JOURNAL OF MATERIALS SCIENCE 22 (1987) 2465–2469

Mechanical properties of glass fibres containing aluminium dispersoids

A. SHRIVASTAVA, D. C. AGRAWAL, D. CHAKRAVORTY
Advanced Centre for Materials Science, Indian Institute of Technology, Kanpur, India

Glass fibres containing metallic aluminium dispersoids up to 7.5 at% Al have been made using ceramic bushings. The metallic granules have diameters ranging from 5 to 40 nm. A new technique based on strength–strain regression analysis has been used to determine the Young's moduli of the glass fibres. The Weibull parameters have been evaluated by both the graphical regression (GRE) and maximum likelihood (MLE) techniques. Fracture studies have also been carried out. The presence of aluminium particles increases the Young's modulus of the fibres but reduces the strength. The latter arises due to the metallic particles acting as stress concentrators within the glass matrix.

1. Introduction

Several investigations have been reported in the literature for developing techniques to improve the strength of glass fibres other than by a change of glass composition. Internal stresses developed in fibres consisting of borosilicate glass clad with 96% silica glass have been shown to increase the glass fibre strength [1]. Soda–alumina–silica glass fibres subjected to an $Na^+ \rightleftharpoons K^+$ ion-exchange treatment are found not to exhibit the expected strengthening because of the weakening caused by the ion-exchange reaction itself [2]. A marginal increase in fibre strength is observed in some low-melting glasses containing a dispersion of metallic silver granules [3]. Recently it has been shown that glass–metal composites prepared by a powder metallurgial technique and containing aluminium as the metallic species have strength values an order of magnitude higher than those exhibited by the base glass [4–6]. This prompted us to explore the possibility of preparing glass fibres containing aluminium dispersoids and to study their mechanical properties. The experimental results as well as the methods used to analyse the data are presented in this paper.

2. Experimental procedure

The compositions of the glass batches prepared are given in Table I. All glasses are prepared from reagent grade chemicals, Na_2O and CaO being added as carbonates. Aluminium powder of 99% purity (supplied by Sarabhai Merck, India) with a particle size distribution ranging from 1.0 to 4.5 μm is used for incorporating aluminium dispersoids. It must be noted that these particles get oxidized during the melting operation. The final content of metallic aluminium within the glasses could not however be estimated. The glasses with aluminium metal powder are melted in alumina crucibles at around 1450° C and cast in the form of rectangular plates. The latter are broken into pieces which are used to draw glass fibres using ceramic bushings [7] with the bushing tip temperature at around 950° C, the velocity of draw being varied from 10 to 20 cm sec^{-1}. For strength measurements untouched fibres are collected by a jig similar to that described in the literature [8]. The microstructure of the fibres is examined in a Philips EM301 electron microscope. The glass fibres in which aluminium is incorporated show a distribution of particles of size varying between 5 and 40 nm in a glass matrix. The latter are identified as consisting of metallic aluminium from selected-area diffraction patterns [9].

For measuring the strength and Young's modulus of the fibres single fibres are pasted on to fibre mounts made of stiff paper. The gauge length used is 5 cm. The two sides of a fibre mount with a single fibre stuck on it are held between the grips of an Instron machine. The sides of the mount are cut so that the load is taken by the fibre as the crosshead starts to move. The load cell used has a range 0 to 50 g. The chart speed and the crosshead speed are 10 and 0.5 cm min^{-1}, respectively. For each glass composition around 40 fibre samples are tested and the data analysed according to the procedure described below. Glass fibre diameters are measured by a Sheffield Accutron (USA), an electronic comparator with an accuracy of 0.25 μm.

The microstructures of fracture surfaces of glass fibres are studied by an ISI 60 (UK) scanning electron microscope.

3. Data analysis

3.1. Young's modulus

Strength S_i and the strain e_i of the ith specimen are given by

$$S_i = \frac{4P_i}{\pi D_i^2} \quad (1)$$

$$e_i = 10^{-3} x_i \quad (2)$$

where P_i is the breaking load of the ith fibre, D_i is the diameter of the ith fibre and x_i is the total distance covered by the chart paper before the breakage.

Young's modulus E_i may be estimated from

0022–2461/87 $03.00 + .12

TABLE I Composition and density of glasses investigated

Glass No.	Composition (mol %)					Density ($g cm^{-3}$)
	Na_2O	SiO_2	CaO	B_2O_3	Al	
1	30.0	55.0	12.0	3.0	0.0	2.62
2	29.9	54.7	11.9	3.0	0.5	2.62
3	27.8	50.9	11.0	2.8	7.5	2.53

$$E_i = \frac{S_i}{e_i} \tag{3}$$

However, for a set of such measurements made on a number of fibres N ($N \sim 40$) the values of E_i show a wide variation. The mean value $\bar{E}$ given by

$$\bar{E} = \frac{\sum_i E_i}{N} \tag{4}$$

is usually taken as an estimator for the Young's modulus of glass fibres. It is proposed that a better estimate of E can be made by regressing a straight line between strength and strain. The slope of this line should give the required value of E.

3.2. Determination of strength

Mean values of strength are evaluated from two sets of data. One of them is obtained using Equation 1, while the other is found from the slope of then regressed line as discussed above. The calculated strength values S_c can be written as

$$S_c = E_R e_i \tag{5}$$

where E_R is the estimated Young's modulus value obtained by the regression analysis and e_i the strain.

The two estimates of strength are then given by

$$E_1(S) = \bar{S} = \frac{\sum_{i=1}^{n} S_i}{N} \tag{6}$$

and

$$E_2(S) = \bar{S}_c = \frac{\sum_{i=1}^{N} S_{c_i}}{N} \tag{7}$$

where $\bar{S}$ and $\bar{S}_c$ are the mean values of the distributions of S_i and S_c, respectively.

3.3. Weibull analysis

The cumulative probability of failure is given by the Weibull distribution [10],

$$P = 1 - \exp\left[-\left(\frac{S}{S_0}\right)^b\right] \tag{8}$$

where S_0 and b are the scaling and shape parameters, respectively, of the distribution. The following two techniques have been used to evaluate S_0 and b.

3.3.1. Graphical regression technique (GRE)

In this method a least-squares-fit line is regressed between $\ln S_i$ and $\ln [-\ln (1 - P_i)]$. If J_1 and J_2 be the slope and intercept, respectively, of the line, S_0 and b are given by

$$S_0 = \exp (J_2) \tag{9}$$

and

$$b = \frac{1}{J_1} \tag{10}$$

To determine P_i, the probability of failure for strength $S \geqslant S_i$, the strength data are arranged in ascending order of values. The cumulative probability P_i is found from this table as

$$P_i = \frac{i}{N + 1} \tag{11}$$

where i is the sequence number for strength P_i.

3.3.2. Maximum likelihood technique (MLE)

In this technique, the likelihood function is minimized with respect to two parameters [11]. The likelihood function for a failed censored sample with failure strength of r fibres that failed out of n fibres is given by

$$L \propto \left(\frac{b}{A}\right)^r \left(\prod_{i=1}^{r} S_i^{b-1}\right) \exp\left(-\frac{\sum_{i=1}^{r} S_i^b}{A}\right) \times \left[\int_{S=r}^{\infty} \frac{b}{A} S_i^{b-1} \exp\left(-\frac{S_i^b}{A}\right) ds\right]^{n-r} \tag{12}$$

where

$$A = S_0^b \tag{13}$$

for maximum likelihood we can write

$$\frac{\partial}{\partial A} \ln L = \frac{\partial}{\partial b} \ln L = 0 \tag{14}$$

In the present work, since all the fibres are being tested for failure, $r = n$.

Using Equations 12 and 14, therefore, values of b and A – the estimators of b and A, respectively – can be obtained from the following equations:

$$\frac{\sum_{i=1}^{n} S_i^{\hat{b}} \ln S_i \ln S_i}{\sum_{i=1}^{n} S_i^{\hat{b}}} - \frac{1}{\hat{b}} = \ln \sum_{i=1}^{n} \ln S_i \tag{15}$$

$$\hat{A} = \frac{1}{n} \sum_{i=1}^{n} S_i^{\hat{b}} \tag{16}$$

The value of b is obtained by solving Equation 15 by the Newton–Raphson iterative method [12]. Substituting this value of $\hat{b}$ in Equation 16 the value of $\hat{A}$ is determined. It can be shown from the general theory of MLE that the variance of b, the variance of A and the co-variance of b and A are given by

$$\text{Var } (\hat{b}) = \frac{1}{U}\left(-\frac{\partial^2 \ln L}{\partial A^2}\right)_{\hat{b},\hat{A}} \tag{17}$$

$$\text{Var } (\hat{A}) = \frac{1}{U}\left(-\frac{\partial^2 \ln L}{\partial b^2}\right)_{\hat{b},\hat{A}} \tag{18}$$

$$\text{Co-var } (\hat{b}, \hat{A}) = \frac{1}{U}\left(\frac{\partial^2 \ln L}{\partial A \partial b}\right)_{\hat{b},\hat{A}} \tag{19}$$

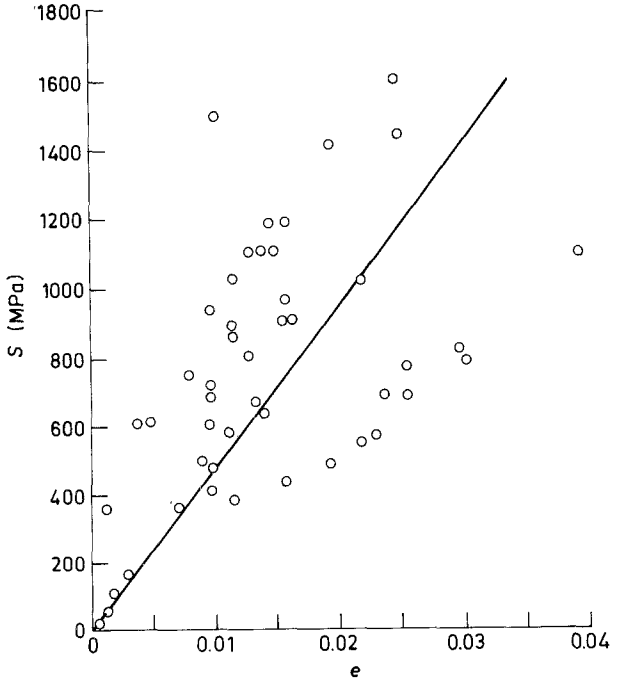

Figure 1 Regression of strength (S) with strain (e) for glass fibre No. 1.

where

$$U = \left[\left(\frac{\partial^2 \ln L}{\partial b^2}\right)\left(\frac{\partial^2 \ln L}{\partial A^2}\right) - \left(\frac{\partial^2 \ln L}{\partial b \partial A}\right)^2\right]_{\hat{b},\hat{A}} \quad (20)$$

$$-\left.\frac{\partial^2 \ln L}{\partial b^2}\right|_{\hat{b},\hat{A}} = \frac{n}{\hat{b}^2} + \frac{1}{\hat{A}}\left[\sum_{i=1}^{n} S_i^{\hat{b}} (\ln S_i)^2\right] \quad (21)$$

$$-\left.\frac{\partial^2 \ln L}{\partial A^2}\right|_{\hat{b},\hat{A}} = \frac{n}{\hat{A}^2} + \frac{2}{\hat{A}^3}\left[\sum_{i=1}^{n} S_i^{\hat{b}}\right] \quad (22)$$

$$-\left.\frac{\partial^2 \mathrm{Ln}\, L}{\partial A \partial b}\right|_{\hat{b},\hat{A}} = \frac{\sum_{i=1}^{n} S_i^{\hat{b}} \ln S_i}{\hat{A}^2} \quad (23)$$

For the Weibull distribution the mean and variance are calculated from

$$\bar{S} = \left(\frac{\hat{b} + 1}{\hat{b}}\right)^{1/2} S_0 \quad (24)$$

$$\mathrm{Var}\,(S) = S_0^2\left[\left(\frac{\hat{b} + 2}{\hat{b}}\right)^{1/2} - \left(\frac{\hat{b} + 1}{\hat{b}}\right)^{1/2}\right]^2 \quad (25)$$

4. Results

Fig. 1 shows a typical regression analysis of glass fibre No. 1. The estimator of Young's modulus obtained from such regression analysis and $\bar{E}$, the value derived from Equation 4, for fibres of different compositions are given in Table II.

TABLE II Young's modulus estimators for different glass fibres

Glass No.	$E + \mathrm{d}E$ (MPa)	$E_R + \mathrm{d}E_R$ (MPa)
1	$(3.0 \pm 1.4) \times 10^4$	$(4.6 \pm 2.3) \times 10^4$
2	$(2.5 \pm 0.8) \times 10^4$	$(5.5 \pm 1.8) \times 10^4$
3	$(2.4 \pm 0.8) \times 10^4$	$(8.4 \pm 2.3) \times 10^4$

Fig. 2 shows typical regression line plots for determining the Weibull parameters in the case of glass fibre No. 2. It is to be noted that Fig. 2a refers to the observed strength data, whereas in Fig. 2b strength values calculated using Equation 5 have been used. The values of the Weibull parameters corresponding to both observed strength (S) as well as calculated strength (S_c) for the different glass fibres as determined by GRE and MLE techniques are summarized in Table III. In Figs 3a and b comparisons of typical Weibull distributions as obtained by GRE and MLE techniques with the experimental data are shown.

The mean values $\bar{S}$ and $\bar{S}_c$ for the different samples corresponding to the Weibull parameters estimated by GRE and MLE techniques are summarized in Table IV.

Fig. 4 is a scanning electron micrograph of a typical fracture surface of glass fibre No. 3. A unique feature of the fibres of glass No. 3 is the presence of holes of diameters ranging from 0.1 to 1.5 μm. Such holes are not observed in the fractographs of other fibres. Another interesting feature is that a part of fracture surface is nearly perpendicular to the tensile axis, while the remaining part is parallel to the same. Such changes in the fracture path are not, however, observed in glasses 1 and 2.

5. Discussion

From Table I it is evident that the Young's modulus estimator $\bar{E}_R$ from strength–strain regression analysis is higher than the simple arithmetic mean $\bar{E}$ obtained from the individual fibre data. $\bar{E}_R$ is believed to be the better estimator of the two because it has been obtained from the slope of the regressed strength–strain line which should effectively represent the hypothetical stress–strain curve for the given material. A further confirmation of this conclusion is provided by the $\bar{E}_R$ values in different glass fibres containing varying

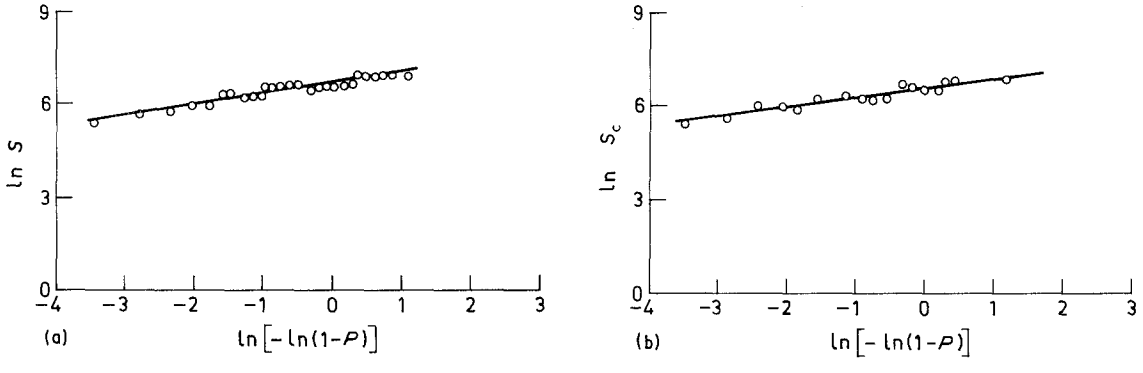

Figure 2 Regression of strength with failure probability for glass fibre No 2. (a) Observed strength (S); $b = 3.02$, $S_0 = 666$ MPa. (b) Calculated strength (S_c); $b = 2.94$, $S_0 = 719$ MPa.

TABLE III Weibull parameters for different glasses

Glass No.	GRE				MLE			
	S		S_c		S		S_c	
	b	S_0 (MPa)	b	S_0 (MPa)	b	S_0 (MPa)	b	S_0 (MPa)
1	1.69	892	1.55	734	2.16	850	1.52	714
2	2.94	719	3.02	666	3.51	708	2.95	617
3	3.50	681	4.65	666	3.00	688	5.59	632

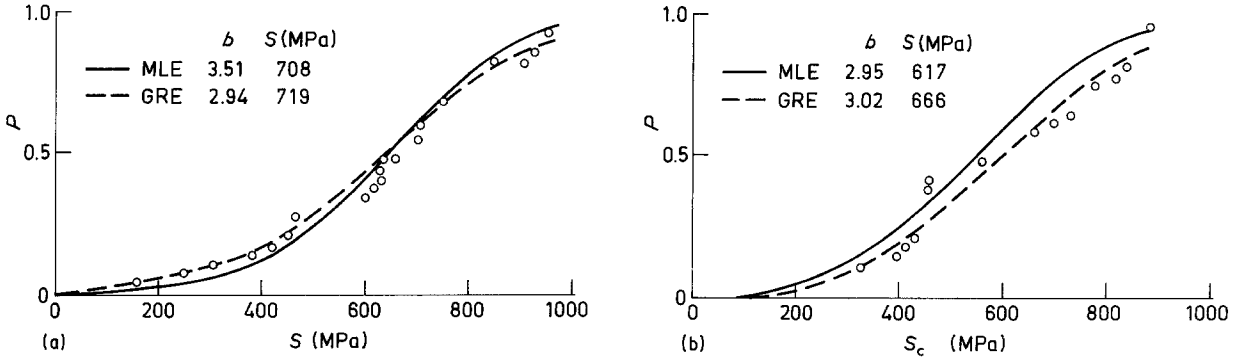

Figure 3 Variation of failure probability (P) with (a) observed strength (S) and (b) calculated strength (S_c) for glass fibre No 2.

amounts of aluminium. $\bar{E}_R$ shows an increase as the aluminium content in the glass is increased, which is the expected behaviour of such composites. Values of $\bar{E}$, however, show just the opposite trend.

The above discussion also tends to imply that the mean strength value $\bar{S}_c$ as estimated from the calculated values using E_R is more representative of a particular fibre than $\bar{S}$. In fact, from the data given in Table IV it appears that the strength distribution as obtained from S_c values is somewhat sharper than that derived from S. This is probably because the error due to possible uncertainties in strength measurements of the individual fibres are averaged out during the regression analysis step.

The failure probability distributions (Fig. 3a) for the GRE and MLE formalisms show a crossover point at some strength value when observed strengths S are used. The corresponding curves when values of the calculated strength S_c are used do not, however, in general show such behaviour. In the latter case, the MLE technique gives estimators of b and S_0 such that the probability of failure is higher than that predicted by the GRE procedure. From an engineering point of view, therefore, MLE formalism in the present case seems better than GRE.

An increase in the aluminium concentration in glass fibres leads to a lowering of the strength, both observed ($\bar{S}$) as well as calculated ($\bar{S}_c$) (Table IV). This is believed to arise due to the aluminium particles acting as inclusions or stress concentrators in the glass matrix, thereby reducing the fibre strength. This is substantiated by the fracture surface micrographs. The observed surface morphology arises because of a complex stress distribution due to the presence of holes and the nearby flaws. The holes observed in the fractograph of glass fibre No. 3 seem to be the sites where large aluminium particles are embedded in the glass matrix. These particles have either been dislodged during the process of fracture or they may have caused the formation of small cylindrical cavities during the fibre drawing operation.

TABLE IV Mean strength values of different glass fibres

Glass No.	GRE		MLE	
	$\bar{S}$ (MPa)	$\bar{S}_c$ (MPa)	$\bar{S}$ (MPa)	$\bar{S}_c$ (MPa)
1	796 ± 483	661 ± 438	753 ± 365	644 ± 433
2	642 ± 237	595 ± 214	637 ± 204	551 ± 204
3	612 ± 196	608 ± 152	615 ± 221	584 ± 121

6. Conclusions

1. Evaluation of Young's modulus based on strength–strain regression analysis in the case of glass fibres gives a better estimate than that obtained from a simple averaging of individual data.

2. Glass fibres containing metallic aluminium dispersoids have lower strengths as compared to those of fibres without any metallic inclusion. This arises

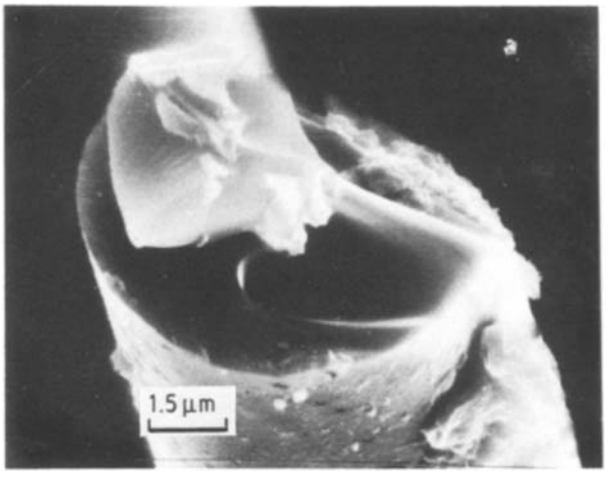

Figure 4 Scanning electron micrograph for the fracture surface of glass fibre No. 3.

due to the metal particles acting as stress concentrators within the glass.

Acknowledgement

Part of this research was supported by the Department of Science and Technology, Government of India.

References

1. D. A. KROHN and A. R. COOPER, *J. Amer. Ceram. Soc.* **52** (12) (1969) 661.
2. A. R. COOPER and D. A. KROHN, *ibid.* **52** (1969) 665.
3. D. CHAKRAVORTY, B. N. KESHAVARAM and A. VENKATESWARAN, *J. Mater. Sci.* **14** (1979) 2991.
4. V. D. KRISTIC and P. S. NICHOLSON, *J. Amer. Ceram. Soc.* **60** (1977) 467.
5. V. D. KRISTIC, P. S. NICHOLSON and R. C. HOAGLAND, *ibid.* **64** (9) (1981) 499.
6. C. STEPHANIE and KUNZ, *ibid.* **66** (4) (1983) c73.
7. D. CHAKRAVORTY, R. BHATNAGAR and B. SHARMA, *Mater. Eng.* **2** (3) (1981).
8. W. F. THOMAS, *Phys. Chem. Glasses* **1** (1960) 4.
9. A. SHRIVASTAVA, PhD thesis, Indian Institute of Technology, Kanpur (1985).
10. W. WEIBULL, *J. Appl. Mech.* **18** (1951) 293.
11. J. F. LAWLESS, "Statistical Models and Methods for Lifetime Data" (Wiley, New York, 1982) p. 299.
12. J. M. McCORMICK and M. G. SALVADORI, "Numerical Methods in Fortran" (Prentice-Hall, Englewood Cliffs, New Jersey, 1964).

Received 5 August
and accepted 1 December 1986