Relation between shear strength at the fibre-matrix interphase and shear properties of resin matrix

M. MIWA, A. TAKENO, K. YAMAGUCHI, A. WATANABE *Faculty of Engineering, Gifu University, 1-1 Yanagido, Gifu, Japan, 501-11*

The shear yield strength and the shear strength of a resin matrix increase almost linearly as the logarithm of the strain rate increases. This increasing tendency is almost the same at various temperatures. The strain rate–temperature superposition held and an experimental equation was found to estimate the strain rate and temperature dependence of these shear properties. The strain rate and temperature dependence of the shear yield strength at the fibre-matrix interphase can be also estimated by the same equation. A strong quantitative relation was observed between the strain rate and temperature dependence of the shear properties of a resin matrix and that of the shear yield strength at the fibre-matrix interphase.

1. Introduction

In any discontinuous fibre-reinforced resin, loads working on the composite are transmitted to the fibre through the fibre-matrix interface. Consequently, mechanical properties of the composite are greatly influenced by the shear strength at the fibre-matrix interface. Hence, in discontinuous fibre-reinforced resins, precise determination of shear strength at the interface is of great importance.

We reported a method for directly measuring the shear yield strength at the fibre-matrix interphase, i.e., if a sufficiently long fibre is embedded in the resin matrix and the system is elongated, the fibre eventually breaks into many pieces. By measuring the lengths of the broken pieces, the shear yield strength at the fibre-matrix interphase for the system can be estimated if it is assumed that the tensile strength of the fibre is both uniform $\lceil 1 \rceil$ and variable $[2, 3]$. Using the second assumption $[2]$, we were clearly able to explain the strain rate and temperature dependence of both the shear yield strength at the interphase and the critical fibre length for glass fibre-thermoplastic resin systems [3], glass fibre-thermosetting resin systems [4], and carbon fibre-epoxy resin systems [5]. We reported that the strong relation was observed between the temperature dependence of the shear yield strength at the interphase and that of the shear yield stress of the resin matrix obtained by torsion test $[3, 5]$.

In this paper, we investigated the relation between the strain rate and temperature dependence of the shear yield strength at the interphase, obtained using our method [2], and the shear properties of the resin matrix in glass fibre-epoxy and carbon fibre-epoxy resin systems.

2. Experimental procedure

Epoxy resin used in this study was the same as that described in a previous paper $[4, 5]$; a bisphenol "A" type epoxy resin (Epikote 828, Yuka Shell Epoxy) and an amine type curing agent (S-Cure 661, Kayaku Akuzo) were used to prepare the specimens.

First, the 100 parts epoxy resin and 10 parts curing agent were mixed. This mixture was agitated thoroughly and then defoamed under vacuum at 30° C for 20 min. Next, this mixture was poured into a mould and subjected to curing at 65° C for 17 h and postcuring at $114\degree C$ for 5 h. The specimen was then allowed to cool to room temperature.

Test specimens were cut from the specimens prepared in this manner in accordance with JIS K 7214. The dimension of a test specimen measured 3 mm in thickness, 60 mm in width and 60 mm in length. Shear test (JIS K 7214) was carried out by a circular punch. For the shear test, an 11 mm diameter hole was made in the centre of the test specimens.

In order to investigate the strain rate and temperature dependence of the shear properties of an epoxy resin matrix, each specimen was subjected to a shear test at various strain rates and temperatures with the aid of a Tensilon UTM-I-2500 (Orientec). More than ten specimens were tested for each test condition.

3. Results and discussion

The relationship between shear yield strength $\tau_{m,y}$ and shear strength $\tau_{m,s}$ at various temperatures and strain rates is shown in Fig. l(a) and (b), respectively. At all temperatures, $\tau_{m,y}$ and $\tau_{m,s}$ increase almost linearly with the increase in the logarithm of the strain rate. This tendency is the same as that found for the tensile properties [6].

Figure 1 Relation between strain rate and shear property as a function of temperature for epoxy resin. (a) Shear yield strength. (b) Shear strength. O, 20 °C; \bullet , 40 °C; \triangle , 60 °C; \blacktriangle , 80 °C; \square , 100 °C.

Figure 2 Shear yield strength (lower curve) and shear strength (upper curve) master curves plotted against shifted strain rate for epoxy resin. O, 20 $^{\circ}$ C; \bullet , 40 $^{\circ}$ C; \triangle , 60 $^{\circ}$ C; \bullet , 80 $^{\circ}$ C; \Box , 100 $^{\circ}$ C; values calculated from Equation 1.

Data in Fig. l(a) and (b) have been shifted along the logarithm of the strain rate axis to obtain master curves of the shear properties, as show in Fig. 2. In Fig. 2, the reference temperature is 60° C. Although a slight deviation exists in both cases, data can be reduced into a single master curve, implying that the strain rate-temperature superposition holds for the shear yield strength and the shear strength of epoxy resin.

The shear yield strength $(\tau_{m,y})_{\hat{\epsilon},\hat{T}}$ and shear strength $(\tau_{m,s})_{\dot{\varepsilon},\Upsilon}$ shown in Fig. 2 increase almost linearly with the increase in the shifted strain rate. This tendency is the same as that seen in results of tensile yield stress of some macromolecular materials [7,8] and glass beadfilled composites $[9]$. We have also found this same tendency for shear yield strength at the fibre-matrix interphase in glass fibre-thermosetting resin systems $[4]$ and carbon fibre-epoxy resin systems $[5]$. Namely, the shear yield stress $(\tau_{m,y})_{\varepsilon,T}$ and shear strength $(\tau_{m,s})_{\varepsilon,T}$ of epoxy resin used in this experiment increase in proportion to the logarithm of the shifted strain rate $log(ia_T)$. The strain rate and temperature dependence of the shear yield strength $(\tau_{m,y})_{\dot{\epsilon},\tau}$ and the shear strength $(\tau_{m,s})_{\epsilon,T}$ can be estimated by the following equation

$$
\tau_{\dot{\epsilon},T} = K_1 + K_2 \log{(\dot{\epsilon} a_T)} \tag{1}
$$

where $\tau_{\varepsilon, T}$ is the shear yield strength or the shear strength, $\dot{\epsilon}$ is the strain rate, a_T is the shift factor, and K_1 and K_2 are constants depending on the materials

TABLE I Constants of Equation 1 for shear properties of epoxy resin

Properties	Constants K_{1}	(MPa) Κ,
Shear yield strength	52.2	3.83
Shear strength	34.9	2.82

Figure 3 Shear yield strength master curve at the glass fibre-epoxy resin interphase plotted against shifted strain rate. O, 30 °C; \bullet , 40 °C; \triangle , 60 °C; \triangle , 80 °C; \square , 100 °C; values calculated from Equation 1. Upper curve, good bonding; lower curve, poor bonding.

Figure4 Shear yield strength master curve at the carbon fibre-epoxy resin interphase plotted against shifted strain rate. O, 20 °C; ●, 40 °C; △, 60 °C; ▲, 70 °C; □, 80 °C; ■, 90 °C; ◇, 100 °C; values calculated from Equation 1. Upper curve, good bonding; lower curve, poor bonding.

and the reference temperature. K_1 and K_2 values obtained from Fig. 2 are shown in Table I. The solid lines in Fig. 2 are values calculated by Equation 1. The calculated values agree approximately with the experimental values within the logarithm of the shift strain rate of -10 min⁻¹ to $+8$ min⁻¹. The strain rate and

TABLE II Constants of Equation 1 for shear yield strength at the fibre-matrix interphase

System	Interphase condition	Constants K_1	(MPa) K_2
Glass fibre-epoxy resin	Good bonding	38.1	3.30
	Poor bonding	26.2	2.13
Carbon fibre-epoxy resin	Good bonding	41.1	2.42
	Poor bonding	34.0	1.25

Figure 5 Shift factors with the reciprocal of temperature. (a) Shear properties of epoxy resin. (b) Shear yield strength at the fibre-matrix interphase. O, shear yield strength; \bullet , shear strength; \triangle , \blacktriangle , glass fibre-epoxy resin system; \Box , \blacksquare , carbon fibre-epoxy resin system; \triangle , \Box , good bonding; \blacktriangle , \blacksquare , poor bonding.

temperature dependence of both the shear yield strength and shear strength of epoxy resin is given by Equation 1 for this experimental strain rate and temperature range.

As mentioned above, we have reported on the strain rate and temperature dependence of shear yield strength at the fibre-matrix interphase for glass fibre–epoxy $[4]$ and carbon fibre–epoxy resin systems [5]. Master curves of interphasic shear yield strength for those systems are shown in Figs 3 and 4. In Figs 3 and 4, the reference temperature is also 60° C. The fibres used in these systems are carbon fibre (Pyrofil, T1, 8.83 µm in diameter, Mitsubishi Rayon) and "E" glass fibre (R2220, MA859, XL16, 12.73 µm in diameter, Asahi Fiber Glass). "Good bonding" in Figs 3and 4 shows the systems in which the surface of the glass fibre is treated with a silan coupling agent $(KBM 403, Shin-etsu Chemical) [1, 4]$, the carbon fibre is oxidized electrolytically, and the surface of the fibre is sized with a bisphenol "A" type epoxy oligomer. "Poor bonding" shows the systems in which the surface of the glass fibre is treated with a release agent (KS 707, Shin-etsu Chemical) $[1, 4]$, the sized carbon fibre is immersed in an acetone solution, and the sizing of the surface of the fibre is dissolved [5]. For both systems, regardless of the nature of the interphasic treatment, these master curves increase almost linearly with the logarithm of the shifted strain rate. In the same manner as mentioned above for the shear properties of the resin matrix, equation 1 holds for shear yield strength at the interphase. Constants K_1

and K_2 obtained in previous papers [4, 5] from Figs 3 and 4 are shown in Table II.

Comparing Fig. 2 with Figs 3 and 4, the value of the shear yield strength at the fibre-matrix interphase, for a good bonding system, is between the shear yield strength and the shear strength of the resin matrix. Furthermore, the gradient, K_2 (Tables I and II), for the logarithm of the shifted strain rate, is almost the same. Accordingly, a strong quantitative relation can be observed between the shear yield strength at the fibre-matrix interphase (in which the surface of the fibre is treated with a suitable coupling agent) and the shear properties of the resin matrix. The shear yield strength at the interphase for a poor bonding system is lower than the shear yield stress of the resin matrix. As reported in a previous paper [1], it can therefore be inferred that the shear yield strength for a poor bonding system is primarily attributed to a secondary bond, e.g., a hydrogen bond and Van der Waal's force, etc., at the fibre-matrix interface.

The relationship between the logarithm of the shift factor in order to obtain these master curves and the reciprocal of absolute temperature is shown in Fig. 5(a) and (b). For both the shear properties of the resin matrix and the shear yield strength at the interphase, although there is an inflexion at temperatures in the region of 60° C, the logarithm of the shift factor increases almost linearly with an increase in the reciprocal of absolute temperature. It is thus recognized that a temperature dependence of Arrhenius type holds on the shift factor. Apparent activation energy

TABLE III **Apparent activation energy**

AH obtained from the gradient of the straight lines is shown in Table III. In each temperature range, the ΔH **obtained from the shear properties of the resin matrix is almost the same as that obtained from the shear yield strength at the fibre-matrix interphase. The inflection point may be caused by the glass transition of the resin matrix.**

4. Conclusions

Attempts were made to discuss the relationship between the shear yield strength at the fibre-matrix interphase, which plays an essential role in determining the reinforcing effect in discontinuous fibre-reinforced resins, and the shear properties of the resin matrix.

The shear yield strength and the shear strength of a resin matrix increase almost linearly as the increase in the logarithm of the strain rate. This increasing tendency is almost the same at various temperatures. The strain rate and temperature dependence of these shear **properties can be estimated by the following equation**

$$
\tau_{\hat{\epsilon},T} = K_1 + K_2 \log{(\hat{\epsilon} a_T)}
$$

where $\tau_{\varepsilon, T}$ is the shear yield strength or the shear strength, $\dot{\epsilon}$ is the strain rate, a_T is the shift factor. K_1 and K_2 are constants depending on the materials and **the reference temperature.**

The strain rate and temperature dependence of the shear yield strength at the fibre-matrix interphase can **also be estimated by the above-mentioned equation.** The shift factor a_T and constants K_1 and K_2 are **almost the same as those obtained for the shear properties of a resin matrix. A strong quantitative relation is observed between the strain rate and temperature dependence of shear properties of a resin matrix and that of the shear yield strength at the fibre-matrix interphase.**

References

- 1. T. OHSAWA, A. NAKAYAMA, M. MIWA **and** A. HASEGAWA, *J. Appl. Polym. Sci.* 22 (1978) 3203.
- 2. M. MIWA, T. OHSAWA and K. TAHARA, *Ibid.* 25 (1980) 795.
- 3. M. MIWA, T. OHSAWA and A. TOMITA, *Kobunshi Ronbunshu* 41 (1984) 353.
- 4. M. MIWA, T. OHSAWA, K. HATTORI and Y. SHUKUYA, *Sen-i Gakkaishi* 35 (1979) T-190.
- 5. M. MIWA, T. OHSAWA and Y. ADACHI, *ibid.* 41 (1985) T-223.
- 6. L. E. NIELSEN, **in "Mechanical Properties of Polymer and Composites", (Mercel** Dekker, Inc., New York, 1974) p. 266.
- 7. J. J. LOHR, *Trans. Soc. Rheol.* 9 (1965) 65.
- 8. A. E. MOEHLENPAH, O. ISHAI and A. T. DIBENEDETTO, *J. Appl. Polym. Sci.* 13 (1969) 123l.
- 9. E. NICOLASIS and M. NARKIS, *Polym. Eng. Sci.* 11 (1971) 194.

Received 12 October 1993 and accepted 8 September 1994