

Glass-ceramics produced from thin-film transistor liquid-crystal display waste glass and blast oxygen furnace slag

Chen-Shiuan Fan, Kung-Cheh Li*

Graduate Institute of Environmental Engineering, National Taiwan University, 71, Chou-Shan Road, Taipei 106, Taiwan, ROC

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Abstract

This study employed waste glass from thin-film transistor liquid-crystal displays (TFT-LCD) and slag from a basic oxygen furnace (BOF) to produce $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ (CMAS) glass-ceramics through vitrification and further heat treatment of compacts of the obtained glass powders for densification and crystallization. CMAS glass-ceramics are known for their excellent mechanical and dielectric properties. MgO and Al_2O_3 were selected as modifying agents to ensure that the composition of the wastes featured these important characteristics.

Our results indicate that a TFT-LCD waste glass to BOF slag ratio of 7:3 yields a glass-ceramic with a primary crystalline phase of anorthite ($\text{Al}_2\text{CaSi}_2\text{O}_8$). The sample with 20 wt% MgO addition produced a glass-ceramic containing diopside ($\text{MgCaSi}_2\text{O}_6$), due to the presence of excess MgO. The densification of the sample with 20 wt% MgO saturated at 820 °C, resulting in a flexural strength of 140 MPa and dielectric constant in the range of 10–12 at 1 MHz, which satisfy the criteria for glass-ceramic insulation.

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1. Introduction

Thin film transistor liquid crystal displays (TFT-LCD) are a major display technology and the disposal of TFT-LCD waste glass has been widely discussed. Current processes employing waste TFT-LCD glass include its use as an additive to provide an adequate proportion of a liquid phase to increase the sinterability of ceramic tiles by reducing the sintering temperature [1]. Other studies have investigated its use as a partial substitute for cement in cement mortar [2,3].

Basic oxygen furnace (BOF) slag is a common byproduct of the steelmaking industry and has been traditionally used as a coarse aggregate in road construction. Nonetheless, the BOF slag material exhibits an undesirable tendency to expand due to the hydration of free CaO [4,5]. Free CaO is the free lime (CaO) content in BOF slag that has not yet undergone hydration, carbonation or stabilization within a crystalline phase. The free lime content in BOF slag can result in volumetric instability (expansion); therefore, it must be managed through appropriate

treatment (steel slag aging, testing or quality control) to ensure the appropriate use of the slag in construction or other recycling routes [6]. BOF slag can be used as an additive in cement [5] or hydraulic road binder [7] without the problem of expansion if the slag undergoes carbonation or hydration to transform the free CaO content into calcium carbonate (CaCO_3) or calcium hydroxide (Ca(OH)_2). However, the calcium hydroxide in cement can slowly react with carbon dioxide from the air to form calcium carbonate and the carbonation process is complex and time-consuming. Therefore, it is necessary to create another route for the disposal of BOF slag.

The use of industrial wastes as raw material in the preparation of glass-ceramics has been studied since the 1960s [8]. These various forms of waste include glass [9], the slag of ferrous materials [10,11] and non-ferrous materials (such as red mud from the aluminum industries) [12] as well as fly ash [13] and bottom ash [14]. Previous studies on the re-utilization of waste have mainly addressed the mechanical characteristics of the resulting ceramics or glass-ceramics. However, glass-ceramics in the $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ (CMAS) system are well studied for their excellent dielectric properties [15], making them excellent insulating materials. Therefore, in the current

*Corresponding author. Tel.: +886 2 2369 8112; fax: +886 2 2363 8173.

E-mail address: kcli@ntu.edu.tw (K.-C. Li).

study, BOF slag was mixed with TFT-LCD waste glass to examine the possibility of transforming such industrial wastes into glass-ceramics for use as insulating materials.

In general, the production of glass-ceramics is a two-stage process that involves the vitrification of a mixture of raw materials and a further heat treatment for densification and a further heat treatment for crystallization [8]. In the case of sintered glass-ceramics, the method involves melting the raw materials, quenching the melt to obtain a frit, and milling to obtain a fine powder that will later be compacted into a specific shape; a subsequent heat treatment for densification and crystallization follows. Waste glass from TFT-LCD screens can provide sufficient SiO_2 for the construction of an interconnected glass network during the vitrification process. BOF slag contains a substantial quantity of Ca^{2+} that can be incorporated within the Si–O structure during the vitrification process to act as a modifying agent to lower the sintering temperature of the resulting glass. Because of the vitrification, environmentally hazardous substances (chromium and vanadium) [16] and the free CaO within the BOF slag can be stabilized within the Si–O glass structure [17]. Therefore, the expansive characteristics of CaO can be nullified. This study selected analytical grade Al_2O_3 and MgO as additives to provide sufficient Al^{3+} and Mg^{2+} for the production of glass-ceramics in the CMAS system. The high Ca content in BOF slag can be expected to transform to the crystalline phase during the crystallization process, resulting in the production of anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$), a crystalline phase that is known for its excellent mechanical properties.

This study used TFT-LCD waste glass and BOF slag as raw materials for the preparation of glass-ceramics with good mechanical and dielectric characteristics through vitrification followed by a heat treatment, including densification and crystallization through sintering. We also investigated the effects of adding MgO and Al_2O_3 as modifying agents on the dielectric and mechanical properties of the resulting glass-ceramics.

2. Materials and methods

In this study, the waste glass and BOF slag were obtained from a TFT-LCD factory and a steelmaking company, respectively, in Taiwan. The chemical composition of the materials was analyzed using the wavelength-dispersive X-ray fluorescence analysis (WDXRF) (Advant'XP-397, Thermo ARL). The crystalline phases of the original waste and the resulting glass-ceramics were examined using X-ray diffraction (XRD) (X'Pert PRO, PANalytical) analysis with $\text{Cu K}\alpha$ radiation. The crystalline phases were identified through semi-automatic phase identification using the Match software package.

The optimal mixture ratio of wastes for the glass-ceramic production was determined by applying the cone test to investigate the softening temperature (S_T) of the frit obtained after vitrification of mixtures with different proportions of LCD glass and BOF slag, according to ASTM D1857-04. The selected ratio was then fixed as a reference to further study the effects of adding MgO and Al_2O_3 on the resulting glass-ceramics.

To ensure homogeneity in the melting process, mixture batches of 100 g were first mixed by dry milling in a planetary ball mill (PM 100, Retsch) at 150 rpm for 60 min, and then inductively melted in a 95% Al_2O_3 crucible at 1450 °C for 30 min in air using high-temperature electrical furnace. The milling jar and the balls were made of tungsten carbide (WC). The melt was quenched by pouring it into de-ionized water to form frits, which were subsequently dried at 105 °C for 24 h and again dry ball milled (300 rpm for 30 min) to obtain a fine glass powder with the required particle size ($< 75 \mu\text{m}$).

The glass powder underwent XRD analysis to ensure that no crystalline phase formed during the vitrification process. To characterize the differences in crystallization among the samples, the glass transition temperature (T_g) and the crystallization temperature (T_c) of each glass composition were determined using differential scanning calorimetry (DSC, SDT Q600, T.A Instruments) analysis, according to ASTM Standard D7426-08 and D3418-12. DSC analysis was performed at a heating rate of 10 °C/min in an alumina cell under an air atmosphere using powdered samples ($< 75 \mu\text{m}$) with analytical grade Al_2O_3 powder as a reference.

The heat treatment was performed on disc specimens with approximately 20 mm diameter and 3 mm height. The samples were obtained by pressing the initial glass powders into a cylindrical steel die at 80 MPa at room temperature using a uniaxial press (No. 3850, CARVER). The disc specimens underwent heat treatment for densification and crystallization at temperatures that ranged from each sample's glass transition temperature (T_g) to the temperature at which the specimen deformed. The temperature interval was 40 °C. The heat treatments were conducted at a heating rate of 10 °C/min, and the samples were held at the heat treatment temperature for 30 min. The developed crystalline phases were identified by XRD.

The porosity and bulk density were analyzed using the Archimedes principle according to ASTM A373-88. The flexural strength was determined according to ASTM C1499-09 standards. The dielectric properties (dielectric constants) were measured using an LCR [inductance (L), capacitance (C) and resistance (R)] meter (E4980A, Agilent) at 1 MHz.

3. Results and discussion

3.1. Fundamental analysis

Table 1 lists the chemical compositions of the LCD waste glass and BOF slag. The main chemical components of the waste glass were SiO_2 and Al_2O_3 . The SiO_2 was used to construct an interconnected network structure of glass in which to envelop the environmentally harmful substances and enhance the chemical stability of the glass-ceramics [18]. The Al_2O_3 can act as an intermediate oxide to participate in the random glass network [17]. The main chemical components of the BOF slag were CaO and Fe_2O_3 . As observed in Fig. 1, the TFT-LCD waste glass has an amorphous structure. Wüstite (FeO), brownmillerite ($\text{Ca}_2\text{Fe}_2\text{O}_5$) and lamite (Ca_2SiO_4) as well as SiO_2 and CaSi_2O_5 were identified in the BOF slag.

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