

Damage of flexural loaded composite beams subjected to fire

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ABSTRACT: Fibre reinforced polymers are used extensively for automobile, marine and infrastructure applications. For these applications, fire events and the resulting effects on the structural integrity are of considerable concern. The strength of composite structures subjected to fire is strongly dependent on the exposure time and the load. In order to verify the mechanical response, the flexural behaviour of the material was investigated experimentally. The problems of predicting the thermal-mechanical behaviour of polymer composite materials may be divided into two different parts: the internal processes and the external boundary conditions. The internal processes include all physical and chemical processes which can occur in a laminate. The external boundary conditions implicate the shape, size and the ambient conditions of the tested specimen. Allowing for this separation, thermal properties of reinforced polymers have been studied for a polyester based composite and additional furnace tests have been carried out. A further step has been taken to reduce the gap in the understanding of composites investigating the combined effect of mechanical loading and thermal loading due to fire.

1 INTRODUCTION

Because of their favorable mechanical, physical and chemical properties as well as high strength to light weight ratios, polyester resin matrices are used in load-bearing structures. With an increasing use of GRP materials in load bearing structures which require long heating resistance there is an apparent need for further experimental, analytical and numerical studies.

Available design guidance does not deal with the resistance of polymer composites to the combined thermal and mechanical loading and therefore does not cover any risk of failure. Knowledge of the thermal and mechanical performance of these materials is essential for protection and evacuation systems in residential construction. The thermo-mechanical response of polymer composites is also important for the integrity, durability and long term performance under fire conditions. Thus, a predictability of service life under rapidly rising temperature is extremely important for FRP structural designers. Depending on the composition and heating rate chemical reactions begin to occur when the material reaches 100 to 300°C. These reactions are commonly referred to as oxidation/pyrolysis. At these temperatures, the resin constituent undergoes thermo-chemical decomposition and the material begins to break down.

The fire performance of polyester-based composite materials is generally assessed by the use of furnace tests (Davies & Currie 2003; Currie *et al.* 2003; Gibson 1998, 2005; Wang 1998).

In further literature are analyzed polymer matrix composite materials during material degradation at high temperatures. From 1996 to 1998 more research was carried out by (Gibson *et al.* 1998, Davies *et al.* 1998) and (Ludwig 2004) in the development of fire experiments and mathematical modelling. (Sorathia *et al.* 1992, 1994, 1997, Scudamore 1994) as well as (Mouritz 2002, 2005) observed that the organic matrix components are susceptible to combus-

tion and fire damage because of their chemical structures. Comparison of the post-fire mechanical properties of GRP composites with a polyester, vinyl ester or phenolic matrix has been investigated by (Mouritz 2002). Fire affects the mechanical properties of polymer composites as well as thermal properties. Particularly the Young's Modulus is the primarily affected factor defining structural behaviour at elevated temperatures. The macroscopic behaviour of materials can only be completely understood and optimized for a specific application when their properties on the microscopic field are well understood. Therefore this work has been divided in two experimental parts. The first part covers of a thermo-mechanical material description while the second part are large-scale fire tests to predict the fire resistance.

2 PART ONE – THERMO-MECHANICAL ANALYSIS

The discussed composite consists of an isophthalic polyester resin as a matrix and E-glass fiber as a roving for reinforcement. According to DIN ISO 1172 different layers of woven strand mats plus non wovens at the border of the composite can be detected. The resin is unmodified without additive- or reactive flame retardants. The fibre content was 69,5 % in weight (average value of eight TGA measurements). The initial density of polyester-based GRP is $1609,5 \text{ kg/m}^3$ (EN ISO 1183-1). The test specimens were water-jet cut from the flange of an I-beam so called IPE 120 girder. In order to reduce the chemical and physical influence of the coating, the specimens (thickness 6 mm) were machined further at both sides down to a thickness of 2,0 mm.

The thermal decomposition behaviour of the resin was determined by thermogravimetric analysis (TGA) using a Mettler Toledo 851° instrument. The TGA was performed in air atmosphere at different heating rates of 2 K/min, 10 K/min and 50 K/min in a temperature range from about room temperature 25 °C to 800 °C. The sample mass was modified from 27,54 mg to 40,12 mg and placed in the oven inside an aluoxide pan without lid.

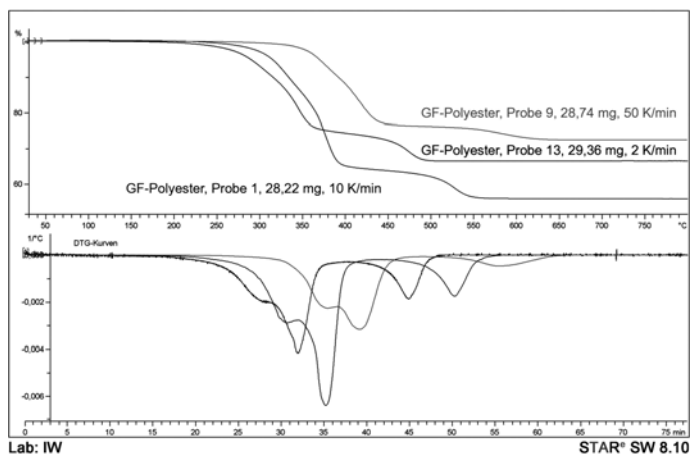


Figure 1. Typical TGA curve progressions with 2/5/10 K/min heating rates and similar sample weights

The complete oxidation could be described in three steps (Fig. 1). The density of resin began to decrease at a temperature range of approximately 220 °C. The average mass loss at this temperature reached up to 2%. The first step represented the loss of mass through vaporization of volatile components (moisture, styrene) by an assigned midpoint temperature of 320 °C with an approximate weight loss of 3 mg. The second step is the polymer decomposition with nearly 5,46 mg loss of mass and a midpoint temperature of 380 °C. In the last decomposition step the material lost 1,91 mg of weight. The assigned values are conforming to the 10 K/min TGA curve. As expected, the curves are shifted to higher temperatures with increasing heating rate (kinetic effect). The residue consisted mainly of glass fibers which fluctuate over a wide range despite of high sample identity by manufacturer.

The Differential Scanning Calorimetry (DSC) using a Mettler Toledo 822° instrument was performed in air atmosphere at a heating rate of 2 K/min in a temperature range from 25 °C to

350 °C. As a specific feature a variable aquarium pump was connected across an olive type tube fitting to the DSC in order to generate air atmosphere of 50 ml/min. The sample with 35,57 mg was placed in an aluminium pan with a pierced lid.

The polyester behaviour was different in the temperature range of 25 °C to 150 °C even though the measurement conditions were nearly identical (slight differences in the sample mass). In some cases the first heating run gives a curve where the region around the glass transition is distorted due to relaxation processes and moisture evaporation (non-reversible). The second heating run without the thermal history yields the shape of a glass transition. The evaluation should be performed on both heating runs. The combustion enthalpy is similar and started around 195 °C. Furthermore the oxidation process is finished at 580 °C. This is certainly dependent on the amount of resin in the sample disk. Due to the inhomogeneity of the material influenced by the presence of fibres, it was not always possible to define an accurate glass transition temperature T_g . From the nine DSC measurements a temperature range for the midpoint glass transition temperature (57,28°C +/- 15°C) and an average value for the specific heat (1470 Jkg⁻¹K⁻¹) were indicated. The evaluation is complex and requires more than one measurement.

The specific heat increases with temperature and moisture evaporation which is normally given by a linear function. The initial specific heat of GRP was 915.84 - 943 Jkg⁻¹K⁻¹. Between 55 °C and 75 °C, the specific heat experiences a slight increase and then drops back to the original value. This is equivalent to the glass transition temperature of 69.55°C of a normal DSC measurement. At 100 °C the specific heat rises by over 19.35% to 1114 – 1230 Jkg⁻¹K⁻¹ and reaches its maximum value of 2368 Jkg⁻¹K⁻¹ at 163 °C.

Dynamic mechanical analysis (DMA) experiments were performed on a TA Instrument 2980, using a three point bending clamp. As a result of higher heat conduction at both bearing points the dual cantilever clamp was no longer in use. The test specimens were water cut with a gauge length of 60 mm, 10 mm width and 2 mm thickness. The Young's modulus measurement was performed at 1 Hz from 0 °C to 330 °C at a heating rate of 3 K/min with a maximum amplitude of 10 µm and 150 % force track as well as an isothermal segment of 3 minutes at 0 °C. Force track is used when a static force needs to be changed in accordance with any change in the sample stillness, e.g., during a temperature ramp. The temperature behaviour of high polymer materials can be visualized by the mechanical damping in obedience of the temperature with forced flexural vibrations (Fig. 3). The thermal transformation is subdivided into thermal transitions and thermal relaxations. True thermal transitions are in equilibrium on both sides of the transition and may be of first, second or higher thermodynamic order. Thermal relaxations, on the other hand, are kinetic effects. They depend on the experimental method. The best known example is the glass temperature T_g (determined by DMA) at which a hard, glassy polymer converts into a visco-elastic material and vice versa, it is not a glass transition temperature.

The DMA measurement separates two relaxation phases. The β - relaxation can only be distinguished as a shoulder in the loss modulus at ~ 55 °C. The α - relaxation is appointed by the proxy expand peak. The glass temperature T_g is defined as an average value at 132 °C by the tan δ curve (Fig. 2). This temperature is approximately 10 °C higher if one would take the definition of the loss modulus at maximum 122 °C. The loss of strength starts at about ~ 50 °C and drops drastically after 75 °C, until the loss of serviceability at 150 °C. Consequently the storage modulus E' decreased from a peak value of 22 - 33 GPa to 8 – 15 GPa. The initial Young's modulus for polyester-based GRP, with a fiber volume fraction of 0.51 was taken as an average value of twelve measurements with 29.55 GPa. The Young's modulus, which is the key property in the equation of motion drops nearly linear by 60.0% at a temperature around 150 °C. The Young's modulus of the polyester-based composite indicated a flexural strength that is unserviceable for temperatures above approximately 150 °C – 160 °C. The onset temperature of 300 °C at the total loss of residual strength is equal to the main peak temperature of the first derivate of the TGA curve. The GRP shear modulus with an initial value of 5.8 GPa (equal measurement conditions), has a direct relationship with Young's modulus and the Poisson's ratio. It therefore decreases with temperature in the same order as Young's modulus does.

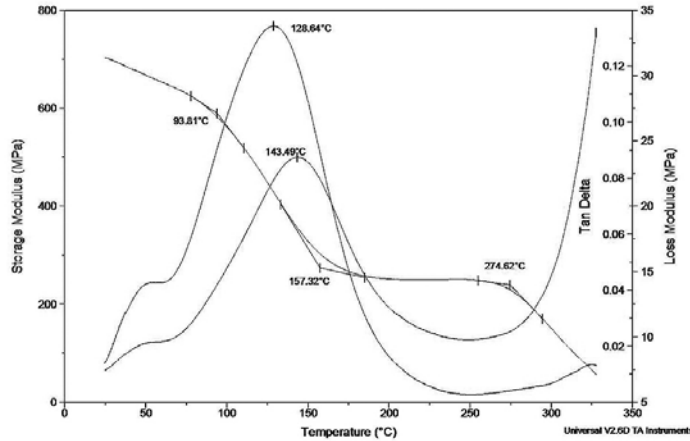


Figure 2. Characterization of GRP (storage modulus and damping coefficient) by DMA TA 2980

The IR Spectra of the evolved gases (3D diagrams with OMNIC software) during the weight loss for the thermal degradation of polyester resin are illustrated in (Fig. 3). The measurement was performed with heating from 20 °C to 550 °C at a heating rate of 10 K/min in air atmosphere. The sample mass of 58,08 mg was placed in an aluoxide 70 μ l pan without lid.

The results of the gas oxidation analysis show carbon monoxide, carbon dioxide, styrene, water, acetic acid and lesser products, the amount of each depending on the specific polymer sample. In the early stage the FTIR indicated the presence of the characteristic bands of water at 1300 cm^{-1} – 1700 cm^{-1} and 3500 cm^{-1} - 4000 cm^{-1} in function of time. The main thermal degradation occurs at temperatures above 270 °C. The presence of a band at 2380 cm^{-1} in the evolved gases which is due to the carbon dioxide indicate a degree of depolymerisation during the degradation. Also the strong presence of acetic acid (1798 cm^{-1} and 1776 cm^{-1}) and methyl styrene (2884 , 3027 , 3075 , 729 and 767 cm^{-1}) as well as the finger print region of styrene indicates that there is some pendant group or chain end group degradation. This is followed by an evolution of the IR – peaks at higher temperature with the bands of carbon monoxide (2120 cm^{-1} and 2175 cm^{-1}).

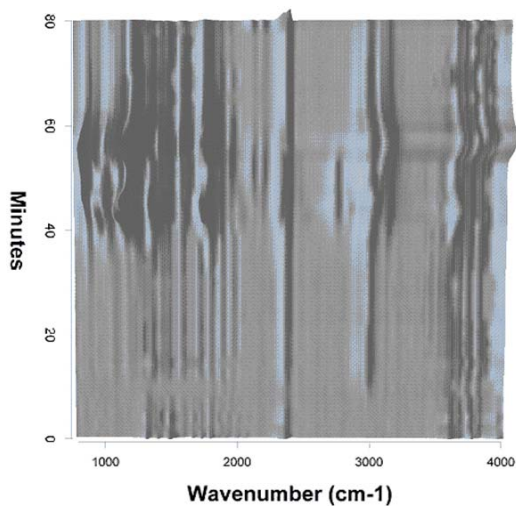


Figure 3. IR-Spectra of the coupled TG – FTIR of GR-polyester

3 PART TWO - FIRE TESTS

Thirteen experiments were conducted in the small furnaces at the EMPA – Dübendorf (Fig. 4). The aim was to determine the fire resistance. As done at the thermal analysis, the experiments were performed with three point bending. Due to the restricted spatial circumstances of the furnace the three point bending of the IPE profiles (1500 mm) was induced by upwards pulling. The compression flange was therefore on the lower side of the profile. In order to avoid an enforced translatory movement perpendicular to the axis of the profile, the tension arrangement was stiffened with a transverse connection to the frame of the test setup. Each sample was provided with twelve measuring points with thermo couples elements type K.

A brick partition made of 20 mm thick fiber cement panels isolated with 50 mm mineral wool was constructed as spatial limit and smoke gas closure. For observation and monitoring purposes two fire resistant glass panels were built in.

The force was applied with an “Amsler”-pendulum manometer. The deformations were measured with displacement transducers directly on the profile. The measuring points were situated at one third of the length of the heated part of the profile (1000 mm). Six furnace temperatures, from which the control of the ISO 834 can be deduced, were registered for the determination of the temperatures throughout the testing time.



Figure 4. Small furnace with test setup, application of measuring points at the different profile types and a 50 mm mineral wool protected IPE 120

The profiles were loaded at room temperature with 10 kN (nearly 30% of the collapse load of an IPE 120 girder). After the loading phase the profile was exposed to an all sided controlled heat treatment up to failure. Between the loading phase and the heat exposure phase a short stop was inserted in order to release residual stress. The profile dimensions were varied (IPE 120 / IPE 160). Further investigations should be done testing the amplification of fire resistance by using the same testing configuration but increasing the resistant moment. Additionally tests under self-weight, using 50 mm mineral wool and using a water based intumescence coating were carried out (Fig. 5). It should be mentioned that the coating was not especially developed for this application. Here the profile surfaces were sandblasted and the intumescence coating was applied per air brush.

The fire resistance of an IPE 120 flexural loaded beam was at approximately 1:44 minutes. The IPE 160 had a 40 second higher fire resistance at 2:24 min. In each fire test the failure occurs in the compression flange through local fiber buckling followed by buckling of the entire web. The test beam under self-weight had a fire resistance of 10:41 minutes. Failure was defined at the point of negative displacement. The self ignition temperature was determined at 3:41 minutes with an oven temperature of 502 °C and a surface temperature of 390 °C. Within 2 seconds the surface temperature rose up to 490 °C and the oven temperature to 522 °C. The experimental investigations showed that even with fire protection and dead load the fire resistance was insufficient (coating 16:11 min; mineral wool 12:06 min.). The coating reaction temperature of 200 °C is too high for GRP products subjected to fire. In general the reaction temperature of a coating should be lower than the onset temperature of strength loss.

4 CONCLUSION

Because of the high influence of a composite's inhomogeneity the experimental results for the thermal analyses have to be based on several measurements. Furthermore these thermal characteristics can only be seen as average values of a multi examined material. The thermal analysis should only be considered as a complementary measurement, for example for the determination of the specific heat as well as the storage modulus as a function of time and temperature. Relaxation phenomena in the material can be investigated by DMA. The glass temperature T_g is a specific temperature which varies according to kinetic phenomena. In conventional DSC the scan is usually made by heating from the glass equilibrium to the viscoelastic equilibrium which is a physical transition. The coupling of the TGA system with an FTIR spectrometer provided a simple technique for the identification of the evolved gases. This provided information of the polyester based composite characteristics such as thermal stability or oxidation as a function of temperature. The methods of the thermal analysis and the basic conditions of the large scale tests should be harmonized, particularly concerning the surrounding atmosphere. A connection was found between the reduced strength and an isotherm temperature of nearly 100 °C. This temperature corresponds to the loss onset temperature of the storage modulus. On the basis of scale tests with early failure a determination of the material characteristic at high temperatures ($> 200^\circ\text{C}$) is only partially useful (Fig. 1, 3).

The fire resistance tests allowed an investigating of the thermal and mechanical response of GRP pultruded beams with different cross sections in a fire situation. Furthermore the efficiency and a possible field of application of two passive fire protection systems could be evaluated. The unprotected GRP beams loaded with 10 kN failed after 2 to 3 minutes, the two different passive protection systems under dead load provided a fire resistance between 12-16 minutes. It may be possible to reach higher fire resistances with different resin compositions.

The latest research included thermal and coupled thermo mechanical numerical simulations. Furthermore master curves (DMA and TGA) with polyester based composites were generated and an extensive thermo mechanical investigation of vinylester- and phenol based GRP was carried out. The results will be presented in future publications.

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