

# INFLUENCE OF STRESS STATE AND TEMPERATURE ON THE STRENGTH OF EPOXY RESINS

BODO FIEDLER<sup>1</sup>, THOMAS HOBBIEBRUNKEN<sup>2</sup>, MASAKI HOJO<sup>2</sup>, KARL SCHULTE<sup>1</sup>

<sup>1</sup>Polymer Composites Section, Technical University Hamburg-Harburg, Germany

<sup>2</sup>Department of Mechanical Engineering, Graduate School of Engineering, Kyoto University, Japan

## ABSTRACT

The yield and fracture behaviour of the neat resin was investigated. The parabolic failure criterion was applied to experimental results. From a neat resin slab, specimens for the tensile-, torsion- and compression - tests were manufactured and the failure behaviour of the neat resin due to the actual stress-state was tested at low and elevated temperatures and discussed in detail. The results of the mechanical tests and the fractographic study of the fracture surfaces were correlated to the stress-state dependent ultimate strength of epoxy resins. This plays an important role in fibre reinforced composites, because just after cooling to room temperature the resin matrix is in a tri-axial residual stress-state. The mechanical properties of the neat resin described by the parabolic failure criterion is able to explain the low strain to failure of unidirectional laminates under transverse tensile loading.

## 1 INTRODUCTION

It is often observed that the deformation behaviour of polymers is different in tensile, compressive and shear loading [1,2]. Ductility, plastic flow and fracture are found to be a function of the state of stress, strain, strain rate, temperature and environment [3]. In previous publications the failure behaviour of the neat resin was tested and discussed in detail [4,5]. In this study further experimental investigations of the fracture behaviour of neat resins were extended to several high performance epoxy resins used for aircraft application and compared with a room temperature (RT) curing system. Furthermore, the influence of test temperature and loading condition was investigated in detail. From a neat resin slab, specimens for tensile-, torsion- and compression - test were manufactured and the failure behaviour of the neat resin was obtained. The tensile and compression tests were performed from low temperature ( $T=-50^{\circ}\text{C}$ ) to elevated temperatures close to the glass transition temperature TG. In case of the compression test it was possible to reduce the friction between the specimens surfaces and the anvils by using a thin polymer film as lubricant according to similar investigations carried out for testing of rocks [6].

From the engineering stress strain curve of the tensile and compression tests true stress strain curves were calculated. Since the cross-sectional area of specimens tested in torsion does not change, as in tension and compression, the engineering stress-strain curve for shear is approximately the true stress-strain curve.

The mechanical tests were carried out from  $T=-50^{\circ}$  to elevated temperatures. The results were correlated to the stress-state dependent strength and fracture stress of epoxy resin. With regard to the test temperature, the parabolic failure criterion was applied to experimental results and describes the ultimate strength of epoxy resins well.

Besides the phenomenological parabolic criterion, the physical reasons for the different strength values of the neat resin under tensile, compressive and shear load are discussed regarding to stress concentration of tensile stresses generated at defects.

## 2 EXPERIMENTAL

The materials under consideration are 5 different epoxy resins. The high performance hot curing ( $T=180^{\circ}\text{C}$ ) resins are represented by RTM-6, 6376 (both Hexcel), and 997-2 (Cytec). The intermediate type resin was cured at  $120^{\circ}\text{C}$  LY556/HY932 (Ciba Geigy) and finally a low viscosity RT curing epoxy resin L135i (MGS), which was cured at room temperature (RT) for 24

hrs followed by a post curing cycle of 24 hrs at 60°C. Neat resin slabs were cured according to the corresponding manufacturers' instructions. Dog bone tensile specimens, tubes for the torsion tests and cubes for the compression tests were prepared from the neat resin slabs to ensure identical resin and curing conditions for all specimens. The surfaces of the specimens were mirror like polished to minimize the influence of surface flaws on the mechanical properties. The dimensions of the specimens were similar to those used in our former work [4]. The tensile-, torsion- and compression tests of the neat resins were performed on a universal-testing machine. All tests were carried out at room temperature (RT). The compression and tensile tests were also performed at low and elevated temperatures. For all tests, the crosshead speed was 1 mm/min. The strain was measured by strain gages (HBM) or extensometer (MTS). At least five specimens were used for each test with each resin.

As a representative example the resulting stress/strain curves for the neat resin L135i are shown in Fig. 1a. As expected, the strength is decreasing with the strain to fracture, respectively start of necking and is increasing with increasing temperature. A maximum strength value of  $\sigma_s=80\pm 12\text{MPa}$  was obtained at  $T=-40^\circ\text{C}$  with a corresponding minimum strain value of  $\varepsilon_s=2.3\pm 0.2\%$ . In Fig. 1b all measured ultimate tensile strength (UTS) data are plotted via the corresponding test temperature. The test temperature was normalized to the glass transitions temperature (TG) of the individual epoxy resin. The TGs were measured by dynamic thermal mechanical analysis (DMTA). The temperature at the maximum of the loss modulus  $E''$  was taken for TG. For the epoxy resins under consideration the values are given in Tab. 1.

Table 1: Glass transition temperature of the epoxy resins.

Epoxy resin	L135i	LY556	977-2	6376	RTM-6
TG [°C]	90	141	195	210	220

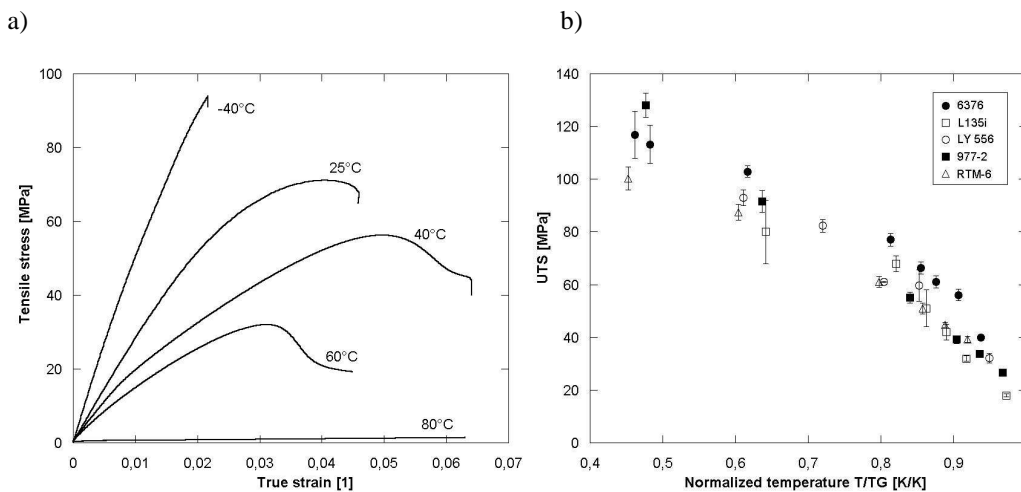


Figure 1: a) Stress–strain curves of epoxy resin L135i measured at different temperatures; b) UTS of all five epoxy resins at different test temperatures normalized to the corresponding glass transition temperatures.

The strength values of the different resins are different and of course decrease with increasing temperature. The influence of the test temperature on the UTS is strongly influenced by the TG

and the ratio of T/TG controls the UTS (Tab. 2) and yields to a total linear decrease for all five resins (Fig. 1b). The influence of the T/TG ratio on the strength is not surprising. Hence, the slope of a linear fit of all data points is  $\Delta = -167$  MPa with a correlation coefficient of  $R^2=0.91$ . The influence of the temperature on the ultimate tensile strength of epoxy resin can be approximated by a simple linear equation. Only the strength at a certain temperature and the TG of resin must be known.

The detailed description of the torsion test is given elsewhere [4]. In case of the L135i resin the shear stress increased linearly with increasing shear strain. After reaching a shear strength of  $\tau=51.1\pm 3.1$  MPa the resin deformed plastic under this pure shear condition. Beyond the maximum the stress decreased slightly and reached nearly constant values with increasing strain. The shear strain to failure is  $\gamma=27.3\pm 13.5$  %. In case of the 6376 epoxy resin a shear strength of  $\tau=90\pm 3.5$  MPa and a corresponding strain of  $\gamma=12.3\pm 2.3\%$  was measured. For all other resins the general mechanical behaviour was very similar.

For all five resins the final fracture of the destructed specimen occurred along the plane perpendicular to the direction of the maximum tensile stress under an angle of  $45^\circ$  to the longitudinal and transverse direction and showed the helical shape, which is typical for a brittle failure under torsion.

When performing the compression test, the friction between the anvils and specimens surfaces was strongly reduced by inserting thin polymer films, acting like a lubricant between the surfaces [4]. In case of the L135i resin the specimens reached a true strength value of  $\sigma_s = 104.7\pm 8.2$  MPa at an applied strain of  $\epsilon_s = 51.1\pm 1.3\%$  at RT. At the same temperature the strength of the 6376 resin is  $\sigma_s = 264\pm 13$  with a corresponding strain of  $\epsilon_s = 44.4\pm 0.5\%$ .

As representatives for all investigated resins the obtained strength values of the resins 6376 and L135i are summarised in Tab. 2.

Table 2: Compressive-, tensile- and shear strength at different temperatures of the neat resins L135i and 6376.

Material	Temperature [°C]	Compressive strength [MPa]	Tensile strength [MPa]	Torsional strength [MPa]
Epoxy resin L135i	-40	139.6±14.2	80±12	-
	25	104.7±8.2	68±3	51.1±3.16
	40	-	51±7	-
	50	95.5±4.4	42±3	-
	60	-	32±1	-
	80	-	2±0.3	-
Epoxy resin 6376	-50	290±14	116.9±8.9	-
	25	264±13	103±2.3	90±3.5
	90	173±5	-	-
	120	-	77±2.4	-
	150	153±0.7	61.1±2.3	-
	160	-	56.1±2.2	-
	180	-	39.9±0.7	-

### 3 PARABOLIC FAILURE CRITERION

*Mohr's* general failure criterion includes the dependence on the hydrostatic component of the stress-state; it has than a parabolic shape [7]. Its mathematical formulation (Eq. 3) was carried out by Tschoegl [8] and is a linear combination of the mean normal stress and the square of the octahedral shear stress (Eq. 3). The octahedral stresses ( $\sigma_0$ ,  $\tau_0$ ) in the principal stress space are given by Eq. 1 and 2 [9].

$$\sigma_0 = \frac{\sigma_1 + \sigma_2 + \sigma_3}{3} \quad (1)$$

$$\tau_0 = \frac{\sqrt{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_1 - \sigma_3)^2}}{3} \quad (2)$$

$$\tau_0^2 = A_1 - A_2 \cdot \sigma_0 \quad (3)$$

According to Eq. 3 the values of the parameters of the L135i resin are  $A_1=1558 \text{ MPa}^2$  and  $A_2=24.6 \text{ MPa}$  at RT, the correlation coefficient is  $R^2=0.997$ . For the other temperature only 2 different loading conditions were performed and the parabolic failure criterion exactly fits the data. In case of the 6376 resin an  $A_1=5144 \text{ MPa}^2$  and  $A_2=113$  was obtained for the RT values, also in this case Eq. 3 fits the strength values obtained by 3 different loadings. The correlation coefficient is calculated to  $R^2=0.964 \text{ MPa}$ .

It could be argued, that the parabolic criterion is not based on physical phenomena. At present no model is able to describe the observed phenomena and it is ambiguous how the hydrostatic stress influences the mechanical behaviour. However, the experimental results follow the parabolic criterion.

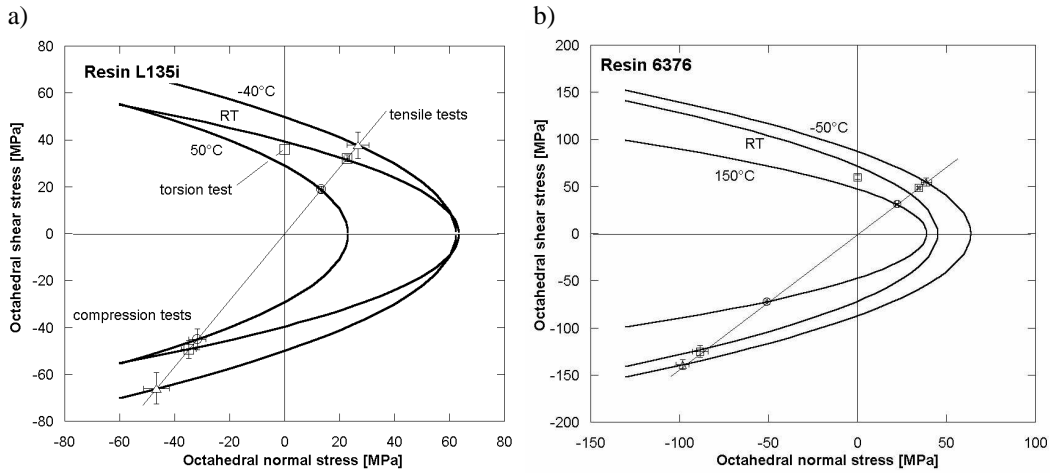


Figure 2: Experimental strength data and parabolic fracture curves for epoxy resins at different test temperatures L135i (a) and resin 6376 (b).

As shown, in Fig. 2 the parabolic criterion is applied to both neat resins. The curves are based on the experimental results and the calculated true stresses and strains. In case of the other 3 resins the results are in general the same. It can be said that the strength at different temperature and loading conditions of epoxy resins can be described by the parabolic failure criterion, which includes hydrostatic stress components to describe the strength.

## 5 SUMMARY

The tensile strength of all bulk resins depends linearly on the test temperature with respect to the particular glass transition temperature (T/T<sub>G</sub>).

Brittle epoxy resins show an extended plastic deformation under shear conditions before fracturing in a normal stress controlled brittle manner.

The fracture depends on the loading conditions and is influenced by the amount of tensile stresses. Hence the yielding of the epoxy resins is controlled by the acting shear stress.

The parabolic criterion includes the influence of the stress state on the final failure. The failure envelope can describe the experimentally observed strength data obtained by tensile, torsion and compression test at low and elevated temperatures.

## 6 ACKNOWLEDGEMENT

This work was supported in the framework of the collaborative program of the German Research Foundation (DFG) and the Japan Society for Promotion of Science (JSPS) and in the frame of the Center of Excellence (COE) program of the Ministry of Education, Sports, Science and Technology, Japan.

## 6 REFERENCES

- [1] Bowden, P. B., Jukes, J. A., The Plastic Flow of Isotropic Polymers, *Journal of Materials Science*, Vol 7, pp. 52-63, 1972.
- [2] Asp, L. E., Berglund, L. A., Talreja, R., A Criterion for Crack Initiation in Glassy Polymers Subjected to a Composite Like Stress State, *Composite Science and Technology*, 56,1291-1301, 1996.
- [3] Manjoine, M., J., *Multiaxial Stress and Fracture*, Fracture, Vol. 3, pp. 265-309 (Liebowitz, Editor), Academic Press, New York, London 1971.
- [4] Fiedler, B., Hojo, M., Ochiai, S., Schulte, K., Ando, M., Failure behavior of epoxy matrix under different kinds of static loading; *Composites Science and Technology*, Vol 61 No. 11 pp 1615-1624, 2001.
- [5] Fiedler, B., Hojo, M., Ochiai, S., The Parabolic failure Criterion applied to Epoxy Resin, *Proc. of 4<sup>th</sup> Mesomechanics*, Aalborg University, Denmark, August 26-30, pp. 533-539, 2002
- [6] Rinne, F., Vergleichende Untersuchungen über die Methoden zur Bestimmung der Druckfestigkeit von Gesteinen, *Neues Jahrbuch für Mineralogie*, No. 12, 121 – 128, 1909.
- [7] Bosch, M. ten, *Lecture of machine elements*, Part 1, J. Springer, Berlin 1929.
- [8] Tschoegl, N. W., Failure surfaces in Principal Stress Space, *Journal of Polymer Science, Part C, Polymer Symposia*, No. 32, pp. 239-267, 1971.
- [9] *Dubbel: Handbook for Mechanical Engineers*, edited by Beitz, W., Küttner, K.-H., Springer-Verlag: Berlin, Heidelberg, New York, London, Paris, Tokyo, 16. Edition, (1987), (in German).