



## Rapid evaluation of long-term thermal degradation of carbon fibre epoxy composites

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### ABSTRACT

Two commercially available carbon fibre reinforced composites (8552/IM7 and M18-1/G939) were exposed to heat above maximum operational temperature at various durations. Mass loss and mechanical properties were measured over time. A chemical analysis was also performed on these composites. The two primary components of each matrix, the epoxy resin and the thermoplastic, were observed to degrade at different rates under various thermal loading conditions. The epoxy resins degrade predominantly as measured by IR spectroscopy and thermal desorption/gas chromatography mass spectrometry. By using mass loss, strength, and IR spectroscopic data, a correlation was made between strength characteristics of each composite and the relative amount of the two primary matrix components. The developed relationship can be used to estimate rapidly the mechanical properties from the intensity ratio of IR bands characteristic of the two components.

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### 1. Introduction

Knowledge of long-term behaviour of high-performance composites is essential for the application in a higher temperature environment. Especially epoxy resin based composites are widely used as laminates for aerospace, ballistic, engineering components, etc. because of their excellent bonding, thermal and mechanical characteristics. Similar to any other material, mechanical properties depend on various parameters. Environmental influences such as moisture absorption are the most crucial, because they can lead to a plastification of the matrix material. Fundamental investigations have been done on the number of factors influencing moisture uptake of fibre reinforced composites [1]. While these materials have been in use for many years, only limited data is available concerning the influence of the long-term thermal loading.

When composites are heated to temperatures in the range of glass transition temperature, thermo-mechanical effects due to softening and/or decomposition of the polymer may occur. Degradation is even more critical when the material is heated in an oxidising environment than in a vacuum or inert atmosphere [2,3]. It has been found that degradation is associated with mass loss from different surfaces at different rates [4–6]. Matrix cracking and delamination have been shown to occur after thermal exposure by many authors [7–12].

Skourlis used fibre fragmentation tests at different temperatures to show that the interphase has a lower glass transition temperature than the surrounding resin [13]. This results in a faster degradation of the interphase when the composite is exposed to higher temperatures. These investigations were predominately performed in temperature ranges above the glass transition temperature.

A decrease in mechanical strength, a severe thermal damage by cracks and delamination, and high mass losses have been reported for 8552/IM7 when the material was heated at 340–450 °C for 30 min [14].

For this investigation, the goal was to obtain an initial understanding of the temperature effects on two different composites (8552/IM7, M18-1/G939) that are common within the aerospace and other industries. Therefore specimens were exposed to isothermal conditions in the range of the glass transition/maximum operational temperature for various durations, comparable to other studies [15], also including accelerated aging methods [16,17]. Mechanical tests and chemical analyses were performed to characterise the degradation. The thermal aging of 8552/IM7 and comparable model type systems were previously investigated by IR spectroscopy with respect to the changes of the matrix [18–20]. Nevertheless the focus of these studies has been to give a detailed mechanism for the thermal degradation of the epoxy resin in the matrix. Also, we consider the tougheners and provide information on volatile degradation products additionally. Furthermore an empiric method is developed to predict mechanical composite properties on the basis of changes in the matrix composition, rapidly analysed by IR spectroscopy.

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## 2. Material

All tests were carried out with the carbon fibre reinforced epoxy systems 8552/IM7 and M18-1/G939 from Hexcel Composites GmbH (Stade, Germany). The laminates were cured in an autoclave according to the manufacturer's recommended conditions [21].

The 8552/IM7 laminates consisted of 16 UD-ply denoting an application oriented quasi-isotropic (QI) lay-up with  $[0/90/+45/-45]_{25}$  fibre orientations for the tensile and compression test specimen. Unidirectional (UD) lay-up was chosen for Interlaminar Shear Strength (ILSS) specimen. The M18-1/G939 laminates were made of eight plies denoting a QI lay-up with  $[(0/90)/(90/0)/(+45/-45)/(-45/+45)]_5$  fibre orientations for the tensile and compression, and a  $[(0/90)]_8$  lay-up for ILSS test specimen. All laminates were visually inspected for any surface defects. Ultrasonic C-scans were performed to ensure that the test laminates were free of delamination, voids and fibre orientation errors. After processing, the laminates were cut into samples of 250 mm  $\times$  100 mm with a water-cooled diamond wheel saw. The samples were redried at 70 °C.

The matrix systems 8552 and M 18-1 consist of aromatic epoxy resins, which are toughened with the temperature resistant thermoplastics polyethersulfone and polyetherimide, respectively [22].

## 3. Experimental

The samples were isothermally aged in standard convection ovens at 180 °C, 190 °C and 200 °C. They were removed and cooled down to room temperature for measuring the mass loss and for testing at various time intervals up to approximately 500 days. The masses were recorded with an accuracy of 0.0001 g.

Tensile tests were performed in accordance with DIN EN 2561, compressive tests with DIN EN 2850A and ILSS tests with DIN EN 2563.

Changes in the composition of the polymer matrix were analysed by micro-attenuated total reflection (ATR) fourier transform infrared spectroscopy (FTIR). Spectra were recorded from the surface and at least 500  $\mu$ m under the surface (bulk). Volatile products formed by thermal degradation and oxidation reactions were explicitly identified by thermal desorption gas chromatography/mass spectrometry (TD-GC/MS) after desorption at 200 °C.

Fracture surface and cross sections were investigated using scanning electron microscopy (SEM). For cross-sectional views, samples were embedded in epoxy resin and polished.

## 4. Results

### 4.1. Mass loss and mechanical properties

Fig. 1 illustrates the measured mass loss of the two materials at three aging temperatures. The mass loss increases with higher

aging temperatures. The overall mass loss and the mass loss rate of M18-1/G939 are about twice as high as those of 8552/IM7, especially for aging times longer than 200 h.

Tensile, compressive and ILS strength values obtained at different temperatures after different aging time intervals are compared as a function of both temperature and mass loss in Figs. 2–4. A decrease in strength with increasing mass loss is observed, but no significant influence of the temperatures applied. M18-1/G939 shows a slightly greater decrease in ILS strength (by 40%) compared to 8552/IM7 (by 25%, Fig. 2), whereas tensile strength decreases more for 8552/IM7 (by about 33% compared to by about 13% for M18-1/G939, Fig. 3). The change of compressive strength does not differ significantly between the two composites (Fig. 4). Typical results of the tensile test are shown in the stress–strain diagrams in Fig. 5. For both materials, the slopes of the curves do not change with increasing aging time at 200 °C. In contrast, the ultimate tensile stress and strain decrease with increasing aging time.

### 4.2. Fracture behaviour

In addition to mechanical strength decrease, the appearance of the specimens after failure also changed. Fig. 6 shows the representative specimens of 8552/IM7 aged at 200 °C compared to an unaged specimen. The photograph illustrates increased “brooming” with increasing thermal aging. This effect was not as obvious for M18-1/G939 since the fibre filaments were locked into position by the fabric structure. Fig. 7 shows a comparison of the cross sections of unaged and aged 8552/IM7 ILSS specimens after aging at 200 °C for 69 days. The entire cross section of the aged specimen exhibits micro cracking, and some of the cracks have developed into delaminations. Similar results were obtained for M18-1/G939. Fig. 8 illustrates differences of fracture surfaces of an unaged and aged M18-1/G939 tensile test specimen. The aged specimen was stored at 200 °C for 34 days. On fracture surfaces of unaged specimens fibres are covered with the matrix representing a good fibre to matrix adhesion. In aged specimens fibres are separated from the matrix, indicating a low fibre to matrix adhesion. The same effects were also observed for 8552/IM7.

### 4.3. Chemical analysis

An IR spectroscopic analysis of the matrix system 8552 shows intensive bands at 1610  $\text{cm}^{-1}$  and 1510  $\text{cm}^{-1}$ , which are attributed to the epoxy resin (Fig. 9). Bands at 1579  $\text{cm}^{-1}$  and 1486  $\text{cm}^{-1}$  are characteristic of the polyethersulfone. A detailed interpretation of the components' IR spectra is given in references [18,19,22]. For the evaluation of the epoxy resin the 1510  $\text{cm}^{-1}$  band was chosen, as well as the 1486  $\text{cm}^{-1}$  band for the polyethersulfone. The spectra of the matrix recorded on the specimen surface are compared for different durations of aging at 180 °C (Fig. 9). A decrease in

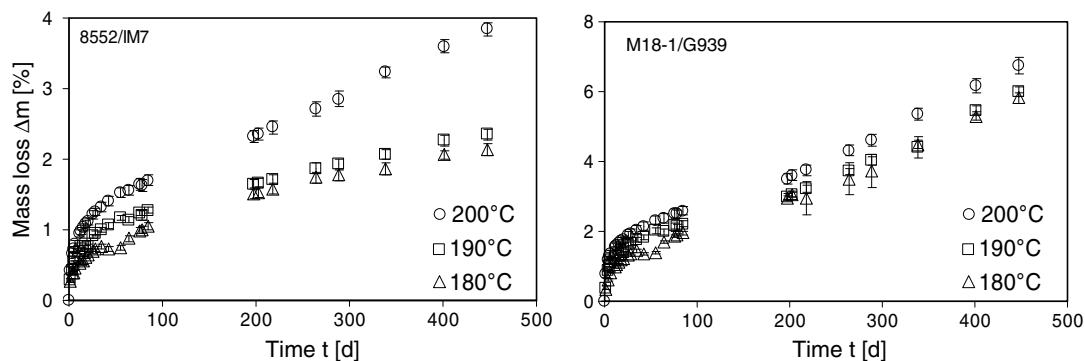


Fig. 1. Mass loss of 8552/IM7 (left) and M18-1/G939 (right) versus aging time at different aging temperatures.

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