Journal of Magnetism and Magnetic Materials 378 (2015) 367–375

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Preparation and characterization of novel glass–ceramic tile with microwave absorption properties from iron ore tailings

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ARTICLE INFO

Article history:
Received 16 September 2014
Received in revised form
3 November 2014
Accepted 19 November 2014
Available online 22 November 2014

Keywords:
Glass ceramics
Magnetic properties
Cation distribution
Microwave absorption properties

ABSTRACT

A novel glass–ceramic tile consisting of one glass–ceramic layer (GC) attaining microwave absorption properties atop ceramic substrate was prepared through quench-heat treatment route derived from iron ore tailings (IOTs) and commercial raw materials (purity range 73–99%). X-ray diffraction (XRD), SEM, Energy dispersive X-ray spectroscopy (EDS), Fourier transform infrared spectroscopy (FTIR), Physical property measurement system (PPMS) and Vector network analyzer (VNA) measurements were carried out to investigate phase, microstructure, magnetic and microwave absorption aspects of the glass–ceramic layer. Roughly 80.6 ± 1.7 wt% borosilicate glass and 19.4 ± 1.7 wt% spinel ferrite with chemical formula of $(Zn^{2+}_{0.17}Fe^{3+}_{0.83})[Fe^{3+}_{1.17}Fe^{2+}_{0.06}Ni^{2+}_{0.77}]O_4$ were found among the tested samples. Absorption of Electromagnetic wave by 3 mm thick glass–ceramic layer at frequency of 2–18 GHz reached peak reflection loss (RL) of −17.61 dB (98.27% microwave absorption) at 10.31 GHz. Altering the thickness of the glass–ceramic layer can meet the requirements of different level of microwave absorption.

1. Introduction

The importance of steel industry is indubitable for the development of modern society. This is special true for new economy in developing countries such as China. Besides offering benefits for human activities, huge amount of iron ore tailings (IOTs) have since generated as rather hazardous byproducts during processing. Official statistics in 2008 showed that the annual discharge of IOTs in China alone was about 0.6 billion tons with less than 7% being recycled [1]. Untreated IOTs not only occupy valuable lands for storage, but also pollute water and damage eco-systems, causing potential threat to human safety [1].

Early attempts for proper treatments of IOTs began in the 1960 s with many prominent achievements being attained ever since, especially in construction and cement sectors [2]. Open literature suggested that IOTs can be converted into many types of glass–ceramic, including much valuable structural or functional materials [3–5]. The majority of those early works, however, were focused on how to enhance mechanical properties of final products for structural applications, while paid less efforts on their functional properties [3–5]. To the best of our knowledge, there is no open literature associated with microwave absorption properties of glass–ceramic derived from IOTs.

With the rapid development of gigahertz-scale electronic systems and telecommunications, indoor electromagnetic pollution is considered as one of those rather serious problems for our daily life [6]. Increasing numbers of associated base stations are becoming widespread sources of non ionizing electromagnetic radiation [7,8]. Some detrimental biological effects to human health may occur even at low-level microwave fields [8]. It has, therefore, long wished to develop novel cost-effective glass–ceramic tiles with microwave absorption properties that are suitable for residential construction applications. According to our previous work [9], this goal can be achieved via conventional glass–ceramic route (quench-heat treatment) using commercial raw materials at purity range of 73–99%. Most impurity elements presented in those raw materials can be locked into glassy matrix of final glass–ceramic tiles without apparent negative effects on microwave absorption performance of the tested samples [3,9]. Our work clearly indicated that it is possible to use IOTs to develop glass–ceramic tiles as microwave absorbers used in gigahertz frequency range.

The objective of the present work is to prepare glass–ceramic tile consisting of one glass–ceramic layer atop ceramic substrate using IOTs as main raw materials via quench-heat treatment route. It is our wish that the glass–ceramic layer will have microwave absorption properties in gigahertz frequency range. Its phases,

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http://dx.doi.org/10.1016/j.jmmm.2014.11.066
0304-8853/

morphology, magnetic properties, structure and microwave absorption properties will be examined accordingly.

2. Experimental

2.1. Raw materials

IOTs were fetched from one tailing reservation in Qingyuan of Guangdong Province, China. Weighted sample were oven dried at 105 ± 0.5 °C for 24 h and hand ground to pass through 60-mesh sieve for later use. Chemical composition of IOTs was analyzed with typical result listed in Table 1. Crystal phases were examined by powder X-ray diffraction technique (XRD, D/MAX 2550, Rigaku, Japan) with spectrum presented in Fig. 1a.

Other raw materials of glass–ceramic layer were provided by venders and listed in Table 2 with purity range of 73–99%.

Green ceramic substrates (porcelain tiles) about 14 mm in thickness were provided by Guangdong Bode Fine Building Material Co. Ltd. (China).

2.2. Preparation and characterization of glass–ceramic tile

According to our previous work and chemical composition of IOTs (listed in Table 1), dried and sieved sample of IOTs was mixed with other raw materials at weight ratio listed in Table 3 (utilization of IOTs is set to be 25.80 wt%). Nominal chemical composition of the mixture was calculated accordingly and presented in Table 4.

The above mixture was homogenized by ball-milling for 2 h prior to be placed into an alumina crucible and melted at 1500 °C for 1 h in bottom loading kiln (JGMT-8/300, Yixing, China). Frit was obtained by pouring the molten glass into tap water. After drying in an oven at 80 ± 0.5 °C for 2 h, the frit was sieved to get samples with median particle size of 2 mm for later use. A few grams of sieved frit were ground with agate mortar for powder XRD analysis (D/MAX 2550, Rigaku, Japan). Differential scanning calorimetry (DSC) of frit powder was also carried out on DSC-200F3 Maia analyzer (NETZSCH, German) in temperature range of 35–1200 °C at a heating rate of 20 °C/min in flowing air.

To obtain glass–ceramic tile (final product) of about 180 × 180 × 17 mm^3, sieved frit of 4–5 mm in thickness was placed on top of green ceramic substrate followed by heat treatment at 1200 °C for 15 min at heating rate of 20 °C/min and cooling inside furnace in high temperature electric oven (SX3-17, Xiangyi, China).

Glass–ceramic layer (GC) of the final product was cut for further analysis: (1) phases of GC powder were characterized by XRD (D/MAX 2550, Rigaku, Japan); (2) after polishing, bulk GC was etched in 2% HF solution for 15 s and coated by thin film of Pt for microstructure analysis by scanning electron microscopy (SEM, SIRION200, FEI, USA, at 20 kV) equipped with energy dispersive X-ray spectroscopy (EDS); (3) *M–H* hysteresis loop for GC at 10 K was measured by physical property measurement system (PPMS, Quantum Design, USA); (4) 1 mg GC powders were mixed with 200 mg solid KBr powders to make one pellet about 13 mm in diameter by hydraulic press for fourier transform infrared spectroscopy analysis (FTIR, Nicolet 6700, Thermo Electron Scientific Instruments, USA) in the range of 200–800 cm^{-1} at room

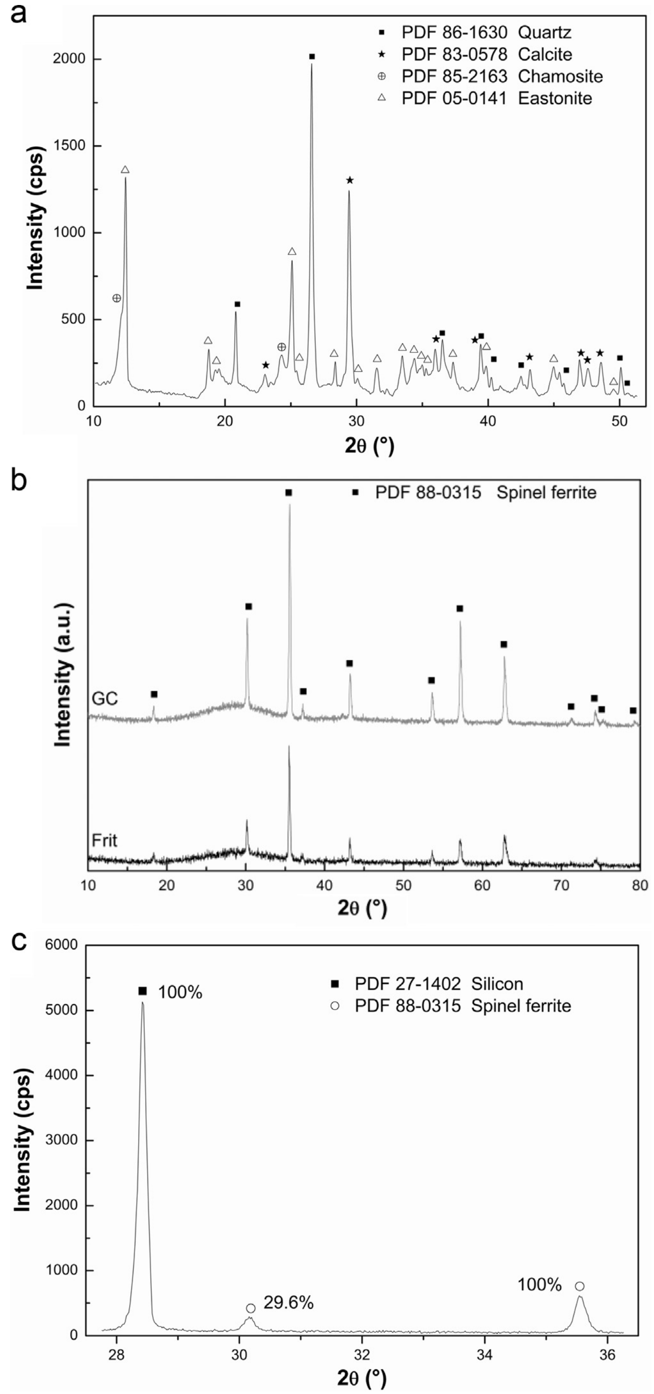

Fig. 1. XRD patterns of IOTs, frit and GC samples: (a) IOTs; (b) frit and GC; (c) mixture of 50 wt% Si + 50 wt% GC.

Table 2
Raw materials except iron ore tailing.

Compound	Purity(%)	Producer
Ferric oxide (Fe_2O_3)	98.8	Gaoshige Co. Ltd., China
Zinc oxide (ZnO)	99.0	Ruanfeng Co. Ltd., China
Nickel protoxide (NiO)	73.0	Gaoshige Co. Ltd., China
Barium carbonate ($BaCO_3$)	99.0	Xinji Co. Ltd., China
Potassium carbonate (K_2CO_3)	99.0	Wentong Co. Ltd., China
Borax ($Na_2B_4O_7 \cdot 10H_2O$)	99.0	Liaobang Co. Ltd., China

Table 1
Chemical composition of iron ore tailing.

Compound	SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	Na_2O	K_2O	LOI[a]
Content (wt%)	44.47	8.07	11.16	13.92	9.95	0.29	1.65	9.58

[a] LOI is short for loss on ignition.