



High-strain shape memory polymers as practical dry adhesives

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A B S T R A C T

The adhesive benefits of incorporating shape memory polymers (SMPs) in various dry adhesive designs have been well documented in recent years. Chemically-crosslinked SMPs can possess remarkable shape fixity and recovery ability, chemical and thermal stability, a sharp glass transition, and other desirable qualities. However, low failure strain and a relatively rigid rubbery state limit their performance as a dry adhesive component in many practical applications. This work explores the adhesive benefits of using an epoxy-based SMP which has been modified to possess a softer rubbery state and a greatly increased maximum strain before failure. One of the modified SMPs forms the active adhesive surface of a prototype consumer level device.

1. Introduction

Substantial research in the area of dry adhesives has been conducted in recent years, yielding many significant discoveries and producing a wide variety of strategies for improving adhesive performance [1]. Despite the steady progress, notable limitations remain in the current state of the art examples which have thus far limited their use. The use of shape memory polymers (SMPs) as a functional material in dry adhesives has been previously explored [2–7], where its dynamic rigidity and ability to passively recover its shape were found to provide enhanced adhesive strength, reduce the required preload to bond, and enable very easy removal when desired (good reversibility). Whether as a structural layer or as a direct-contact adhesive, the current body of work primarily utilizes epoxy-based SMPs which possess many desirable characteristics including excellent shape fixity and recovery, thermo-mechanical stability, and ease of processing. Less desirable are their tendencies to remain stiff in their rubbery state relative to most pressure sensitive adhesives (PSAs), and their susceptibility to tearing under moderate strains. A soft rubbery state is desirable to facilitate adhesive bonding by reducing the required preload, while large recoverable strains are desirable to enable greater flexibility in the adhesives' structural design and to improve general durability.

A recent study has demonstrated a successful modification of an oft-used epoxy SMP formula, wherein the authors used a heavier (longer) epoxy monomer and greatly increased the concentration of the curing, or crosslinking, agent [8]. The resulting SMP was found to be both softer in its rubbery state and substantially more stretchable, while retaining its superlative shape memory properties. The differences in the SMP's properties are directly attributed to a decrease in crosslinking

density, which allows the polymer chains greater freedom of movement in the polymer's rubbery state. The chains therefore may rearrange more readily when subjected to stress, aligning in the direction of the stress more completely and with less resistance. The present work seeks to investigate the effects of these modifications to the epoxy SMP formula regarding dry adhesive performance, and to produce a prototype practical dry adhesive using similar formulations described herein. The effect of increased curing agent and increased monomer chain length are investigated independently through the production of several SMP formulations. These SMPs are tested with regard to their dry adhesive performance, and to confirm that they possess excellent mechanical and shape memory properties on par with those previously reported, for example in Ref. [8].

2. Materials and methods

2.1. SMP materials and sample fabrication

The SMPs used in this work were created from a mixture of epoxy monomer and curing agent Jeffamine D230 (poly(propylene glycol)bis(2-aminopropyl) ether; Huntsman), hereafter referred to as Jeffamine. In general, the epoxy monomer was comprised of a mixture of two chemically similar products with different average molecular weights: EPON 826 obtained from Momentive (molecular weight, $M_n \sim 362 \text{ g mol}^{-1}$), and Poly(Bisphenol A-co-epichlorohydrin), glycidyl end-capped obtained from Sigma-Aldrich (molecular weight, $M_n \sim 1075 \text{ g mol}^{-1}$), hereafter referred to as E1075. Intermediate epoxy monomer weights, determined on a molar basis, were created by mixing EPON 826 and E1075 in ratios provided in Table 1. The

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Table 1
Component mixing proportions for the high-strain SMPs.

Formulation	Epoxy M_n (g/mol)	Molar proportion			Weight proportion		
		EPON 826	E1075	Jeffamine	EPON 826	E1075	Jeffamine
E362-J100	361.9	1.000	–	1.000	1.000	–	0.636
E362-J075				0.752	1.000	–	0.478
E362-J060				0.599	1.000	–	0.381
E362-J050				0.500	1.000	–	0.318
E448-J100	447.6	0.899	0.101	1.000	1.000	0.334	0.707
E448-J075				0.752	1.000	0.334	0.531
E448-J060				0.599	1.000	0.334	0.423
E448-J050				0.500	1.000	0.334	0.353
E533-J100	533.3	0.760	0.240	1.000	1.000	0.940	0.837
E533-J075				0.752	1.000	0.940	0.629
E533-J060				0.599	1.000	0.940	0.501
E533-J050				0.500	1.000	0.940	0.418

proportion of Jeffamine used for each SMP formulation is likewise included in Table 1, on both molar and weight bases. Formulation names are derived from the average molecular weight of the epoxy monomer mixture, followed by the ratio of Jeffamine to epoxy monomer mixture. E1075 and EPON 826 were combined in a glass container, heated to 120 °C in order to completely melt the E1075, then thoroughly mixed by manual stirring while still at 120 °C. Room temperature Jeffamine was then stirred into the epoxy mixture. Test samples for dynamic mechanical analysis (DMA) and adhesive blister testing were created by pouring the resulting mixture onto glass plates and curing for several minutes at 100 °C until the precursor becomes viscous enough to spread into a uniform and thin sheet. Curing was completed in an oven at 100 °C for 90 min. Samples were cut from the resulting sheets using a laser cutter, then removed from the glass at elevated temperature.

The formula for NGDE2 has been previously described [9], while in this work the curing procedure and sample preparation are the same as described above for the high-strain formulations.

2.2. DMA testing

A Q800 DMA from TA Instruments (New Castle, DE) was used to perform all SMP mechanical analysis, failure strain measurements, and SMP fixity and recovery factor measurements. Laser-cut samples of rectangular cross section approximately 1.75 mm wide and 0.5 mm thick were used. Storage modulus data is reported at 1 Hz excitation, with data collection at discrete temperature intervals where the chamber was allowed to equilibrate for four minutes. Failure strain data was collected in uniaxial tension by ramping tensile force on each sample at a rate of 1 N min⁻¹ until failure. Engineering stress and strain are reported, based on the test sample's initial length and cross sectional area. Shape fixity and recovery factors were determined according to the definitions provided in Ref. [10] for a single shape fixing and recovery cycle.

2.3. Blister adhesion testing

Thin (~ 0.50 mm) circular samples of several SMP formulations were bonded to form a seal over the 1 cm diameter circular glass opening of a pressure chamber by first heating the glass surface to 75 °C, followed by light pressure to ensure SMP-glass contact and maintaining 75 °C for three minutes. Temperature was controlled by an AGPtek® universal digital PID temperature controller. Tests were performed after allowing the sample to cool to the desired temperature. The flexibility of the thin SMP membranes allowed them to self-conform to the glass adherend when heated, obviating the need for preload during the bonding process. Pressure was applied through a manual

regulating system, increasing pressure by ~ 70 kPa min⁻¹ until interfacial failure was observed to have initiated.

2.4. Practical adhesive demonstration

Nichrome wire with 0.62 mm diameter was obtained from Sigma Aldrich. 3D printed parts were fabricated using an EOS Formiga P100 selective laser sintering machine, using PA 2200 polyamide powder. The power source is a 7.4 V lithium polymer battery.

3. Results and discussion

Plots of storage modulus versus temperature for each of the SMP formulations listed in Table 1 are provided in Fig. 1. The data indicate the effects of increasing Jeffamine concentration are very similar to those reported in Ref. [8], decreasing the glass transition temperature (T_g) and rubbery state storage modulus (E_r) while maintaining a similar glassy state storage modulus. Comparing the E448-J050 and E448-J100 formulations, for example, shows a decrease in E_r by a factor of eight from 16 MPa to 2 MPa by doubling the Jeffamine concentration. These observations indicate that the expected reduction in crosslinking density was achieved. The flat rubbery and glassy plateaus, together with the narrow T_g band, suggest the presence of strong shape memory performance in each of the twelve formulations. Similarly to Ref. [8], changing the proportion of Jeffamine has a strong effect on the material's T_g , particularly as the concentration becomes large. This is clearly evident in E362 and E448 series tests where for example E362-J050 and E362-J060 are nearly the same, whereas E362-J060, E362-J075, and E362-J100 are substantially different, though significant and nearly uniform changes to T_g are observed between each variant of the E533 series of SMP. Varying the average molecular weight of the epoxy base has little effect on the shape and character of the modulus curves, except a noticeable reduction in rubbery state modulus for the higher-weight formulations. Among those formulations with the highest Jeffamine concentration, E362-J100, E448-J100, and E533-J100, the storage modulus decreases with increasing epoxy base molecular weight from 4 MPa to 2 MPa and 1.7 MPa, respectively.

The shape memory performance of E533-J100 was tested, with results depicted in Fig. 2a. Both fixity and recovery factors of > 99% were found for a 6% peak strain, showing that even the formulation with the lowest crosslinking density shows excellent shape memory properties. Recovery at larger strains was tested using E448-J100. The first ten cycles of ~ 100% strain with temperature held constant at 50 °C shown in Fig. 2b. The first cycle appears to show some irrecoverable strain, which is partially due to the slower, more viscous response of the SMP at relatively low temperature not allowing complete recovery during

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