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CERAMICS INTERNATIONAL

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Some studies on nucleation, crystallization, microstructure and mechanical properties of mica glass-ceramics in the system $0.2BaO \cdot 0.8K_2O \cdot 4MgO \cdot Al_2O_3 \cdot 6SiO_2 \cdot 2MgF_2$

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Received 16 July 2012; received in revised form 31 August 2012; accepted 5 September 2012

Abstract

The crystallization, microstructure, microhardness and theoretical machinability have been investigated by DTA, XRD, SEM and Microhardness Indenter of resulting glass-ceramics. Two distinct crystallization exotherms in the DTA curve are observed and resolved. The first peak corresponds to the initial formation of potassium fluorophlogopite and the second is due to the formation of barium fluorophlogopite. The activation energy for precipitation of each crystalline phase has been evaluated, and the crystallization mechanism has been studied. DTA analyses were conducted at different heating rates and the activation energy was determined graphically from Kissinger and Ozawa equation. The average activation energy is calculated as 276 KJ/mol for the first and 366 KJ/mol for the second crystallization peak. The Avrami exponent for first and second crystallization peak temperature determined by Augis and Bennett method is found to be 3 and 3.9, respectively. The results indicate that the growth of mica is a two and three dimensional process, controlled by the crystal-glass interface reaction. The Vicker's hardness decreased steadily at intermediate heat treatment temperature with the formation of barium and potassium fluorophlogopite phase, but the decrease in hardness is more rapid at higher temperature with the development of an interconnected 'house of cards' microstructure.
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Keywords: B. Microstructure-final; D. Glass ceramics; Crystallization; Machinability

1. Introduction

Machinable glass-ceramics, wherein two-dimensional mica crystals are nucleated internally and crystallized from fluorine containing glasses have been developed [1], which can be machined to precise tolerances and surface finish with conventional metal working tools. Beall [1] first undertook the studies of the alkaline earth fluormica glass-ceramics. Later on Hoda and Beall [2] investigated different glass compositions containing barium, calcium, and strontium close in composition to the respective fluorophlogopite stoichiometry. Most of the compositions were susceptible to crystallization during casting. Only a few stoichiometric compositions were investigated and despite offering improved mechanical and dielectric strength, no further studies exist in the literature where compositional details are given. Henry and Hill [3,4], have shown that reduction in alumina content reduces the glass transition temperature, first crystallization peak temperature and promotes bulk crystal nucleation. The glasses with high alumina contents gave rise to feathery microstructures that did not coarsen readily to give blocky crystals of high aspect ratio and therefore could not produce the classic 'house of cards' microstructure. Hardness and machinability were found to be highly dependent on the formation of an interconnected 'house of cards' microstructure.

Greene et al. [5] investigated the $(1-Z)$ BaO: Z K_2O: (6–X) MgO: X MgF_2:(3–Q) Al_2O_3: Q B_2O_3: $8SiO_2$ system (where $Z=0$, 0.25, 0.5, 0.75 and 1.0, $X=2$, 2.5 and 3.0 and $Q=0$, 0.5 and 1) and observed that the substitution of

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0272-8842/$ - see front matter © 2012 Published by Elsevier Ltd and Techna Group S.r.l.
http://dx.doi.org/10.1016/j.ceramint.2012.09.015

Please cite this article as: A. Mallik, et al., Some studies on nucleation, crystallization, microstructure and mechanical properties of mica glass-ceramics in the system..., Ceramics International (2012), http://dx.doi.org/10.1016/j.ceramint.2012.09.015

barium by potassium results in increase in molar volume and co-efficient of thermal expansion and decrease in fractional glass compactness, microhardness and glass transition temperature values.

In the earlier work [6], authors had studied the kinetics as well as the crystal growth with respect to fluorine content in the barium fluorophlogopite glass-ceramics based on the system $BaO \cdot 4MgO \cdot Al_2O_3 \cdot 6SiO_2 \cdot 2MgF_2$, where it was indicated that the crystallization of the glass was largely homogenous and fluorine promotes initial crystallization.

Authors [7] had studied the kinetics as well as crystallization behavior, microstructure and mechanical properties of the system $Ba_x \cdot K_{1-2x} \cdot Mg_3 \cdot Al \cdot Si_3 \cdot O_{10} \cdot F_2$ ($x=0.0$, 0.3 and 0.5), where it was indicated that machinability and strength can be customized by judicious substitution of potassium by barium and also by the duration of heat treatment schedule.

It can be noted from above work that there is further scope of study for the improvement of machinability and hardness. In order to achieve the improvement of above properties, the present work has been undertaken wherein we have studied the crystallization, microstructure and mechanical behavior of $Ba_{0.1} \cdot K_{0.8} \cdot Mg_3 \cdot Al \cdot Si_3 \cdot O_{10} \cdot F_2$ glass.

2. Experimental

2.1. Glass synthesis and ceramisation

Analytical grade chemicals in powder form were used as starting materials in this study. The powders were from Merck Specialties Private Limited, India. The weight percentage chemical composition of the glass is BaO 3.59, K_2O 8.82, MgO 18.87, Al_2O_3 11.93, SiO_2 42.20, MgF_2 14.59. An overall 2.00 weight percentage of B_2O_3, was added purposefully to reduce the viscosity and thereof to increase the rate of diffusion of different ionic species in glass, which may result in the natural tendency towards directional growth of crystals [8]. The glass batches were properly mixed in an attrition mill thereafter melted in a platinum crucible for 4 to 5 h in an electrically heated furnace operating at 1450–1500 °C with occasional stirring with a glass rod (quartz) in order to achieve the homogeneity of the molten glass. After the melting operation is over, the molten mass was poured into a preheated cast iron mold to make rectangular slabs of dimension 50 mm × 25 mm × 10 mm. Immediately after casting, the prepared rectangular slabs of glass samples were released from the mold and were quickly introduced into an annealing furnace operating at 650 °C and soaked for 1 h followed by natural cooling to room temperature.

After annealing, the block was cut into pieces to about 1–2 mm thickness with the help of a precision low speed cutting machine (Buehler). These cut samples were fired at 680 °C for 2 h for nucleation. This nucleation temperature was determined by Differential Thermal Analysis (DTA) study. Samples after nucleation were then heated to various crystallization temperatures (800, 900, 1000, 1100 and 1150 °C) at a rate of 2 °C/min and the samples were kept at the crystallization temperatures for 5 h.

2.2. Characterization techniques

2.2.1. Differential thermal analysis (DTA)

The DTA measurements were performed using Shimadzu DT40 thermal analyzer with α-alumina powder as a reference material. In this work, non-isothermal experiments of finely powdered samples (~200 μm) of about 17 mg placed in a platinum crucible were carried out at different heating rates (5, 10, 15 and 20 °C/min.) from ambient temperature to 1000 °C. Analytical models developed independently by Kissinger and Ozawa were used to analyze the DTA data and to determine the activation energy for crystallization. Avrami exponent was calculated by Augis and Bennett's equation.

2.2.2. X-ray powder diffraction (XRD)

X-ray powder diffraction experiment was carried out for the samples heat treated according to the schedule as described above. The ceramised glass samples were ground to ~75 μm. XRD experiments were performed by an X-ray powder diffractometer (PW 1830, Panalytical) using Ni filtered Cu-k_α X-radiation with scanning speed of 2°(2θ)/ min. The diffraction pattern was recorded within Bragg's angle ranges $5° < 2\theta < 70°$. The phases were identified by JCPDS numbers (ICDD-PDF2 data base).

2.2.3. Scanning electron microscopy (SEM)

Different samples heated at the crystallization temperatures according to the schedule mentioned before were studied to investigate the development of microstructure with back scattered electron imaging (BEI) mode in a Hitachi, S3400N, Japan, scanning electron microscope. Before microstructural study, surface of all the samples were polished by following standard procedures and finally with diamond paste. The polished samples for scanning electron microscopy were prepared by etching for 1/2 min with an aqueous solution of 5% NaOH and 1% EDTA at about 80 °C.

2.2.4. Vicker's hardness

For this study, glass ceramic samples were mounted on resin and after curing, the samples were finely polished. The indenter was pressed onto the polished surface of the material at a load of 500 g with a dwelling time of 15 s.The size of the impression was measured using LEITZ microhardness indenter. The machine used was carver press. The Vicker's number (H_V) is calculated using the following formula [9]:

$$H_V = 1.854 \frac{P}{D^2} \quad (1)$$

where, P=applied Load (measured in kilogram-force) and; D=length of the diagonal of the indentation (measured in millimeter).

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