate that zircon samples of natural and pre-heat treated shown the difference in the intensity of zircon mineral phase but not observed any phase change from zircon mineral such as zirconia and silica phases. It may be noted from the literature that the zircon samples subjected to microwave heating (2.45 GHz and 900 W) in a microwave oven between 4 and 60 minutes and heating in high temperature conventional muffle furnace (1300 – 1600 °C, holding for 120 minutes) exhibit the dissociation of zircon (ZrSiO₄ → ZrO₂ + SiO₂). The dissociation of zircon to zirconia and silicon has been accelerated in the microwave-sintered sample for 60 minutes heating. But they observed there were not any phase changes for zircon mineral to dissociate into zirconia and silica phases. They gave reason that the given restricted input power (1.5 kW) and time in microwave sintered furnace was not sufficient to dissociate the zircon mineral to zirconia and silica. The author also observed in the same experiments that the effect of grinding of zircon sample with and without heat treatment followed by thermal shock treatment reveals that the thermal shock treated sample contains numerous micro- cracks and fractures by which the grinding performance has been improved in a planetary ball mill. The grindability characteristics of the zircon samples, which have been heat treated using microwave sintering furnace as well as conventional muffle furnace followed by thermal shock treatment show a saving in grinding time over natural untreated zircon sample ground is 73.6%.

Result and Discussions

The effects of microwave heat treatment of zircon of various locations across the world were investigated by many researchers. The results indicate that zircon mineral absorbs the heat energy from microwave oven and microwave sintering furnace heat sources. The investigation also indicates that microwave heat treatment of zircon sample with a susceptor showed a higher temperature for any heating time as compared to zircon samples heated in the absence of the susceptor.

Conclusions

Although the quantity of research carried out for zircon heating with microwave technology is limited, the results published indicate that the future applications of microwave energy of zircon minerals in the mineral and ceramic industries can be encouraged as the microwave technology provides clean and eco-friendly process for heating and production of value added products from zircon sample. The heating of zircon sample with microwave energy offers specific advantages in terms of speed, energy / power efficiency, process simplicity, improved properties.

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