



Physical properties of elastomer composites with scintillating additives

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ABSTRACT

Well-designed radiation detectors reconcile high detection yield with preserving optical, mechanical and thermal properties over time. This work summarizes thermal, mechanical, and optical properties of three different elastomer based potential scintillators (PDMS, ASP and KER) doped with up to 5% PPO/POPOP. Properties of doped and undoped samples were assessed using digital scanning calorimetry, quasistatic tensile and compressive tests and spectrophotometry. It was found that all elastomers are thermally stable up to 220 °C and remain unaffected by the inclusion of the dopant. Their stiffness is < 1 MPa and is minimally affected by doping, with the exception of PDMS. In terms of optical properties, KER and ASP based composites lose 10% and 70% of their transmittance from the undoped to 5% doped state at a wavelength of 420 nm whereas PDMS based composite is completely opaque after doping. Phenyl-type elastomers may possess some of the critical combination of physical properties necessary in designing light-weight, flexible radiation detectors.

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1. Introduction

Scintillators luminesce when struck by ionizing radiation, a property that makes them excellent radiation detectors used in medical, energy and defense fields [1–5]. Over time, natural occurring scintillating materials have been found to suffer from failure due to brittleness or environmental degradation [6,7]. Recent work has focused on fabrication of composite scintillators with the aim of limiting environmental degradation while enhancing ductility [8,9]. To this end, polymer based scintillator matrices composed of poly(styrene) (PS) and/or polyvinyltoluene (PVT) have been used [10–12]. Their fabrication protocol extends over a period of 3–5 days at temperatures between 70–130 °C with exposure to inert atmosphere. These types of PS based scintillators are fairly stable; however, under certain radiation conditions, aging will occur leading to reduced light yield. Furthermore, exposure to solvent vapors, high humidity, operating temperatures greater than 80–100 °C, or even mechanical loading can accelerate the deterioration processes [13], which can adversely affect light yield.

Elastomers have low specific modulus (i.e., they are very compliant) and can be stretched to more than 400% their original size and still return back to their original dimensions [14]. These prop-

erties, coupled with their inertness and chemical stability, make them excellent materials for numerous applications including medical devices [15,16], advanced electromechanical devices [17,18], flexible UV photodetectors [19], and sensors [20–22]. Due to their high compliance, a thorough understanding of the underlying failure mechanisms may be necessary [23] especially if fabricating sensors from thin compliant films. A large body of work has been dedicated in incorporating additives to enhance physical properties of elastomers [24–27]. Recent studies have shown that some elastomers also possess desirable properties that could prove beneficial in solving many issues with current scintillating materials including ability to preserve their transparency even after 10 Mrad dose exposure [28], compared to 1–3 Mrad for Polyvinyltoluene(PVT) and Polystyrene (PS) composite scintillators [29] as well as the ability of elastomers to intrinsically relax to non-equilibrium conformations and avoid issues such as crazing/micro-cracks that are common in other type of plastic scintillators (e.g. PVT) [11,30,31].

In elastomeric scintillator design, a key property is the elastomer's optical transparency, since transfer of light to sensors is crucially important [32–34]. Higher scintillator dopant concentrations may be advantageous from the point of view of detection. However, for most silicone rubbers, there are material limits to the concentration of scintillating dopant that they can contain while maintaining good optical properties. In addition to high optical transparency, composite scintillators should also be stable (under radiation, loading, environment) and easy to manufacture [35].

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In this study, we examine three elastomer composites, evaluating their viability as scintillating materials by concurrently measuring three key properties. We note that it is advantageous for newer scintillators to possess multifunctional properties so they can be used as both structural and radiation detection components in future devices such as unmanned aerial vehicles [36]. The present investigation focuses on optical, mechanical and thermal properties, with radiation detection performance left for a future study. Two of the investigated formulations, which are based on phenyl rubbers from Shin-Etsu Silicones of America, are specifically designed to have excellent optical properties (e.g. high transmittance >80%) for a large range of wavelengths since they are commonly used in fabrication light-emitting diode (LED) assemblies. KER and ASP are newly available commercial formulations that were identified based on prior work highlighting that increasing phenyl content in an elastomer can increase radiation hardness after exposure to ion beam induced luminescence [29]. The phenyl rubber properties are compared to polydimethyl siloxane (PDMS), a well characterized elastomer [37–41], with known behavior during irradiation [42,43].

The organization of the paper is as follows: Section 2 describes the experimental protocol describing synthesis and method of doping of the elastomers as well as details on the physical property characterization. Section 3 describes a summary of the results and discusses the implications. Section 4 is a summary of the presented work.

2. Experimental protocol

2.1. Material synthesis

Three different elastomers are used in this work: polydimethyl siloxane (PDMS), Sylgard® 184 from Dow Corning, and Phenyl Rubbers, (KER-6000 A/B) and (ASP-1120 A/B) from Shin-Etsu. For PDMS, we combined the two-part mix in a 10:1 ratio of polymer base to curing agent, and cured the mixture at 70 °C for 5 h. KER-6000 and ASP-1120 involved mixing the two-part mix (A and B) in a 1:1 ratio and cured at 80 °C in air for <20 h. All samples were degassed, prior to curing. The elastomers were cured at low temperature to preserve ABS 3D printed molds, which could not be heated beyond ~85 °C. It is possible to cure these elastomers at higher temperature, e.g. 100–150 °C, for substantially less time. Curing temperature can impact mechanical properties such that the stiffness of PDMS can range from 750 KPa to as high as 3MPa [37–39]. To our knowledge, no studies have reported information that suggests a dependence of optical properties on curing temperature.

Elastomers were doped with a mixture of PPO and POPOP dissolved in toluene. 2,5-diphenyloxazole (PPO, primary dopant) and 1,4-bis(5-phenyloxazol-2-yl) benzene (POPOP, secondary dopant) in a 50:1 mass ratio of PPO to POPOP. This PPO/POPOP mixture was dissolved in a small amount of toluene (<100 µL), which aids dissolution in the elastomer precursor. Doped samples of each elastomer were created by incorporating the necessary mass of the toluene mixture to the uncured elastomer of measured mass. The amount of the combined dopant incorporated into the elastomers ranged from 0% (the undoped control case) up to 5% by mass. The mass of the dopant mixture added was equivalent to the relevant fraction of the mass of the uncured elastomer, so that ~1% of the mass was used in the ~1% doped samples and ~5% in the 5% doped samples. Such a scintillator is often used to enhance light yield e.g. under x-ray irradiation of polymer composites [44]. The optical, thermal and mechanical properties of doped and undoped elastomers were obtained.

Following their preparation, the elastomer composites were cured in molds of 3D printed ABS as well as cylindrical Teflon molds

attached to a smooth, detachable base. Fused deposition modelling (FDM) was used with ABS plastic, to generate the unique geometry required for tensile testing of samples. An example of the 3D printed samples is shown in the Supplementary Material section Fig. SM1. The walls ensured that the cured elastomers took a desired shape, while the detachable base allowed for easy release with minimal distortion of the elastomer and mold interface during the release process.

Fig. 1 shows images of the tensile sample encased in the 3D printed mold before (Fig. 1a) and after (Fig. 1b) release demonstrating good shape preservation. A UV-illuminated doped sample, (5% doped KER-6000 pictured), released from the tensile mold is shown in Fig. 1c. Fig. 1d shows the preserved flexibility of the doped elastomer after bending. We note that the KER sample of Fig. 1d was not used in mechanical testing.

2.2. Material characterization

The optical, thermal and mechanical properties of doped and undoped samples were obtained as follows.

2.2.1. Optical characterization

An ultraviolet-visible (UV-vis) spectrophotometer (the Evolution 220 system from Thermo Scientific) for a wavelength range of 200 nm to 800 nm with a 1 nm step was used to measure the light transmittance, or percentage of incident light of a given wavelength that passes through each sample. The change in light intensity is normalized to provide a consistent estimate of optical properties, independent of sample thickness. The normalization employs Beer's law, where the transmittance value at a certain wavelength can be expressed in terms of the absorbance at that wavelength, which in turn can be expressed in terms of the sample thickness. By normalizing the absorbance by the thickness, we can obtain a transmittance expressed in terms of a reference thickness as:

$$\%T_n = \frac{100}{10^{A_{norm}}} \quad (1)$$

In this equation, A_{norm} represents the normalized absorbance, which is expressed per unit thickness, and $\%T_n$ is the normalized transmittance, calculated for our reference thickness. The absorbance provides an avenue to express the transmittance in terms of the sample thickness. This is done by obtaining the molar absorptivity ϵ from the thickness and absorbance as:

$$A = \epsilon bc \quad (2)$$

where

$$\epsilon = A_{norm} = \frac{A}{b} = \frac{1}{b} \log_{10} \frac{100}{T} \quad (3)$$

and b is the thickness, c is the concentration of particles in the sample/medium, and ϵ the molar absorptivity. Assuming the absorptivity and concentration are constant, a normalized absorbance, independent of the thickness, can thus be calculated and used to estimate the transmittance for other thicknesses.

2.2.2. Mechanical Characterization

An important effect of doping is the potential change in mechanical properties, specifically the modulus or strength. Hence, for each of the elastomer composites, quasistatic uniaxial compression and tensile tests are conducted. Mechanical tests were performed with a Kammrath and Weiss tension/compression stage with a 5 kN load cell. The compressive samples are cubes with an average length of 12.7 mm and cross-sectional area of ~70mm². The tensile samples are standard dogbone samples with a total sample length of 40 mm and a testing region of 8 mm × 5 mm × 13.7 mm in size (complete dimensions shown in Fig. SM 1). Compression samples

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