



Structure, crystallization and dielectric resonances in 2–13 GHz of waste-derived glass-ceramic

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ABSTRACT

Structure, kinetics of crystallization, and dielectric resonances of waste-derived glass-ceramic prepared via quench-heating route were studied as a function of dosage of iron ore tailing (IOT) within 20–40 wt% using X-ray diffraction (XRD), fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS), and vector network analyzer (VNA) measurements. The glass-ceramic mainly consisted of ferrite crystals embedded in borosilicate glass matrix. Crystallization kinetics and morphologies of ferrite crystals as well as coordination transformation of boron between [BO₄] and [BO₃] in glass network were adjustable by changing the amount of IOT. Dielectric resonances in 6–13 GHz were found to be dominated by oscillations of Ca²⁺ cations in glass network with [SiO₄] units on their neighboring sites. Ni²⁺ ions made a small contribution to those resonances. Diopside formed when IOT exceeded 35 wt%, which led to weakening of the resonances.

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1. Introduction

Glass-ceramics (GCs), accidentally discovered in 1953, are inorganic materials featuring the presence of crystals, corresponding to one or more phases, embedded in glass matrix [1–4]. Among the impressive variety of GCs developed during the past six decades, waste-derived GCs are those of high scientific, ecologic and economic interest because of their potential to divert a wide range of industrial solid wastes away from landfill to marketable products in terms of 3R (reduce, reuse and recycle) [4–6]. The majority of early work on GCs made from wastes, however, focused on how to enhance their mechanical properties, regardless of their potential of functionalities beyond conventional structural uses [3,4,6–9]. The amazing accumulation of industrial wastes, especially in developing countries, together with growing threats of depletion of resources, has called for wider applications besides structural ones of these “green” GCs, with hope to reduce demands for natural resources and minimize possible undesirable environmental impacts from waste discharges [10–13]. To achieve this goal, novel and/or improved GCs from wastes need to meet more

requirements from various areas of human societies, among which those with functionalities are paramount [2,4].

Permittivity, or dielectric constant ϵ , is one measure of how easy it is to establish, or “permit”, an electric flux in materials [14], which is the key property of interest in electronic and telecommunication industries. Due to rapid development achieved in technologies for integrated circuit engineering, more and more products, from handheld devices to infrastructure equipment, are designed to function at as high frequency as possible [14,15]. To conform with this trend, exponentially growing attention has been drawn recently on permittivity in GHz range for numerous materials, such as tantalate and niobate based ceramics and glass-ceramics [16–21], titanate based ceramic composites [22–25], yttrium iron garnets [26,27], spinel and hexagonal ferrites [14,15,28,29], etc. Studies on glasses, glass-base composites (containing >50 wt% glass) and spinel ferrites indicated that their permittivity tends to keep constant near zero at frequencies beyond MHz range [30–34].

Some interesting phenomena, to our surprise, were observed in our previous work [35,36] that, for GC tile derived from iron ore tailing (IOT) consisting of 65–85 wt% glass matrix and 35–15 wt% spinel ferrite, multiple dielectric resonance peaks occurred in 6–13 GHz, which is far different from otherwise a constant almost independent of frequency for glasses and spinel ferrites as stated

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above. As a result, at frequencies without those resonance peaks (2–6 GHz), permittivity of the tile was small (within 7–10) accompanying by very low dielectric loss, which is suitable for microwave transmission; while in 6–13 GHz, the appearance of those resonance peaks leads to absorption of microwaves (maximum absorption reached –34.35 dB). Once strength and frequencies of those dielectric resonances become tunable as desired, the GCs from IOT can then be used to select and filter microwave signals at certain frequencies, which of value for many high-frequency applications [14,37–39], such as electromagnetic interference (EMI) suppressors, signal filters and microwave absorbers, etc.

To the best of our knowledge, however, few experimental data and investigations of relevancy on those abnormal dielectric resonance peaks in GCs are available up to date. Little is known about those phenomena and further studies are needed in order to better control those peaks and thereby make good use of them.

The objective of the present work is then to explore mechanisms behind those dielectric resonances by altering dosage of IOT within 20–40 wt% in our GC tile. Structure, crystallization kinetics and permittivity properties of the tile will be examined and discussed in detail.

2. Experimental

2.1. Raw materials

IOT powders, primarily consisting of SiO₂, CaO, Fe₂O₃, MgO and Al₂O₃, were sampled from one tailing reservation in Qingyuan City of Guangdong Province, China. Analysis of chemical composition of such IOT was conducted with results listed in Table 1.

Producers and purities of other starting chemicals, i.e., ferric oxide (Fe₂O₃), zinc oxide (ZnO), nickel protoxide (NiO), barium carbonate (BaCO₃), potassium carbonate (K₂CO₃), and borax (Na₂B₄O₇·10H₂O) were given in Ref. [35].

Green ceramic substrates (porcelain tiles) of 200 × 200 × 15 mm³ in size were provided by Guangdong Bode Fine Building Material Co. Ltd. (China).

2.2. Preparation

Molar ratio of all starting materials was kept constant except for IOT. 20.0, 25.0, 30.0, 35.0 and 40.0 wt% IOT were added respectively to make five mixtures. Weight percentages of all starting materials are tabulated in Table 2. These five mixtures were converted into frits by melting at 1500 °C for 1 h (furnace: JGMT-8/300, Yixing, China) and quenching into tap water. After washing, drying at 80 °C, and sieving to be about 2 mm in median particle size, frit samples were named as FT20, FT25, FT30, FT35 and FT40 respectively, where “F” stands for frit, “T” is short for tailing, and the digital numbers represent tailing content in wt%. Each frit sample of 4–5 mm in thickness was placed on top of one green ceramic substrate, followed by heat treatment at 1200 °C for 15 min (furnace: SX3-17, Xiangyi, China) and cooling inside furnace to attain one GC tile as final product. Top layer of each tile about 3 mm in thickness was cut into size of 10 × 10 × 3 mm³ as bulk GC samples for characterization. GC samples were denoted as T20, T25, T30, T35 and T40, respectively.

Table 1
Chemical composition of iron ore tailing (IOT) sample.

Compound	SiO ₂	MgO	Fe ₂ O ₃	CaO	Al ₂ O ₃	K ₂ O	Na ₂ O	LOI ^a
Content (wt.%)	45.24	16.05	13.85	10.40	4.72	0.25	0.11	8.98

^a LOI is short for loss on ignition at 1000 °C.

Table 2

Samples and their corresponding weight percentage of starting materials.

Sample		Starting materials (wt.%)						
Frit	Glass-ceramic	IOT ^a	Fe ₂ O ₃	NiO	ZnO	BaCO ₃	K ₂ CO ₃	Borax
FT20	T20	20.0	21.4	6.4	6.5	20.0	3.2	22.5
FT25	T25	25.0	20.1	6	6.1	18.8	3.0	21.0
FT30	T30	30.0	18.8	5.6	5.7	17.5	2.8	19.6
FT35	T35	35.0	17.4	5.2	5.3	16.3	2.6	18.2
FT40	T40	40.0	16.1	4.8	4.9	15.0	2.4	16.8

^a IOT is short for iron ore tailing.

2.3. Characterization

GC samples were pulverized for powder X-ray diffraction (XRD) measurement by D/MAX 2550 diffractometer (Rigaku, Japan) with Cu K α radiation ($\lambda = 0.154178$ nm) at room temperature to determine their phases. 25 wt% silicon powders (purity 99.995%, Tianjing Chemical Reagent Research Institute, China) were added into each testing samples as internal standard. Contents and lattice parameters of crystalline phases were obtained from XRD pattern fitting under the condition that residual error of fitting (R_f) was less than 7.50%.

1 mg of each GC sample after grinding was mixed with 200 mg solid KBr powders to make one pellet of about 13 mm in diameter by hydraulic press for fourier transform infrared (FTIR) spectroscopy analysis using Nicolet 6700 (Thermo Electron Scientific Instruments, USA) in range of 4000–400 cm^{−1} at room temperature.

Differential scanning calorimetry (DSC) of frit powders were carried out on DSC-200F3 Maia calorimeter (NETZSCH, German) in temperature range of 25–1100 °C (298–1373 K) under flowing air. Each sample was measured at five heating rates, i.e. 10, 15, 20, 25, 30 °C/min, to evaluate their kinetics of crystallization.

Scanning electron microscopic (SEM) investigation was performed by Quanta-200 (FEI, USA, at 20 kV) equipped with energy dispersive X-ray spectroscopy (EDS). Prior to SEM observation, samples were subjected to two sets of treatments: (1) to observe microstructure of as-prepared bulk GC samples, freshly fractured surfaces were polished, etched in 2 vol% HF for 15 s and coated by thin film of Au; and (2) to remove glass matrix and study morphologies of crystals inside, bulk GC samples were soaked in 40 vol % HF for 90 min, then filtered, washed by distilled water, dried at 80 °C and coated by Au layer.

Each GC sample was pulverized and dispersed into paraffin wax (CAS no. 8002-74-2, Sinopharm Chemical Reagent Co. Ltd., China) and compacted into ring of 7 mm in outer diameter, 3 mm in inner diameter and 3 mm in height for complex permittivity measurement over 2–13 GHz frequency range by vector network analyzer (VNA, AV3629, CETC41, China). Bulk GC sample T30 was soaked in 40 vol% HF for 90 min, then filtered, washed by distilled water, dried at 80 °C and selected by a Nd-Fe-B magne. The non-magnetic powders left, which are fragments of glass matrix according to XRD and SEM analysis in Sections 3.1 and 3.2, were used for complex permittivity measurement by repeating the above steps.

The valence state of Ni ions in glass matrix of T30 was investigated by X-ray photoelectron spectroscopy (XPS, K-Alpha 1063, Thermo Fisher Scientific, UK) equipped with a monochromatic Al K α source (1486.7 eV) with 0.3 eV full width at half-maximum. The spectra were calibrated with a corresponding measurement of the Au 4f_{7/2} level (84.0 eV) of a gold foil.

3. Results and discussion

3.1. Phase, structure and crystallization kinetics

Phases and their contents of GC samples T20–T40 were studied

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