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Low-stress creep deformation in two opto-electronic glass-epoxy joints: Part I – adhesive creep data

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A R T I C L E I N F O	A B S T R A C T
Keywords: Epoxy Adhesive Creep Model Opto-electronic Low stress	This two-part paper presents measurements and modelling of creep deformation at relatively low stress of two epoxy adhesives used in opto-electronic applications, one having a relatively high modulus and the other a low modulus. Part I describes the bulk tensile creep tests at various temperatures (75–95 °C) and stress levels representative of those in opto-electronic devices. A novel laser-based method of non-contact creep strain measurement was used to eliminate errors associated with adhesive specimen contact. The high modulus epoxy, tested at stress levels between 5 and 20 MPa, displayed non-linear behavior at high stress levels but was linear over the range of interest. The low modulus adhesive, tested between 0.25 and 0.5 MPa, was linearly viscoelastic over the full range of tested stress levels. Part II assesses the accuracy of finite element models to predict the creep deformation in two different glass-epoxy joints designed to represent stress states in some opto-electronic applications.

1. Introduction

Opto-electronic devices use glass optical elements such as lenses and mirrors that are often bonded to various substrates. This is normally done using adhesives that can be subject to creep due to external loads and residual stresses. Even very small perturbations in the position of the optical elements can be detrimental to the performance of the device. Residual stresses are generated during the adhesive cure, particularly if it is at elevated temperature and the optical element and substrate have different coefficients of thermal expansion [1]. These cure stresses are typically of greater significance in adhesives with a higher tensile modulus. Creep due to external loads tends to be of greater concern than residual stress in adhesives having a lower modulus or a glass transition temperature (T_g) below the service temperature [2]. Design tools are needed to predict the viscoelastic behavior of opto-electronic adhesive joints under the conditions of relatively low stress that are typical of application environments.

The vast majority of published epoxy creep studies have been conducted using levels of applied stress typically greater than about 3.5 MPa, which is relatively high compared to the stresses that are encountered in opto-electronic applications of adhesives. Consequently, little is known about the accuracy of creep models at the low stresses typical of opto-electronic applications. Many creep models have been proposed in the literature, with the more complex models having larger numbers of parameters to be determined, but able to capture greater variations in the creep behavior according to the material and the loading conditions. However, no single creep model can represent all types of materials and all loading conditions. From an engineering perspective, it is usually desirable to identify the simplest possible creep model that provides adequate accuracy while having the fewest parameters to be determined.

The viscoelastic behavior of a wide range of materials can be represented using networks of springs and dampers [3]. Maxwell elements, consisting of a spring and a damper in series, and Kelvin-Voigt (KV) elements, consisting of a spring and damper in parallel, are commonly used. These models generally relate the strain to either the Von Mises equivalent stress or, in cases where the hydrostatic stress influences failure, the Stassi equivalent stress [3], thereby allowing treatment of complex stress states. Del Nobile et al. [4] found that an integral form of the generalized Maxwell model, consisting of a spring and several Maxwell elements all in parallel, could describe the creep deformation of various types of low-modulus food at low compressive stress levels. Hu et al. [5] used strain gages to measure creep strains in cylindrical epoxy specimens loaded at stress levels between 11 and 64 MPa in various stress states. They saw that the Stassi equivalent strains were proportional to the applied loads at low stress levels, however the stress-creep strain-rate relationship became non-linear as the applied stress exceeded 22.8 MPa. Xia et al. [6] then modelled these

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data using a generalized KV model, consisting of a spring and several KV elements all in series. They found that such standard viscoelastic models could be used to relate complex stress states to creep strain, and that by allowing the model parameters to vary with the applied stress, non-linear viscoelastic materials could be modeled. Feng et al. [7] found that the coupling model of Ngai et al. [8] could describe the creep behavior of epoxy adhesives at relatively low stress in the linear viscoelastic range (7.5 MPa). The authors used a linear variable displacement transducer (LVDT) integrated between the specimen grips to monitor strain without contacting the gage section of the specimen. According to Ngai et al. [8] the shear creep compliance of some polymeric materials can be divided into recoverable and non-recoverable components when operating near the material T_g . The model requires four experimentally-determined parameters: Young's modulus, the equilibrium compliance, a coupling parameter and an apparent relaxation time.

It is known that strain gauges can reinforce relatively flexible materials, such as cast adhesives, and distort strain measurements, particularly at low stress levels with relatively thin specimens; therefore, non-contact methods of measuring strain were preferred for the present work [9].

The objective of the first part of this two-part paper was to measure the tensile creep properties of two opto-electronic epoxy adhesives over a range of temperatures and relatively low stresses. Testing was done between 5 and 20 MPa for the high-modulus material and between 0.25 and 0.5 MPa for the low-modulus material, at temperatures between 75–95 °C. The accuracy of various creep models was then assessed. These data were then used in the finite element models of Part II [10] in order to predict the creep deformation of two opto-electronic glassepoxy adhesive joints.

2. Methods and materials

2.1. Adhesives

Epoxy A was a two-part, relatively high-modulus, high T_g epoxy used in fiber optics, electronics and medical applications. Epoxy B was a two-part, much more flexible adhesive also used in fiber-optic applications. Both adhesives were shipped premixed in frozen syringes. Table 1 compares the two epoxies in terms of the manufacturer's properties as provided by Lumentum. As explained below, the creep models used measured values of the relevant properties rather than the manufacturer's data of Table 1.

2.2. Bulk adhesive creep testing

2.2.1. Creep test specimens

Specimens for both adhesives were made in accordance with ISO 15166 [11]. The adhesives were first dispensed in a single bead (about 2 mL for epoxy A and 3mL for epoxy B) on a polished aluminum plate ($50 \times 100 \times 12 \text{ mm}$), coated with a mold-release agent (Miller-Stephenson MS-122E), and then degassed in a vacuum oven at a gauge pressure of approximately -100 kPa at room temperature for 10 min. A second coated aluminum plate was then clamped in place to press the adhesive into a sheet with the thickness controlled using polytetra-fluoroethylene shims. The sheets of epoxy A were 0.5 mm thick and

Table 1

Adhesive properties as provided by Lumentum.

Property	Epoxy A	Ероху В
Glass transition temperature (°C)	110	0
Estimated tensile modulus (MPa)	2000	2
Poisson's Ratio	0.4	0.49
Density (g/cm ³)	1.2	1.1
Coefficient of thermal expansion (mm/mm °C)	55×10^{-6}	200 $ imes$ 10^{-6}

cured in a preheated oven at 120 °C for 1 h, including the heat-up time (~45 min). A precision cut-off saw with a water-cooled diamond blade was used to cut 6×75 mm rectangular specimens from the cast sheet of epoxy A. Epoxy B was cast into 0.8 mm thick sheets which were cured at 85 °C for 1 h in a preheated oven, after which the oven temperature was increased to 120°C and the specimen was removed after 1 h. A thermocouple embedded in the adhesive layer was used to establish the required ramp up and dwell times. A custom-made die cutter and a hydraulic press were used to cut the rectangular specimens from the cast sheet of the relatively soft epoxy B.

An average cross-sectional area was calculated from the cross-sectional dimensions measured at 3 points along the 40 mm gage length. The degassing and curing procedures produced specimens that were usually free of significant voids, and if voids were present they were scattered and widely-spaced. Occasionally, a void might create a stress concentration that would lead to premature failure. To identify such voids, each specimen was inspected visually by shining a bright fiberoptic light (NCL 150, Volpi MFG, Auburn, NY) through the cast epoxy sheet.

2.2.2. Creep test apparatus and strain measurement

The relatively low loads and flexible adhesives in the present study made it highly advantageous to avoid contact methods of strain measurement such as strain gages or extensometers. These methods of measuring strain can lead to errors due to loading and reinforcement of the specimen along its gauge length [9]. Consequently, a laser displacement sensor (Model LK-G157, Keyence Inc., Itasca, USA), with an accuracy of \pm 8 µm, was used to measure the strain during the tensile creep tests of both epoxies. As shown in Fig. 1, reflectors of different height, attached with spring clips on either end of the gage length, provided the required displacement measurements. Knife edges were positioned at least 9mm away from the grips. In order to prevent the reflector knife edge from slipping on epoxy A, these specimens were gently scribed at each end of the gage length. This was unnecessary for the softer epoxy B.

The laser was mounted on a micrometer stage to measure the distances to the four measurement tabs in sequence, and to repeatedly return to the same position for each cycle of measurements during a creep test. One major advantage of this measurement system is that a single laser displacement sensor could be used to track the deformation of several specimens by moving the sensor between loading setups. The laser response was found to be quite sensitive to temperature, and so it was cooled by a fan and left for 1 h to reach thermal equilibrium before measurements were taken. The average of the strain measurements on the two sides was taken to be the specimen strain. This method amplified the effects of minute specimen rotations since the laser was reflected at distances *a* and *b* from the specimen neutral axis, as shown in Fig. 1. The measured laser gage-length displacements on either side of the specimen, δ_R and δ_L , were corrected to give the actual bending strain, ϵ_B , using the equation:

$$\epsilon_B = \left(\frac{\delta_R - \delta_L}{2}\right) \left(1 + \frac{a+b}{h}\right)^{-1} \tag{1}$$

where *h* is the specimen thickness. As will be seen below, the actual bending strains, ϵ_B , were negligibly small. The standard deviation of gauge length measurements including error due to repositioning the laser was experimentally determined from 10 measurements of a fixed gage length. The standard deviation was determined to be 7.65×10^{-3} mm, which led to a standard deviation in strain measurements of 0.027%.

Light-weight miniature wedge grips with serrated gripping surfaces (Mark-10 Corp., Copiague, NY, USA) were used to mount the epoxy A specimens to the loading frames, while two-part bolted clamp grips with sand paper on their faces were used to load the free ends of the specimens as shown in Fig. 2. The laser extensometer was positioned

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