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Test method

The ratio of flexural strength to uniaxial tensile strength in bulk epoxy resin polymeric materials

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ABSTRACT

The flexural behavior of epoxies was investigated by performing mechanical tests and applying statistical Weibull theory and analytical methods to the results. The effects of loading systems and environmental conditions were also considered. Three kinds of epoxies were studied: Epon E863, PRI 2002, and PR520. In total, 53 three-point-bending (3PB) Epon E863 samples and 26 3PB PR520 were tested immediately after curing, together with 26 four-point-bending (4PB) PRI2002 samples stored at 60°C and 90% Rh for 48 weeks. The Weibull parameters were estimated using both linear regression and the moments method. The statistical character of the Weibull model leads to uncertainty in the evaluated parameters, even for a large number of experiments. This study analyzed the ratio of flexural strength to tensile strength in bulk epoxy resin polymers. An analytical method previously developed by the authors to study the relationship between uniaxial tension/compression stress-strain curves and flexural load-deflection response was used to obtain the ratio. The results show that the Weibull model overpredicted the aforementioned ratio in different load arrangements.

1. Introduction

Epoxy resin polymeric materials are one of the most important classes of thermosetting polymers and are becoming increasingly popular for domestic and industrial uses. They are used as adhesively bonded joints for structural members in different applications, such as civil infrastructure (e.g., pipe coatings) and transport (e.g., aerospace and automotive industries). They are also extensively used in dentistry and medical orthopedic prostheses. Despite their wide range of potential applications, the use of epoxy resin polymers is still limited due to their variable mechanical properties. In addition, these highly cross-linked networks are inherently semi-brittle. In practice, the material system is a two-part thermoset epoxy resin in which an epoxy component is mixed with a hardener to initiate curing and create a chemical reaction. The hardened material contains various defects that control fracture initiation, and their efficiency as crack initiators is dependent on their size and shape [1–4]. The Weibull model has been widely used to describe the statistical distribution of mechanical properties in brittle materials [5–7]. This model is based on the "weakest link" hypothesis, in which the most serious flaw controls the strength. The estimation of Weibull parameters has been an interesting theme of scientific research in the last 50 years [8–12]. Much effort was spent on studying the biasing of the parameters evaluated by different methods, including linear regression, the method of moments and the maximum likelihood method. There are still two major limits to the description of mechanical properties using the Weibull model. (i) Due to its statistical

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nature, there is uncertainty in parameters obtained from a limited number of experiments. An infinite number of samples should be tested to obtain the true values of Weibull parameters. (ii) Complex flaw distribution within a material can have various effects on different superimposed Weibull distributions. A large number of studies have been undertaken to assess the strength dispersion of different epoxy resin systems using the Weibull theory [13–22]. Some examples include the flexural strength distributions of unmodified and thermoplastic modified epoxy resin [13], size effects on flexural strength of poly(methyl methacrylate)-based bone cements [14], flexural behavior of sepiolite-modified epoxy resin [15], effects of voids on tensile strength and Young's modulus of structural adhesives [16], viscoelastic properties of epoxy-phenolic reinforced with multi-walled carbon nanotubes (MWCNTs) [18], strength distribution of E-glass/epoxy MWCN composites [19] and tensile failure of unidirectional composites reinforced with flax [20]. In most of these studies, the most serious flaw controls material failure.

The complicating factor in determining the strength of bulk epoxy resin materials is their complex failure mechanisms. As in brittle materials, the volume of the body subject to stress can influence the measured strength in semi-brittle polymeric materials. This phenomenon is termed size and stress gradient effects [23–25]. In general, the strength of an epoxy resin is determined by the presence and interactions of defects (e.g., voids and microcracks), the generation of the tensile stresses at these defects and the stress gradient along the fracture path. Flexural testing is considered an appropriate measure of the strength because it combines elements of compression, tension and shear, which more closely mimics in vivo stresses than either compression or tension testing alone. The effect of the stress gradient on material behavior is shown in Fig. 1.

The ratio of the flexural strength to the uniaxial tensile strength has not been fully examined in previous studies. The results presented in this paper are a part of a much broader investigation into the mechanical behavior of bulk epoxy resin polymeric materials [26–30]. The main objective of the present study is three-fold: (i) to investigate the effect of loading systems on mean strength using the Weibull model, (ii) to obtain the ratio of mean strength measured in a flexural test to the mean strength in a uniaxial tension test using both analytical [30] and statistical approaches that reflect stress gradient effects on strength, and (iii) to study the effects of environmental conditions and aging on this ratio.

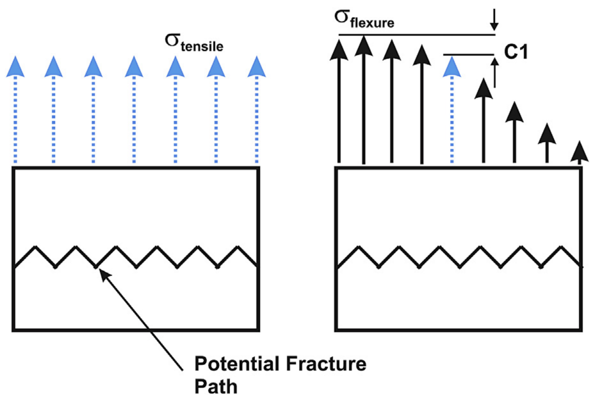

Fig. 1. Stress gradient and nonlinear stress distribution (C_1 is the ratio of the flexural strength to the tensile strength).

2. Materials, characterization, and methods

Epon E863 and hardener EPI-CURE 3290 with a 100/27 weight ratio, PRI 2002-3-R-A and hardener PRI2000-5-HR-B with a 100/22 weight ratio, and PR520 dyed with Nigrazine were used in this study. PRI has a low glass transition temperature and a wide application for civil infrastructure repair and rehabilitation due to its high workability. Epon E863 and PR520 have relatively high glass transition temperatures and have wide applications in the aerospace industry. The PRI2002 samples stored at 60°C and 90% Rh for almost 48 weeks were conditioned and tested. The two-part thermoset epoxies were hand mixed using spatulas and deposited into moulds while minimizing the enclosure of large defects (air bubbles). The same casting procedure was used to make all the tension, compression and bending samples to ensure that the method of fabrication did not influence the mechanical properties. All samples were cured according to the suppliers' recommendations and were machined from the plates. ASTM standards D638 [31], D695 [32], D790 [33], and D 6272 [34] were considered for tension, compression, and three-point-bending (3PB) and four-point-bending (4PB) flexural tests, respectively. All the tests were conducted at room temperature, in displacement control and at a loading speed of 493 μstr/sec. The loading rates corresponding to the desired strain rates were calculated based approximately on the elastic linear assumption and geometry of the samples. Beams of Epon E863 and PR520 with average dimensions of 3.2 mm (width) × 3 mm (thickness) × 25 mm (length) and 20-mm span were tested in the 3PB setup. Each 3PB sample had a groove with an average radius of 0.5 mm at the middle of the beam. The detail of the notch in PR520 was different compared to the shape of the groove in the other two material systems. 4PB beams of PRI2002 with average dimensions of 4.2 mm (width) × 4 mm (thickness) × 25 mm (length between two supports) and 12 mm (middle span) were tested. Tension and compression stress-strain curves were required to complete the analytical study; therefore, tensile tests were conducted using dumbbell-shape samples with average dimensions of 3.18 mm (thickness) × 3.5 mm (width) × 16 mm (length). The average dimensions of the prismatic compression samples were 3 mm × 3 mm × 8 mm. Each sample's thickness and width were carefully measured prior to testing. The strain field was determined using ARAMIS [35], a 3D digital image correlation (DIC) system that enables non-contact measurement of displacement and strain fields. The 2D surface strains in the sample were extracted using continuum mechanics principles. Using DIC, we examined the heterogeneous behavior of the materials and the effects of stress concentrations on premature failure. The tension, compression, and flexural behavior of epoxy resin Epon E 863 [26], PR520, and PRI subject to 60°C and 90% Rh were obtained. Sample sizes of 20 or greater are advised to obtain results that can be considered representative of the statistical analysis [5,9]. After testing, the surfaces of epoxy resins were examined by scanning electron microscopy (SEM) to investigate fracture behavior. Fig. 2 (a,b) shows smooth fracture surfaces of PRI2002 and E863, which are typical of a glassy material. The surface is flat with some quasi-straight, quasi-parallel lines that indicate a relatively brittle failure. Fig. 2 (b) shows unstable failure with less crack propagation interruption after initiation in the tension test and voids in the cured E863.

3. Estimation of Weibull parameters

Because we observed a quite large dispersion of the flexural strength, a reasonable hypothesis is that a statistical model can describe this dispersion. The Weibull distribution model was analyzed to assess its capacity to represent the modulus of rupture stress (σ_{MOR}) data and to estimate the ratio of mean flexural strength to mean tensile strength. The subscript MOR denotes the flexural strength. Therefore, the purpose of this part of the study was to determine the scale parameter and Weibull modulus, m, of the Weibull distribution associated with the flexural strength of the tested materials. Weibull statistics link the failure probability, P_f, to σ_{MOR} by means of the modified Weibull model [8,9,36].

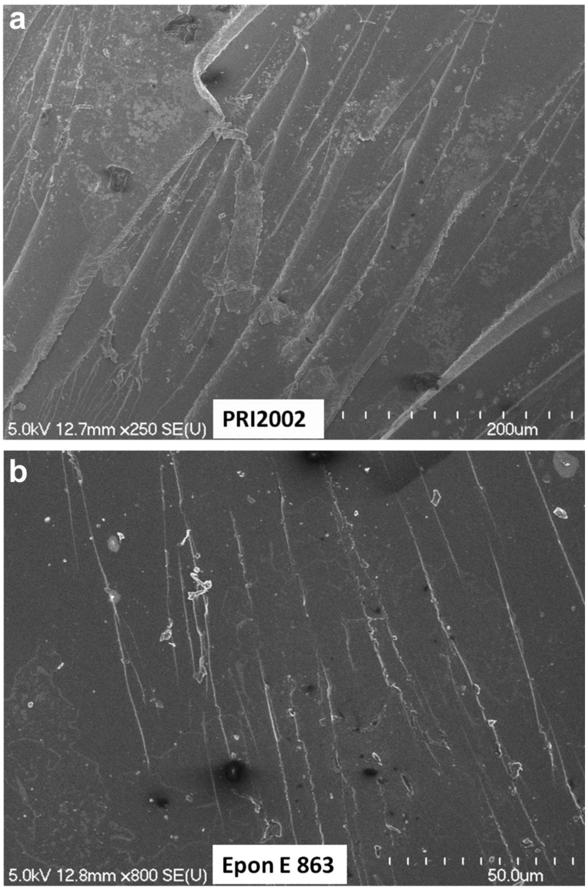

Fig. 2. SEM micrograph of fracture surfaces (a) PRI 2002 flexural sample; (b) voids present in the cured Epon E 863 tension sample.

$$P_f = 1 - \exp\left[-\left(\frac{\sigma_{MOR}}{\sigma_0}\right)^m Ve\right] \tag{1}$$

where σ_{MOR} is the flexural strength (rupture stress), σ_0 is a normalizing factor, m is the Weibull modulus that characterizes the scattering in the measured values of strength, and Ve is the volume of the material subjected to a uniaxial tension that would have the same probability of failure as the sample. The Weibull modulus is a statistical value without any physical meaning. However, it could be linked to the flaw size distribution; a higher m normally leads to a steeper function and a lower dispersion. σ_0 is related to the mean flexural strength of the distribution. Monte Carlo simulation was used to compare the different estimation methods [5,8,9], and the results revealed that the method of linear regression with an appropriately selected estimator and at least 20 samples provides a reasonable estimate for the Weibull model. However, in this paper, linear regression and the method of moments were used for a more comprehensive comparison between the statistical and analytical methods.

3.1. Method of moments

In this method, the first and second moments of a set of data are assumed to be the mean value and standard deviation of the whole distribution (i.e., and infinite number of samples), respectively [5]. The coefficient of variation (Equation 2) for the Weibull distribution is only a function of m, which was solved for using the Newton-Rhapson method.

$$C_{var} = \frac{\sqrt[2]{\Gamma\left(1+\frac{2}{m}\right) - \Gamma\left(1+\frac{1}{m}\right)^2}}{\Gamma\left(1+\frac{1}{m}\right)} \tag{2}$$

where Γ is the gamma function.

3.2. Method of linear regression

Linear regression is a special case of the least squares method. This method is based on the minimization of the squares of deviation of the data from the fitting function. Taking the natural logarithm of equation (1) twice gives the linear equation

$$\ln\ln\left[\frac{1}{1-P_f}\right] = m\ln(\sigma_{MOR}) - m\ln\left(\frac{\sigma_0}{Ve^{1/m}}\right) \tag{3}$$

In this method, the slope of the Weibull model determines the Weibull modulus, and the y-intercept is equal to $-m\ln\left(\sigma_0/Ve^{1/m}\right)$. As the true value of failure probability for each σ_{MOR} is not known, we employed the most common estimators [9].

$$P_{i1} = \frac{i-0.5}{N}(a),\quad P_{i2} = \frac{i}{N+1}(b),\quad P_{i3} = \frac{i-0.3}{N+0.4}(c),\quad P_{i4} = \frac{i-0.5}{N+0.25}(d) \tag{4}$$

where i is the rank of the measured strength.

4. Statistical approach

Table 1 shows the σ_{MOR} of all the bending samples ordered from the lowest to highest stresses using the definition of P_{i1}. The σ_{MOR} of PR520 was not available for the statistical analysis due to premature sample failure. The flexural load-deflection response of PR520 is described in the “Analytical Simulation” section. The data for Epon E863 for all the estimators were plotted as shown in Fig. 3. Table 2 presents the least square equations and the Weibull modulus for Epon E863. Very close agreement was observed between all the estimators. By comparing the method of moments with the results from linear regression, it was clear that the definition of P_{i1} yielded the best results of all the estimators. Therefore, estimator P_{i1} was used to analyze the rest of the samples. Fig. 4 illustrates the Weibull plot for Epon E863 and PRI2002. The linear plots in Fig. 4 are nearly parallel to each other, which indicates that the epoxies have a similar Weibull shape parameter. The Weibull shape parameter does not vary much; therefore, it can be considered to be independent of the type of epoxy resin. The mean flexural strength of the Weibull distribution σ_{mean} is given by:

$$\sigma_{mean} = \frac{\sigma_0}{V e^{1/m}} \Gamma\left(1 + \frac{1}{m}\right) \tag{5}$$

The Weibull distribution parameters, m (Weibull modulus), and σ_{mean} (mean flexural strength) for all samples are listed in Table 3. The correlation coefficients R^2 were above 0.85 and thus considered acceptable. Closer examination of Fig. 4 shows no bimodal distribution or “extrinsic” defects related to poor production or human error, such as poor mixing or inadequate mould filling. The “intrinsic” flaws are related to features within the material engendered by uncontrollable aspects of production of the

Table 1
Flexural strength data for the thermoset epoxy resins Epon E863 and PRI2002. “*i*” is the sample rank, and P_i is the failure probability calculated from Equation 4(a).

i	P_{iE863}	P_{iPRI}	σ_{E863} (MPa)	σ_{PRI} (MPa)	i	P_{iE863}	σ_{E863} (MPa)
1	0.01	0.019	86.5	67	27	0.51	110.3
2	0.029	0.058	93.5	68.5	28	0.529	110.4
3	0.048	0.096	94.4	69	29	0.548	110.5
4	0.067	0.135	99.5	69.5	30	0.567	111.1
5	0.087	0.173	100.2	70.1	31	0.587	111.4
6	0.107	0.212	100.4	70.6	32	0.606	111.5
7	0.125	0.25	100.5	71.3	33	0.625	111.9
8	0.144	0.288	100.9	71.8	34	0.644	113.3
9	0.163	0.327	101.6	72.4	35	0.663	113.7
10	0.183	0.365	102.0	73	36	0.683	114.5
11	0.202	0.404	102.5	74	37	0.702	115.3
12	0.221	0.442	103.5	76.8	38	0.721	116.3
13	0.24	0.481	103.8	77	39	0.74	116.6
14	0.26	0.519	103.9	77.3	40	0.76	117
15	0.279	0.558	104.8	77.5	41	0.779	117.7
16	0.298	0.596	105.5	78	42	0.798	118.1
17	0.317	0.635	105.6	79.1	43	0.817	118.6
18	0.337	0.673	107.1	79.6	44	0.837	119.4
19	0.356	0.712	107.5	81.2	45	0.856	119.8
20	0.375	0.75	107.8	81.5	46	0.875	120.0
21	0.394	0.788	108.0	83	47	0.894	120.1
22	0.413	0.827	108.9	84.2	48	0.913	120.7
23	0.432	0.865	109.1	85	49	0.933	120.8
24	0.452	0.904	109.7	86.4	50	0.952	121.3
25	0.471	0.942	109.8	88	51	0.971	121.4
26	0.49	0.981	109.9	91.5	52	0.99	121.8

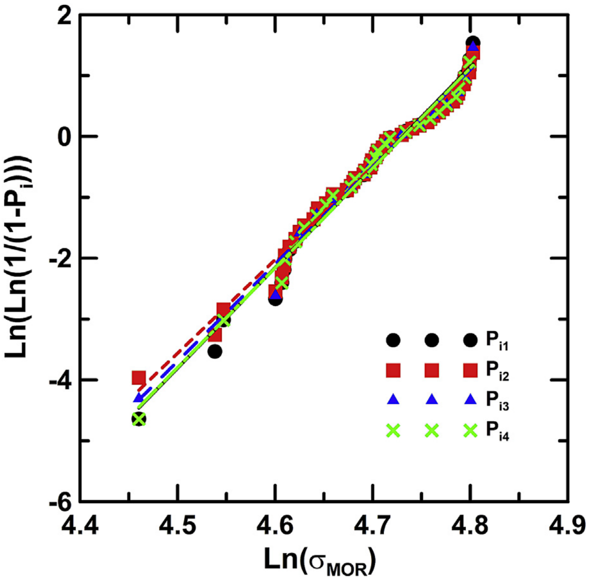

Fig. 3. Weibull plot for Epon E863 with various estimators.

Table 2
Linear regression analysis and method of moment results for Epon E863.

Estimator	Least square equation	Weibull modulus
P_{i1}	16.49 ln(σ_{MOR}) − 78	16.49
P_{i2}	15.35 ln(σ_{MOR}) − 72.7	15.35
P_{i3}	15.97 ln(σ_{MOR}) − 75.6	15.97
P_{i4}	16.40 ln(σ_{MOR}) − 77.6	16.40
Method of moment	N/A	16.72

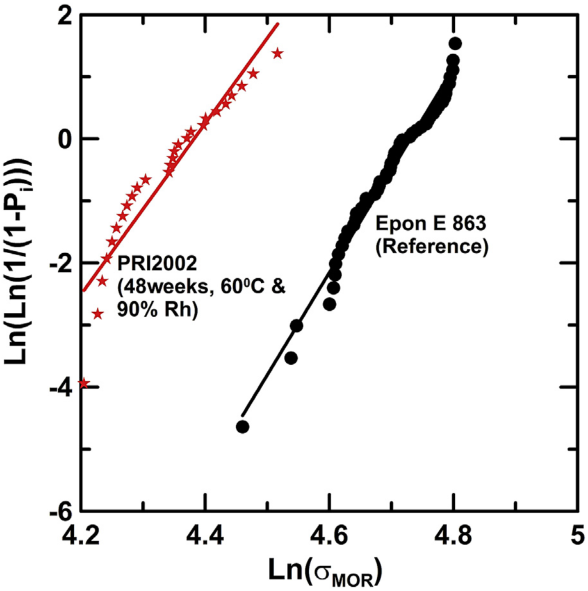

Fig. 4. Weibull model for Epon E863 and PRI2002 using estimator P_1.

Table 3
Weibull parameters for Epon E863 and PRI2002.

Material	Storage Condition	Number of Samples	MLR		MM		σ_{mean} (MPa)
			m	C_1	m	C_1	
Epon E863	Room temperature (ref.)	52	16.49	1.47	16.72	1.46	109.7
PRI2002	60°C & 90% Rh for 48 weeks	26	13.7	1.38	13.8	1.37	77.03

final product, such as segregation, small-scale porosity due to the escape of bubbles within the polymer, or any centers that give rise to incompatible deformation. These flaws are in variable size, shape and orientation. In one sample, the largest crack may be normal to the applied load, while in another sample it may be at an angle to the applied stress. Obviously, the latter has a higher strength than the former; therefore, variable defects with respect to the applied load can account for the flexural strength scatter. The morphological features developed during epoxy resin curing, as shown in Fig. 2(b), can be considered as the cause of the scatter. The ratio of the mean flexural strength to the mean strength in uniaxial tension based on the Weibull model is

$$\frac{\sigma_{MOR,3PB}}{\sigma_{UTS}} = \left[2(m+1)^2\right]^{1/m} \text{(a)} \quad \frac{\sigma_{MOR,4PB}}{\sigma_{UTS}} = \left[\frac{6(m+1)^2}{m+3}\right]^{1/m} \text{(b)} \tag{6}$$

Table 3 indicates that the above ratio based on the Weibull model was between 1.37 and 1.47. The Weibull model is based on an empirical formula that relates the probability of failure to the rupture stress. The number of specimens tested has a large influence on the calculated Weibull modulus. The first limit in describing the fracture behavior of brittle and semi-brittle materials by the Weibull model is due to the statistical nature of the model itself. This limitation leads to an uncertainty in the parameters obtained from a limited number of experiments. The true values of the Weibull parameters can only be obtained from an infinite number of samples. For any small number, only an estimate, not the actual value, can be achieved. In addition, the Weibull model is based on the "weakest-link hypothesis," which means that the most serious flaw controls the strength. However, there are quite complex flaw distributions in the material, which may affect two superimposing Weibull distributions. It should be noted that σ_{MOR} used in the Weibull model was obtained using the approximate linear stress distribution.

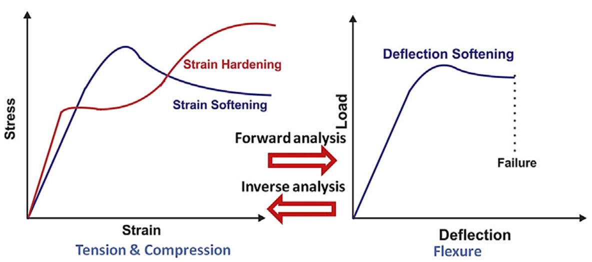

Fig. 5. Analytical model for the simulation of flexural load-deflection response in epoxy resin polymeric materials.

5. Analytical simulation

The main output of flexural testing is the load-deflection curve, which does not provide much direct information regarding the stress distribution or the constitutive law. The assumption of linear elastic stress distribution also does not match the nonlinear behavior of semi-brittle

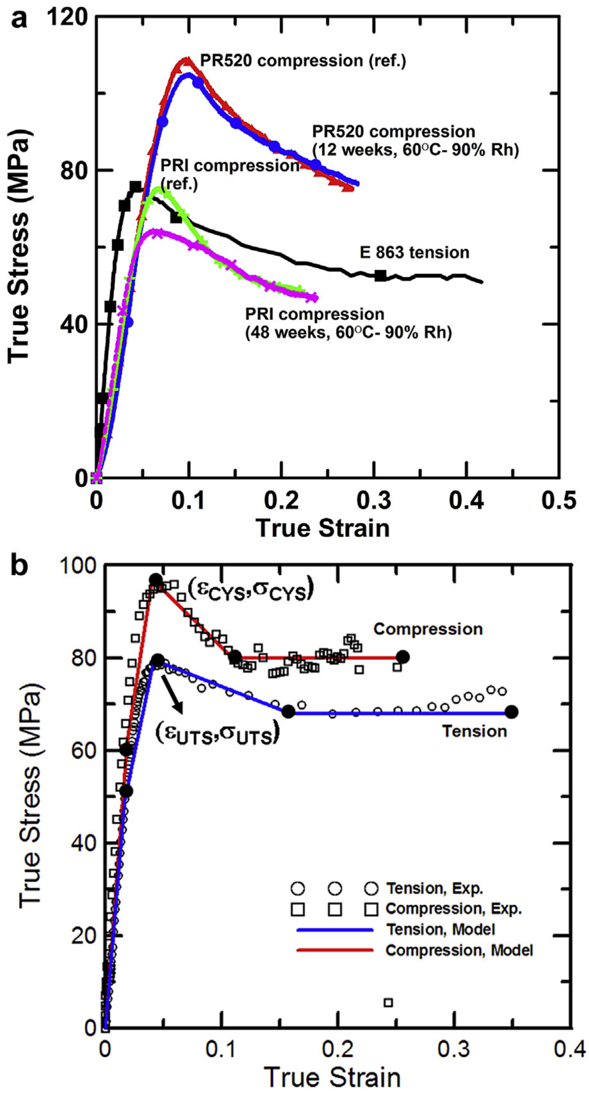

Fig. 6. (a) Tension and compression stress-strain curves of epoxy resin polymers under different environmental conditions (the stress-strain curve for E863 is from Yekani Fard et al. [26]); (b) general features of stress-strain curves (CYS: compressive yield strength, UTS: ultimate tensile strength).

materials. To overcome these problems, Yekani Fard et al. [28] developed a novel analytical technique based on a piecewise-linear strain-softening tension and a compression stress-strain model to simulate the flexural load-deflection response of different epoxy resin structural systems. The developed model can be used for both forward and inverse analysis, as shown in Fig. 5. This model is based on the general shapes of the stress-strain curves in tension and compression in epoxy resin materials [26,37–40]. As shown in Fig. 6 (a,b), all the samples appeared to follow similar tension and compression stress-strain evolution. The stress-strain curves in epoxy resins in Fig. 6 (a) are highly nonlinear and exhibit the following distinctive features (Fig. 6b): linearly elastic, nonlinearly ascending, yield-like (peak) behavior, strain softening, and nearly perfect plastic flow. The details of the model are described in our previous publications [29,30]. Flexural load was used instead of approximate linear flexural stress to avoid flexural stress calculation errors. When a material with strain-softening behavior, such as Epon E863, is loaded beyond the peak, the increase in deformation decreases the load-carrying capacity. If no premature failure occurs for resins, the load-deflection curve exhibits a deflection-softening behavior in the post-peak part of the response. Fig. 7 (a) illustrates the load deflection curves of the “Avg.” and “Avg. − Std.” of E863 experiments. Fig. 7(b) shows the mean load-deflection curve of PRI2002 in the aged condition (48 weeks, 60°C and 90% Rh). Fig. 7(c) illustrates the load-deflection response of PR520 in the reference condition. The stress concentration due to the right angle of the notch caused lower failure strength and premature failure in PR520 samples. However, all of the simulations show that the direct use of tension and compression stress-strain curves underestimated the flexural load-deflection response, as expected. An inverse analysis of the load-deflection curves shows that the ratio of mean flexural strength to the mean tensile strength for the epoxy resin polymers is between 1.18 (PRI2002) and 1.21 (E863 and PR520).

6. Conclusions

Mechanical tests were conducted on three structural epoxy resins under different environmental and loading system conditions. The results indicate that the direct use of tension and compression data leads to underestimation of flexural strength. The Weibull model was used to describe the data scatter in the flexural strengths of Epon E863 and PRI 2002. The true value of the Weibull parameter m and, consequently, the ratio of flexural strength to tensile strength C_1 could only be obtained for an infinite number of specimens. For any smaller number, only an estimate can be achieved. Our comparison of the different Weibull methods and estimators revealed that there are no

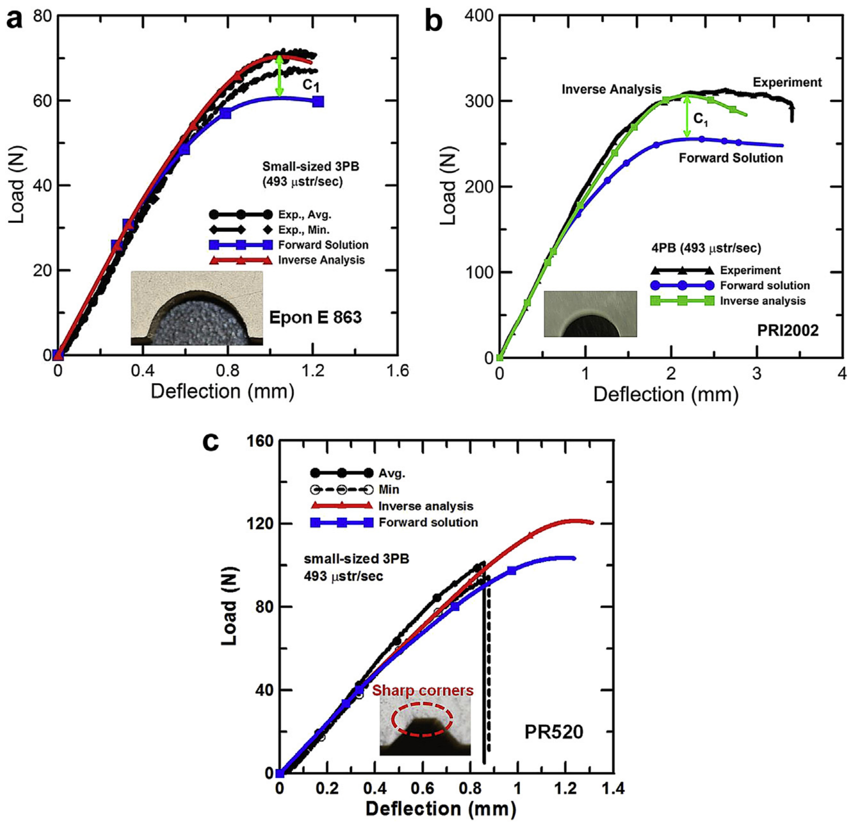

Fig. 7. Simulation of the flexural load-deflection response for (a) 3PB Epon E863 in the reference condition, (b) 4PB PRI2002 in the aged condition (48 weeks, 60°C and 90% Rh), and (c) 3PB PR520 in the reference condition.

statistical advantages in using the moments method compared to the simple linear least squares method. For thermoset epoxy resins with a Weibull modulus of about 13.8, the Weibull model predicts a mean flexural strength about 37% higher than the tensile strength. However, analytical simulations of 3PB and 4PB demonstrated that this ratio is around 1.20 for all tested epoxy resin polymeric materials. Due to the intrinsic limitations of the statistical models, the Weibull model can only estimate the ratio. It should be noted that the flexural strengths provided in commercial data sheets of epoxies are usually much higher, sometimes by as much as 50%, than the corresponding values from tensile tests. This discrepancy can be explained in terms of the underlying assumptions of the Weibull model that do not fit with the semi-brittle nature of thermoset epoxy resins and the unrealistic assumption of linear stress distribution.

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