



Effect of the sintering temperature on the microstructure, properties and formation mechanism of ceramic materials obtained from polysiloxane elastomer-based ceramizable composites



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ARTICLE INFO

Article history:

Received 27 December 2015

Received in revised form

23 February 2016

Accepted 4 April 2016

Available online 6 April 2016

Keywords:

Ceramics

Elastomers

Sintering

Scanning electron microscopy

X-ray diffraction

ABSTRACT

Polysiloxane elastomer-based ceramizable composites can be used as insulation materials in wires and cables. These composites are transformed into hard ceramics in high-temperature environments to protect the inner copper. The sintering temperature is one of the key factors to determine the formation of ceramic protecting layers. The effect of sintering temperature on the weight loss, linear shrinkage and ceramic strength of the ceramic residue was investigated. The appearance of the ceramic residue was observed using a digital camera. The morphology of the impact fracture surface was observed by SEM. The element composition of the ceramic residue and their concentrations were revealed by EDS analysis, and the ceramic mechanism was revealed by XRD analysis. The results showed that the weight loss and linear shrinkage, as well as the flexural strength and impact strength of the ceramic residue, increased as the sintering temperature increased from 700 to 1100 °C. SEM analysis showed that the structure of the ceramic residue was converted from two phases to a uniform matrix with the increased sintering temperature. XRD analysis showed that quartz and MgSiO₃ crystals were produced in the ceramic residue at 800 °C and 1000 °C, respectively. Quartz and MgSiO₃ crystals are beneficial to the mechanical strength of the ceramic residue.

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1. Introduction

Recently, much attention has focused on polymer-ceramic composites because they exhibit dielectric properties [1], electro-mechanical properties [2] and flame retardant properties [3]. Among these composites, polymer-based ceramizable composites show special flame retardant properties for applications in wires and cables [3–5].

Ceramization is a procedure that ensures the compactness of polymer-based composites in the case of its thermal degradation caused by open fire or exposed to high temperatures. Ceramifying polymer composites ideally possess the dual performance characteristics of polymers at typical temperatures and ceramic residues at elevated temperatures [4,5]. Acting as some novel flame resistant

materials, ceramifying polymer composites, especially polysiloxane elastomer-based ceramizable composites, can be utilized in the cable industry with the goal of protecting copper wire in cables from fire melting. The composites exposed to flames and elevated temperatures create a ceramic phase that protects the wire and ensures electric integration of the circuit up to 120 min [6].

In the case of polysiloxane elastomer-based ceramizable composites, the Si–O backbone in polysiloxane chains endows the polymer with various properties. High bond strength gives them sufficient stability for high temperature applications and for use as precursors of ceramic residue [7]. Consequently, the polysiloxane elastomer, especially the most popular polydimethylsiloxane (PDMS), is the best polymeric family for manufacturing ceramizable composites [8,9].

The ceramization effect of the polysiloxane elastomer composites is achieved by adhering together mineral particles (e.g., mica [5–8], quartz [9], alumina [10], long fibers [11], wollastonite [12] and montmorillonite [13]) dispersed in a polymer matrix in

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different processes, generally in the presence of fluxing agents [14–18]. The fluxing agents (glass frits, zinc or ferric oxide, zinc borate, etc.) are melted at a specific temperature to produce the liquid phase, and they limit the escape of its volatile products [19]. Consequently, the sintering temperature has a significant effect on the ceramizable properties of ceramic residues. However, if ceramization occurs at a low temperature (550–800 °C), then maintaining the durability and shape preservation of the ceramic residue is difficult because of the low strength of the ceramics [20].

Some research has been performed on the effect of the sintering temperature. For example, Mansouri et al. [16] investigated the properties of ceramic polysiloxane elastomer at different sintering temperatures. The results showed that the flexural strength of the ceramic residue was lower at 600 °C than at 1000 °C. Pędzich [21] found that the ceramization conditions could strongly influence its final microstructure, such as the porosity, pore size distribution and ceramizable body compactness. The samples fired slowly up to 1000 °C showed a compact microstructure. However, the microstructures of samples fired rapidly to 1000 °C were much more porous.

Although there are many works on the transformation of silicone compositions into ceramic residue, the effect of sintering temperature on the strength and morphology of ceramic residue has not been discussed in detail.

In the present work, the effect of sintering temperature (700, 800, 900, 1000 and 1100 °C) on the microstructure of ceramic materials obtained from polysiloxane elastomer-based composites is reported using various technologies, including SEM, EDS and XRD analysis. The effects of sintering temperature on the weight loss, linear shrinkage, flexural strength and impact strength of polysiloxane elastomer-based composites were investigated.

2. Experimental methods

Silicone gum compounds are summarized in Table 1. All of the compounds were prepared on a two-roll mill with a gear ratio of 1:1.4 at room temperature. The polysiloxane elastomer (Shenri 110-2, China) was first softened, and then, fumed silica (Cabot M-5), mica and glass frits were added until a homogenous batch was obtained. The curing agent 2,5-dimethyl-2,5-bis(tert butyl peroxy) hexane (DBPMH) was then added and processed until a visually good dispersion was achieved. The polysiloxane elastomer compounds were cured with flat sheets by compression molding in a hydraulic press at 170 °C for 15 min under 15 MPa pressure. The post curing of samples was carried out at 180 °C for 2 h.

The ceramifying of flat sheet samples (50 mm long, 4 mm thick and 6 mm wide) was performed using a muffle furnace (Shenjia KL-12B, China). The samples were heated from room temperature to the various target temperatures (700, 800, 900, 1000, 1100 °C) at a heating rate of 10 °C min⁻¹, held for 30 min, and then cooled with the muffle furnace.

The flexural strength of pyrolysis samples was determined by a flexural method using a universal testing machine (Zwick Roell Z010) with a cross-head speed of 0.5 mm min⁻¹ according to ASTM D790-10. The impact strength was evaluated on an electronic charpy impact tester (Suns, China) according to ASTM D256-10e1.

The weight loss of the ceramic residue was calculated according to Eq. (1) where W is the weight loss, and m_1 and m_2 are the mass of the sample before and after sintering, respectively.

$$W = (m_1 - m_2)/m_1 \times 100\% \quad (1)$$

Shrinkage of the ceramic residue was measured by heating flat sheet samples to 600, 700, 800, 900, 1000, and 1100 °C for 30 min at each temperature in a muffle furnace. The linear shrinkage of the ceramic residue was calculated according to Eq. (2) where L is the linear shrinkage, and l_1 and l_2 are the length of the samples before and after firing, respectively.

$$L = (l_1 - l_2)/l_1 \times 100\% \quad (2)$$

The surfaces of mica and glass frits and the impact fracture surface of the ceramic residue were observed by scanning electron microscopy (SEM, Zeiss EVO18). The element composition and concentration of the ceramic residue on the impact fracture surface were detected using an energy dispersive X-ray spectroscope (EDS, Oxford X-Max). X-ray diffraction spectra of the mica, glass frit and ceramic residue were obtained using an X-ray diffractometer (XRD, PANalytical X'pert). Each scan was conducted from a 2 θ angle of 5°–90° at a scan rate of 1°·min⁻¹.

3. Results and discussion

3.1. Weight loss and linear shrinkage of ceramic residue

The effect of sintering temperature on weight mass and linear shrinkage of the ceramic polysiloxane elastomer is shown in Fig. 1. The weight loss increases from 48.6% to 50.2% as the sintering temperature increases from 700 °C to 1100 °C due to the increased pyrolysis of the polysiloxane elastomer at higher temperatures. The linear shrinkage increases from -1.4% to 4.0%; it is unusual that the value of the linear shrinkage is negative, which indicates bloating of the ceramic residue fired at 700 °C. When the sintering

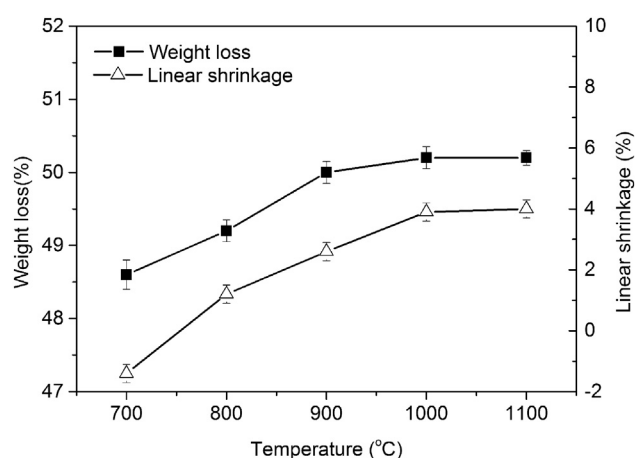


Fig. 1. Effect of sintering temperature on weight loss and linear shrinkage of the ceramic polysiloxane elastomer.

Table 1
Basic formulation of ceramic polysiloxane elastomer.

Components	Polysiloxane elastomer	Fumed silica	Mica	Glass frit	DBPMH	Hydroxyl silicone oil
Content(phr ^a)	100	30	50	18	2	2.5

^a Parts per hundreds of rubber.

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