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Prohibition of radiation-induced cross-linking of silica-reinforced silicone foam by oxygenation with H_2O_2

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ABSTRACT

Prohibition of radiation-induced cross-linking of silica-reinforced silicone foam by oxygenation with hydroxyl radicals generated from radiolysis of H_2O_2 solution was investigated. The elongation at break of samples irradiated under air decreases monotonically with irradiation, from a control value of 100 ± 6.0% to 32.3 ± 0.1% for samples exposed with a dose of 500 kGy. Compared to samples irradiated in air, the elongation at break of samples irradiated in H_2O_2 solution shows of a less decrease while that in H_2O shows more of a decrease at the same dose. Our results indicate that radiolysis of H_2O_2 can weaken the increase of crosslink density of silicone foam, while H_2O promotes the increase of crosslink density of silicone foam compared to that in air. The property and morphology changes of silicone foam are probably attributed to the irradiation induced cross-linking and radiolysis, the possible mechanisms for which are proposed.

1. Introduction

Silicone rubbers are synthetic polymers with silicon and oxygen in their backbones that exhibit excellent performance, including a wide service temperature range (Zeigher and Fearon, 1990), superior chemical resistance (Samuel and Steven, 1987), excellent insulation (Meyer et al., 2004; Yoshimura et al., 1999) and low toxicity, and have been extensively used in multiple fields (Breiner and Mark, 1998; Diao et al., 2011; Wei et al., 2002). These useful properties have been further expanded by the addition of silica as a reinforcing agent[33, 34]. When silicone foams are exposed to gamma rays, the polymers generally undergo chemical changes and their performance inevitably deteriorates (Hanisch et al., 1987; Traeger and Castongu.Tt, 1966). The high-energy rays or particles cause the formation of free radicals that then lead to crosslinking or radiolysis, influencing the property and lifetime of the materials (Gillen et al., 1996; Ito, 2005; Patel and Skinner, 2001; Wilski, 1987). Therefore, it is essential to investigate the changes of the chemical structure and macroscopic properties of silicone rubber under complicated harsh environmental conditions.

The effect of radiation on silicone rubbers has been widely studied (Basfar, 1997; Burnay, 2001; Huh et al., 2000; Maxwell et al., 2003; McGrath et al., 2002; Seguchi et al., 2011; Warrick, 1955; Wu et al., 1983; Zhang et al., 2006), including the influences of adsorbed dose, radiation temperature, and radiation atmosphere on the performance of silicone foams (Assink et al., 2001; Chen et al., 2015; Ito et al., 2009; Labouriau et al., 2015b, 2007; Sui et al., 2014). It was found that radiation-induced cross-linking of polymer chains was one of the most important factors leading to the quick degeneration of the performance of silicone foam during the radiation process (Barnes et al., 1959; Hill et al., 2001; Miller, 1960; Seguchi et al., 1981; Sui et al., 2014). Oxidizing species, such as O_2, play an important role during the radiolysis of polymer materials. The radiolysis of silica-reinforced silicone foam in high pure nitrogen showed that the foam hardening occurs and crystallization of polymer matrix decreased with increasing dose in nitrogen compared with that of in air (Liu et al., 2018, 2017). Miller (1961) previously found that the cross-linking density of PDMS in nitrogen after radiation was 3 times that of in oxygen in 1961. Labouriau et al. (2015a) studied the oxidative degradation of a silica-filled silicone elastomer and found that the elastomer can produce long time-life peroxide radical after irradiation in oxygen, and the increment of the crosslinking density of PDMS in the presence of oxygen is less than that in nitrogen. Therefore, discovering how to prohibit the occurrence of radiation-induced cross-linking is of great importance for improving the life-time of silicone foam during practical radiation condition.

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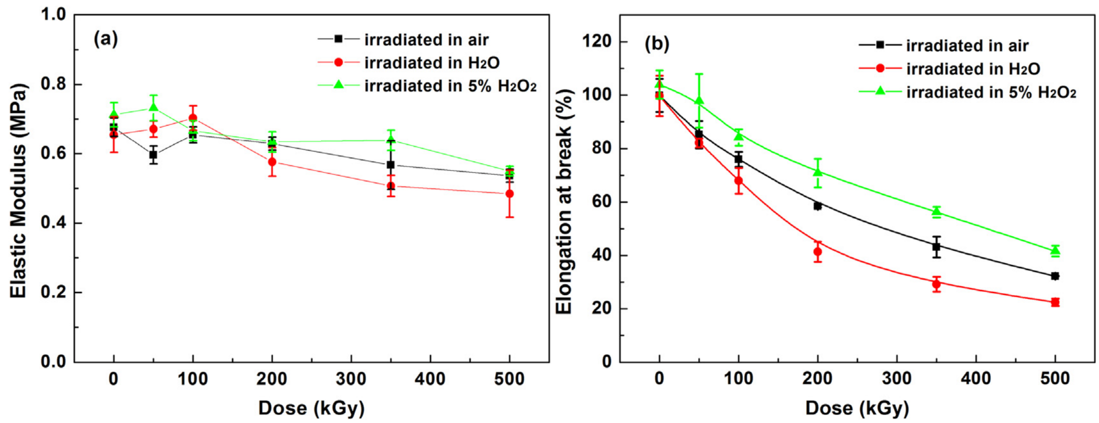

Fig. 1. Mechanical properties of silicone foam with various absorbed dose. (a). Elastic modulus; (b). Elongation at break.

However, there has been little work focused on the prohibition of the radiation-induced cross-linking mechanism of silicone foam. It is well known that the formation of radiation-induced active radicals on the side of polymer chains is the essential reason for the cross-linking of polymer chains. If these active radicals were captured or decreased their activity, the performance of polymer materials under radiation conditions would be improved greatly.

In this study, silica-reinforced silicone foam was exposed to gamma rays under H_2O_2 solutions for the first time. The effect of radiation on the microstructural, thermal and mechanical properties, and swelling degree of silicone foam was evaluated. For comparison purposes, the influence of pure H_2O on the radiation-induced cross-linking of silica-reinforced silicone foam was also studied. Finally, we found that oxygenation can improve the performance of silicone foam and the possible mechanism responsible for these performance changes was proposed.

2. Experimental section

2.1. Materials

The silicone foam used in this study with chemical structure of methyl vinyl silicone was prepared as reported in previous publications and reinforced by silica (Shi et al., 2008, 2007). The gel fraction of this silicone foam is determined at near 95–97%.

2.2. Irradiation

Silicone foam samples were irradiated in air (298 ± 4 K), pure water and 5% H_2O_2 solution using a ^{60}Co source with an average dose rate of ca. 110 Gy min^{-1} at Institute of Nuclear Physics and Chemistry, China Academy of Engineering Physics, China. The pure water was purged with high purity nitrogen for clearing away soluble O_2 in water. The sample was put into glass tube with size of diameter 20 mm × length 200 mm. The amount of liquid is ca. 30 mL and the mass of silica-reinforced silicone foam is near 2 g, and the solid sample was fixed and always kept under liquid level avoiding to contact air. The dose rate was measured by Fricke dosimeter which was placed at sample position. The dose rate was measured by Fricke dosimeter which was placed at sample position.

2.3. Characterization

2.3.1. Mechanical properties testing

Sample sheets were 2 mm in thickness and were cut into dumbbell specimens. Mechanical properties test was performed with using a SANS CMT7000 testing machine, fitted with a 100 N load cell, at a crosshead of 50 mm/min. For each composition, three samples were tested for reproducibility. The testing curves and corresponding data, including elasticity modulus and elongation at break, were recorded.

2.3.2. Hardness Testing

The hardness of the samples was determined using Shore durometer OU2700 (Cangzhou OUPU Testing Instrument Co. Ltd). Each sample was tested for three times, and three samples for each composition were tested for reproducibility.

2.3.3. DSC

DSC experiments were recorded on TA Instruments Q200 DSC equipped with a liquid nitrogen cooling system. The samples were placed in the aluminium pans to test thermal properties. The first cool-heat cycle was used to erase thermal history of the samples (cooled from 0 °C to −160 °C at 10 °C/min, and kept at −160 °C for 2 min, then heated to 0 °C at 10 °C/min, and kept at 0 °C for 2 min). The second cool-heat cycle was recorded for data analysing (cooled from 0 °C to −160 °C at 5 °C/min, and kept at −160 °C for 2 min, then heated to 0 °C at 5 °C/min).

2.3.4. SEM

The microstructures of the samples were characterized using a ZEISS EVO 18 special edition scanning electron microscope at acceleration voltage of 10 kV. The surface of the samples was characterized for comparison.

2.3.5. ATR-FTIR

The samples were recorded on a Thermo Scientific Micro Fourier transform infrared spectrometer (ATR-FTIR). ATR-FTIR spectra were acquired over a range from 650 to 4000 cm^{-1}.

3. Results and discussion

The effect of radiation on silica-reinforced silicone foam with oxygenation by H_2O_2 was investigated, and the influence of air or pure H_2O on radiation-induced cross-linking of silica-reinforced silicone foam was also analyzed separately. Crosslink density is an important parameter for polymer and has a close relationship with their mechanical properties, such as the elastic modulus, elongation and hardness. The mechanical properties of silicone foam under different irradiation conditions were determined and are shown in Fig. 1. The elastic modulus of irradiated samples increases slightly with dose level, eventually decreasing with extreme exposure. This observed result is in accordance with the literature (Chen et al., 2015). The elongation at break of samples irradiated under air decreases monotonically with irradiation, from a control value of 100 ± 6.0% to 32.3 ± 0.1% for samples exposed with a dose of 500 kGy. Compared to samples irradiated in air, the elongation at break of samples irradiated in H_2O_2 shows of a less decrease (41.6 ± 2.0% at 500 kGy) while that in H_2O shows more of a

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