Specimen size effect during tensile testing of an unreinforced polymer

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Various specimen sizes of an unreinforced polymer, Hercules 3501-6 thermosetting epoxy, were subjected to a tensile test. The general specimen geometry was a rectangular dog-bone shape with constant gauge length, but with each specimen size having a different cross-sectional area. These cross-sectional areas were obtained by varying the thickness of the epoxy during casting, and the gauge section width during grinding. The resulting failure surfaces of the specimens were observed and photographed using scanning electron microscopy. The results indicate that failure stress, dimensions of the critical flaw which caused failure, and a quantity which is proportional to the fracture toughness, are all correlated with specimen size.

1. Introduction

Determining the strength of a material is probably the most common mechanical test performed. The results are useful for comparing one material with another, and for utilization as a design limit. For ductile materials the determination of strength, comparison with other materials, and establishing design limits is reasonably straightforward. But brittle materials require special effort during specimen preparation and mechanical testing to ensure repeatable results. Additionally, comparison with other materials and setting design limits must be done with caution. The complicating factor for brittle materials is that the volume of the body subjected to stress can influence the measured strength. This phenomenon is identified as the size or scale effect.

All materials contain flaws at some level. However, it is the manner in which the material responds to these flaws that determines whether a size effect is present. If the material is ductile, a flaw-induced stress concentration is diminished by localized irreversible deformation. This plastic deformation desensitizes the material to the presence of a flaw. For a brittle material, the stress concentration due to a flaw is not diminished. As a result, the material is subjected to the entire stress concentration. Local fracture initiates and propagates, leading to global failure of the material. It is this local material response that leads to the presence of a size effect.

The size-effect phenomenon has been observed in many materials throughout history. For example, Hertzberg [1] describes the simple testing apparatus and methodology utilized by Leonardo da Vinci to measure tensile strength. His results indicated that short wires are stronger than longer wires. However, it is worth noting that he believed it necessary to run replicates of the test, which implies that he was cognizant of the statistical nature of the problem.

Others have investigated the presence of a size effect during mechanical testing of various materials. These individuals, and the materials of interest, include Peirce [2] on cotton yarns; Davidenkoff and Shevandin [3] on steel; Kase [4] on vulcanized rubber; and Beams *et al.* [5] on thin films of metals. Additionally, a complete survey of the size-effect literature was performed by Harter [6].

Support for the occurrence of a size effect was first provided by Griffith [7] who proposed the presence of flaws as being responsible for reducing the strength of isotropic materials. As the specimen size increases, the number of flaws and the likelihood of the presence of larger flaws also increases. These larger flaws and the associated stress concentration lead to material failure at lower overall stresses.

The results presented here are part of a much broader investigation into the mechanical response of thermosetting resins [8]. The objective of the present work was to determine whether a size effect is present when tensile testing a thermoset epoxy resin. The particular material selected was Hercules 3501-6 epoxy. This is a highly cross-linked amorphous polymer, classified as a brittle material because of its low fracture toughness [9] and its apparently linear stress-strain response. The presence of a size effect for the material selected would be of unusual interest, as it is used as the matrix material in a number of composite materials. Therefore in use, the characteristic dimensions of the material would be of the order of the diameter of the fibres for which it serves as a matrix. These dimensions are quite small: for example, the diameter of a commonly used carbon fibre is 6.8 µm [10]. A size effect would raise the question as to what

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strength the matrix material has in a composite, and whether the matrix-dominated properties reflect this strength.

2. Experimental procedure

The specimen geometry utilized in this study included a dog-boned shape, constant gauge length and a rectangular cross section. The shape selected forced the failure to a particular portion of the specimen where the dimensions could easily be measured and volume (size) calculated. A constant gauge length for all the specimen sizes allowed one machining jig to be used throughout specimen fabrication. The extremes of the dimensions of the gauge section ranged from 0.25×0.05 to 1.27×0.76 cm, all with a 5.1 cm gauge length. To determine if and when a size effect existed between these extremes of specimen size, ten intermediate specimen sizes were selected. The specimen gauge section dimensions and volume, along with the number of specimens tested, are shown in Table I. As can be seen, the volume of the largest specimen size was about 70 times greater than the volume of the smallest specimen size.

Specimen fabrication began by casting the Hercules 3501-6 epoxy resin into blanks 15.2 cm long, 1.27 cm wide, and either 0.25, 0.64, or 1.27 cm thick. The steel box mould used for casting the epoxy consisted of a square bottom and four sides which were bolted together. All surfaces were ground to facilitate release of the cured epoxy resin. Individual resin blanks were obtained by placing dividers in the mould bottom. The dividers were separated by spacers with a thickness which corresponded to the thickness of the specimen desired. The casting process consisted of preheating the mould used to cast these specimen blanks to 110 °C, and adding the proper amount of uncured epoxy which was then degassed under vacuum for ~ 1 h. After removing the vacuum, the epoxy was cured for 8 h at the same temperature (110 °C) in the same oven. The epoxy specimen blanks were then removed from the mould. After the requisite number of blanks was obtained, all were simultaneously postcured for 8 h at 177 °C. The dog-bone specimen shape was obtained by grinding the width of each specimen

TABLE I Specimen dimensions used in present size-effect study.

Size No.	Number of specimens tested	Nominal gauge section dimensions width × thickness (cm)	Gauge section volume (cm ³)	
1	13	0.05 × 0.25	0.066	
2	13	0.10×0.25	0.131	
3	10	0.25×0.25	0.328	
4	10	0.51×0.25	0.655	
5	10	0.76×0.25	0.938	
6	10	1.02×0.25	1.311	
7	10	0.51 × 0.64	1.475	
8	10	0.61×0.64	1.966	
9	10	0.69×0.64	2.294	
10	10	0.76 × 0.64	2.458	
11	10	1.02×0.64	3.227	
12	10	0.71×1.27	4.588	

blank with a diamond-coated router. Specimen geometry was assured by utilizing a template during this operation. The specimen geometry and dimensions of the largest and smallest specimens are shown in Fig. 1.

All tensile testing was performed in an Instron Model 1125 electromechanical testing machine. After all mechanical testing had been performed, one of the two failure surfaces of each specimen was prepared for observation with scanning electron microscopy (SEM). During this process the complete failure surface was photographed. A typical failure surface is indicated in Fig. 2 along with a sketch to indicate the features: the flaw which caused failure; the mirror which was the region of slow crack growth; and the hackle which indicates the region of rapid crack growth and branching in an attempt to stabilize the failure process.

3. Results

The results of the tensile testing are presented in Table II. Indicated are the average and coefficient of variation of the strength, the flaw size and the stress flaw constant. This latter value was calculated by multiplying the ultimate tensile strength of each specimen by the square root of the diameter of the flaw causing the specimen to fail. This quantity can be shown to be related to the fracture toughness of the material [11]. As indicated, the observed strength for the smallest specimen size (No. 1) was over twice the observed strength of the largest specimen size (No. 12). The coefficient of variation of the strength was $\sim 10\%$ for the specimens with larger volumes, but approached $\sim 20\%$ for the specimens with the smallest volumes. This table also indicates the average flaw size to be decreasing, and the average stress flaw constant to be increasing as the specimen size decreased. A plot of the average strength against the volume of the speci-



Figure 1 Tensile specimen shape and dimensions (cm) of smallest and largest specimens.



Figure 2 Example of a typical failure surface of a Hercules 3501-6 epoxy tensile specimen as observed in scanning electron microscope. (a) SEM of a typical fracture surface; (b) sketch of a typical fracture surface with major features identified.

TABLE II Average tensile strength, flaw size and stress-flaw constant of Hercules 3501-6 neat epoxy as a function of specimen size.

Specimen Size Number	Tensile* strength (MPa)	Average flaw* size (10 ⁻⁵ m)	Stress-flaw* constant (MPa m ^{1/2})
1	94 (18)	10(24)	0.93(15)
2	70(23)	13 (40)	0.73 (9)
3	61 (12)	13 (30)	0.68(15)
4	56(11)	15(23)	0.67(14)
5	52 (9)	15(11)	0.64 (9)
6	50(11)	18(21)	0.66(15)
7	49(11)	15(23)	0.62(11)
8	50 (8)	18(17)	0.64 (5)
9	48(13)	18 (20)	0.65(10)
10	48 (9)	18(16)	0.64 (7)
11	41 (12)	25(31)	0.63(16)
12	44(11)	20(14)	0.62(11)

* C_v in parantheses.

men tested is shown in Fig. 3. Also indicated in this figure are the relative cross-sectional areas of the specimens. Since the gauge length was constant for all specimens, these relative cross-sectional areas depict a graphical representation of the difference in the volume of the gauge section.

4. Discussion

The observed tensile strength of Hercules 3501-6 epoxy is dependent on the size of the specimen tested, as indicated in Fig. 3. At the larger specimen sizes, the measured tensile strength appeared to level off. At the smaller specimen sizes, the tensile strength can be seen not only to increase, but to increase more and more rapidly, with a decrease in specimen size. Obviously, if the observed strength is to stay finite, this behaviour must also level out. Fig. 3 also indicates a reasonably smooth transition between the two extremes of material behaviour.

The tensile strengths of specimens with larger gauge-section volumes appeared to be approaching a limiting value, as can be seen in Fig. 3. Davidenkov



Figure 3 Average tensile strength against gauge section volume for neat Hercules 3501-6 epoxy.

and Shevandin [3] suggest that the levelling out occurs because, at sufficiently large specimen volumes, a complete set of all possible flaws and discontinuities will be present. While this reasoning has intuitive appeal, empirical support is difficult to find. For example, Fig. 4, which indicates the average size of the critical flaw against the specimen volume, suggests that a levelling off of flaw size may occur at larger specimen sizes than tested here, if the flaw size for specimen size No. 11 is discounted. As will be discussed, discounting this value is not unreasonable. The average flaw size in Fig. 4 does decrease for the smallest specimen sizes, which supports the contention that there is a lower probability of larger flaws in smaller specimens.

One final point to note is that the average flaw sizes indicated in Fig. 4 are substantially larger than the diameter of a typical fibre. Thus it is doubtful that such flaws would exist in an actual composite: they would probably be disturbed during the prepregging process. Therefore although the strength of the unreinforced (neat) polymer material may be dictated by these flaws, the effect (if any) which matrix flaws have on a composite remains unknown.

Another approach to understanding the observed strengths of Table II is based on fracture mechanics arguments. The following equation is suggested by Rice [11]:

$$\sigma \sqrt{d} = \kappa \tag{1}$$

where σ is the failure stress and *d* is the characteristic dimension of the flaw that caused failure. The quantity κ , the stress flaw constant, was shown by Rice [11] to be related to the material fracture toughness divided by a geometric factor which is dependent on the stress field around a flaw. A plot of the average stress flaw constant κ against the size of the specimen is shown in Fig. 5. This figure indicates several important details. Firstly, any doubt about the approach of calculating the stress flaw constant can be dispelled by considering specimen size 11. This specimen size plots as a lower strength than the trend in Fig. 3, a larger than expected flaw size in Fig. 4, but a stress flaw constant



Figure 4 Flaw size causing failure against gauge section volume for neat Hercules 3501-6 epoxy.



Figure 5 Average stress-flaw constant against volume of gauge section for neat Hercules 3501-6 epoxy.

totally consistent with the other data in Fig. 5. This latter correlation suggests, at least for larger specimen sizes, that the stress-flaw constant is representative of a material behaviour that is independent of specimen size, i.e. the stress-flaw constant represents an inherent material property. Fig. 5 also indicates that the value of the stress flaw-constant is unchanging for all but the two or three smallest specimen sizes, where it increases. This suggests either that the material fracture toughness increased, or the geometric factor decreased. It has been shown [1] that the geometric factor increases as the ratio of flaw size to specimen size increases. This ratio can be seen to increase for small specimen sizes by comparing flaw sizes from Table II with the nominal specimen dimensions from Table I. Therefore the geometric factor cannot be the cause of the increase in the stress-flaw constant, which means the apparent material fracture toughness must have increased.

An increase in fracture toughness can be explained if the specimen stress is in transition from a condition of plane strain to one of plane stress with decreasing specimen size. If this occurs, Hertzberg [1] suggests that the size of the plastic zone at the tip of a crack would not be restricted, which would effectively toughen the material. This would increase the strength of the material by desensitizing it to flaws. Hertzberg [1] suggests that a transition from a plane strain to a plane stress condition could occur if one of the specimen characteristic dimensions becomes smaller in comparison to the flaw size. This suggests a correlation could exist between the stress-flaw constant and the minimum cross-sectional dimension of the tensile specimen. Fig. 6 is a plot of the stress-flaw constant against the inverse of the specimen thickness. An increase in the stress-flaw constant for the smaller specimen thicknesses is indicated, as is the possible existence of a limiting value for specimens with larger minimum gauge section dimensions. The behaviour indicated in Fig. 6 is very similar to that expected during a fracture mode transition from plane strain to plane stress with decreasing specimen size [1].



Figure 6 Average stress-flaw constant against the inverse of the minimum cross-sectional dimension for neat Hercules 3501-6 epoxy.

The increase in strength due to this apparent transition from plane strain to plane stress is not inconsequential. For example, if the stress-flaw constant for specimen size 4-12, which appear to be approximately constant in Fig. 6, are averaged, the result is 0.64 MPa $m^{1/2}$. Assuming this value of the stressflaw constant to be in effect for specimen size 1, and using the flaw size for this specimen size from Table II along with the relationship in Equation 1, results in an ultimate tensile strength of 64 MPa as opposed to the experimentally observed value of 94 MPa. This calculated strength is associated with the concept of smaller specimen volumes having smaller flaw sizes. As this indicates, the change in observed strength due to the increase in the stress-flaw constant, which is apparently due to an increase in fracture toughness, is substantial.

5. Conclusions

The observed strength of Hercules 3501-6 neat epoxy resin was shown to be dependent on specimen size. There appear to be two reasons for this dependence. Firstly, the observed strength increases as the specimen size decreases. This is as expected based on arguments presented by Griffith [7], and the argument that smaller specimens have a lower probability of having larger flaws. Data on the size of the flaw which caused failure support this latter contention. Secondly, the observed strength increases due to a transition from plane strain to plane stress for specimens with smaller dimensions. Although this behaviour can be explained, it was quite unexpected when first observed, as the literature survey indicated no evidence for this type of behaviour during past sizeeffect studies. The effect of this transition on observed strength was quite substantial.

The existence of a size effect requires any comparison between materials to be performed with caution. It could be quite possible for one material to appear stronger for a given specimen size, due to the distribution of flaws being smaller. If this distribution is inherent, then the difference in observed strengths is also inherent. However, if processing of the material could change the flaw-size distribution, then the results of a comparison could be quite different. Making comparisons becomes difficult if test results for bulk specimens are compared while the materials are actually to be used only as a matrix in a composite material. As previously suggested, the same distribution of flaws as observed in the neat resin would undoubtedly not exist in the composite. Furthermore, the difference in size between any practical neat matrix specimen and the characteristic dimensions of the matrix in a composite are so vast that extrapolation is impractical.

The observations made during this study suggest a need to determine inherent, i.e. volume- or dimensionindependent strengths. This would allow a uniform comparison between materials. The present investigation also suggests that further study is needed at the appropriate local scale to determine how the matrix material and fibre reinforcement interact to produce global behaviour of a composite.

Finally, the existence of a size effect suggests that measured tensile strengths should not be presented as if the value is intrinsic to the material. Instead, the tensile strength should always be accompanied by the dimensions of the specimen.

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