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Polymer nanocomposites based on epoxy resin and ATH as a new flame retardant for CFRP: preparation and thermal characterisation

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Abstract Nanoscale aluminium trihydroxide (ATH) is examined to its suitability for carbon fibre reinforced plastics (CFRP) as a novel flame retardant. In particular the influence of particle size and concentration of ATH to the fire behaviour of epoxy polymer matrices is determined. The particle size is adjusted by means of different dispersing techniques. By SEM images and viscosity measurements the homogeneity of the produced ATH dispersions for the liquid ones and of cured epoxy–ATH nanocomposites is determined. Based on these pre-results, selected ATH dispersions are used for the manufacturing of CFRP which are produced by the proved injection technology. The thermal stability of the ATH nanocomposite plates and the corresponding CFRP plates are analyzed by means of quantitative single difference thermoanalysis (QSDTA). The fire behaviour is characterised by using the OSU chamber test. Both tests indicate a reduced heat release rate by decreasing the ATH particle size, i.e. the thermal load decreases. A combined fire protection mechanism is discussed for the improved fire protection through ATH nanoparticles.

Introduction

Fibre composite materials are used in many areas today. These include for example aviation and automobile, ship and rail vehicle construction. The broad field of application for this class of material is mainly attributable to its outstanding characteristics with regard to strength and rigidity and its suitability for lightweight construction. Important issues that have to be resolved in the near future include a reduction in the required maintenance as well as fatigue behaviour and flame resistance of the material. The utilisation of fibre composites in public transport will make the fire protection of these materials particularly important. Fire safety requirements and hence the requirements that modern flame retardants have to meet in this area, are especially high. Important aspects of the use of flame retardants for fibre composites include not only effectiveness in the event of fire or compatibility with the resin system, but also toxicity, resistance to ageing, emission behaviour and recyclability.

Commercial flame retardants are classified into four categories according to their chemical composition: organohalogen, organophosphorus, organonitrogen and inorganic retardants [1, 2]. These differ in their principal modes of action.

Organohalogen flame retardants act in the gas phase by interrupting the radical chain mechanism. In the event of fire, these flame retardants release halogen radicals, which capture hydrogen and hydroxide radicals liberated by pyrolysis. An increase in the smoke density and the release of toxic dioxins are however problematic. Halogenated flame retardants are therefore considered very risky with regard to the environment and health and their use is declining significantly.

Flame retardants on the basis of organonitrogen compounds work in different ways, such as by cooling and dilution of the pyrolysis gases. The compounds dissociate with withdrawal of heat (endothermic), and therefore remove energy from the system. The foaming up of the matrix caused by additional release of decomposition gases such as nitrogen or ammonia, leads to a heat insulation effect between the fire and the flammable material.

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Organophosphorus flame retardants initiate the development of a protective layer of charred material, which acts as a barrier for oxygen and heat (intumescence).

Inorganic flame retardants mainly include inorganic metal hydroxides, such as for example aluminium trihydroxide (ATH) and magnesium dihydroxide (MDH), in addition to "red phosphorus". Metal hydroxides decompose at a specific temperature with elimination of water (decomposition temperature of ATH: 200 °C; MDH: 300 °C [1–3]). This endothermic process removes energy from the system, while the evaporating water serves to dilute the flammable pyrolysis gases at the same time. Large quantities have to be added for effective flame retardance.

Currently commercial flame retardants in polymeric materials are predominantly used in the form of microscale fillers [1]. The use of nanoscale flame retardants (1–100 nm) represents a new and innovative approach. It is to be expected that nanoscale flame retardants are more efficient than conventional microscale flame retardants. The relatively large surface area provided by nanoscale flame retardants should lead to a significant improvement in the fire protection of the polymer matrix even at small concentrations, allowing a reduction in the required filler content (weight saving, cost saving).

The most interesting nanoscale fillers [2], which international research activities currently focus on as flame retardants for polymer matrices, include:

- Carbon nano tubes (CNT) [4–8],
- Layered silicates (Organoclays) and Boehmites [8–13],
- Aluminium trihydroxide (ATH) [14] and
- Magnesium dihydroxide (MDH) [15–17].

The mechanisms underlying the flame protection effect of these nanoparticle classes vary significantly and are not yet fully understood. The effects involved are, however, presumably overlapping rather than individual effects. The central flame protection mechanisms discussed include, e.g. heat reduction (CNT), the influence of gas barrier layers (longer diffusion paths of the pyrolysis gases in the case of layered silicates), vitrification (silicates), as well as the release of water (cooling and diluting effect such as for ATH and MDH).

This study involves an analysis of the suitability of ATH as a nanoscale flame retardant for CFRP (carbon fibre reinforced plastic). Quantitatively, ATH is the most significant and economical mineral fire protection agent for polymer materials. In contrast to the still used organohalogen flame retardants, ATH is environmentally friendly and non-toxic. Particular focus is on investigation of the influence of ATH particle size and concentration on the fire behaviour of epoxy resin matrices. Therefore, selected ATH dispersions are used for the manufacture of CFRP which are produced using the well established injection technology. The nanoscale dimension of ATH is an important prerequisite in this context, in order to retain the injectability of the epoxy resins and to prevent filtering out effects. The flow characteristics of the ATH-modified epoxy resins are determined by preliminary rheological studies. Thermal characterisation of both the nanocomposite plates and the analogous CFRP plates is carried out using quantitative single difference thermoanalysis (QSDTA) and an OSU chamber test, in order to understand how nanoscale ATH works as a flame retardant. A better understanding of the mode of action of nanoscale ATH might reveal new promising routes for the development of even more successful flame retardant systems in the near future.

Experimental part

Materials

The polymer matrix used in this study is an epoxy resin from Huntsman and based on diglycidyl ether of bisphenol A (DGEBA) (Araldite LY556), cured by an anhydride curing agent, 4-methyl-1,2-cyclohexanedicarboxylic anhydride (Aradur HY917) and accelerated by an amine, 1-methyl-imidazole (DY070). The nanoparticle system is a commercially available aluminium trihydroxide (ATH) with spherical agglomerates (average particle size of dry powder: 9.3 μm) provided by Sasol (Germany). The particles are not surface modified. A biaxial carbon fibre fabric (style 475-5T; satin weave; without binder; mass per unit area: 298 g/m^2) from ECC (Germany) was used for production of the fibre composite test plates.

Dispersion technology

For the identification of the most efficient dispersion technology for the homogenisation of ATH particles different devices were used like a homogenizer (Ultra Turrax, IKA, Germany), a dissolver (DISPERMAT AE03-C1, VMA-Getzmann, Germany) and a basket mill (Torus Mill; TML 1 DISPERMAT SL-012 C1, VMA-Getzmann, Germany). In each case an ATH master batch with 38 wt% ATH particles in epoxy resin was produced. The required particle mass was dried for 30 min in an oven at 110 °C beforehand and then stirred into the epoxy resin (Araldite LY556) at 50 °C. These preliminary dispersions were then processed further using the homogenizer (24.000 rpm for 20 min at 70 °C), the dissolver (3000 rpm for 20 min at 70 °C) or the basket mill (1200 rpm for 1 h at 70 °C).

The dispersed master batches were then degassed at maximum 5 mbar. The finished master batches are stable in storage at −20 °C. Final Dispersions with concentrations of 5, 10, and 20 wt% ATH in the resin system were produced from the master batches. Therefore, the master batches of ATH (38 wt%) were first diluted with the neat epoxy resin and then well mixed with the hardener and accelerator. In each case the mix ratio of the epoxy resin (Araldite LY556), the hardener (Aradur HY917) and the accelerator (DY070) was 100:90:0.5 wt%.

Preparation of the test plates

The epoxy–ATH nanocomposite plates were produced using a casting mould composed of three parts (geometry of the plates: height: 200 mm; width: 300 mm; thickness: 2 mm). The casting mould was pre-warmed in an oven at 80 °C and filled with the resin mixture. The modified resins were pre-cured at 80 °C for 4 h and post-cured at 120 °C for 4 h (ramp-up and ramp-down rate: 1 °C/min). After complete curing, the nanocomposite plates were demoulded and cut to size for the individual fire tests.

The CFRP plates were manufactured using the differential pressure resin transfer moulding (DP-RTM) method [18]. This injection technique allows variable adjustment of the fibre volume content by means of the autoclave pressure. A fibre volume content of 60% was selected for all the plates. The autoclave was preheated to 80 °C for production of the CFRP plates. The ATH nanoparticle modified epoxy resin was injected using a cartridge, brought to a steady temperature of 70 °C before use. The curing cycle of the CFRP plates in the autoclave corresponded to the temperature cycle of the epoxy–ATH nanocomposite plates previously manufactured in the oven. CFRP test plates with particle concentrations of 10 and 20 wt% were produced with the ATH particle system, as well as a reference plate without any nanoparticles.

The injectability of the nanoparticle-modified resin was checked before production of the CFRP plates. Important on the one hand, was an initial viscosity not higher than 500 mPas. Resins with a higher viscosity are considered as no longer injectable. On the other hand, the particle size distribution had to be determined, since microscale particles lead to filtering out effects by the semi-finished fibre product. The absence of microparticles was verified by exemplary analysis using SEM images.

Characterisation methods

The homogeneity of the particle distribution and particle size were analysed with a high resolution scanning electron microscope (SEM type: LEO 1550; resolution: 1.2 nm; magnification: max. 900×10^3; Zeiss; Germany) using cured ATH nanocomposite samples. A thin layer of gold was applied to the surface of the specimens for providing electrical conductivity. The images were recorded with an accelerating potential of 5 kV and a magnification between 10×10^3 and 50×10^3.

The viscosity of the modified and unmodified resin was determined by means of rheological measurements conducted with a rotational viscometer (Malvern Instruments—Gemini; Germany). The measurements were made using the parallel plate method. The plate diameter was 40 mm and the distance between the plates was 1 mm. The viscosities were determined as a function of temperature. The temperature was continuously increased from 30 to 140 °C. In the course of this, the shear rate increased from 0.7 to 13 Hz, at a constant shear stress of 1 Pa.

The thermal analysis was performed using a TGA/SDTA 851 (Mettler Toledo, Germany). That device allows the measurement of the weight lost and the detection of enthalpies simultaneously by the so called quantitative single difference thermoanalysis (QSDTA). On that way information about mechanisms and mode of action with respect to the used flame retardant might be easily provided. The measurements were conducted using oxygen as a purge gas with a gas flow rate of 195 mL per minute. The temperature programme was set to run from 40 to 700 °C and the heating rate was 2 K per minute. Samples with an initial weight of 50 mg were used.

Flammability test

The fire behaviour of the ATH modified CFRP plates was investigated using an Ohio State University (OSU) test chamber. That kind of test chamber is well established in the aircraft industry for the measurement of heat release rates (HRR) of aircraft parts and was developed in accordance with the FAA Fire Test Handbook (Ch. 5). The measurements performed here based on the specifications mentioned in the FAA regulations FAR 25.853 (a-1) as well as in the ASTM E 906 [19]. Accordingly the heat release rate (HRR) under defined radiation exposure conditions was used as an assessment criterion. The first step involved heating up the chamber for about 90 min until a constant temperature was achieved. Calibration is performed after adjustment of the radiant heating system. The sample was placed vertically in a sample holder in the pre-chamber. After 1 min, the sample holder was brought to the test position and the measurement was started. The samples were wrapped in aluminium foil and fitted in a test frame before placement in the test chamber. Any aluminium foil residues were then carefully removed from the front surface facing the radiator. The samples were 150 × 150 mm in size. They were tested for a period of 300 s after preconditioning.

Results and discussion

Particle size distribution

It is well known that the degree of dispersion of nanoparticles in a polymer matrix is a governing parameter which controls the final properties of the resulting nanocomposites. Only an extremely homogeneous particle distribution with the development of primary nanoparticles efficiently reinforces the polymer matrix. Therefore, the dispersion technique controls the particle size distribution. Three different dispersion techniques (homogenizer, dissolver and basket mill) were investigated to find the most efficient method for the preparation of nanoparticle-modified resins.

Figure 1 illustrates the homogeneities of the dispersions achieved using SEM images. The particle content is ~20 wt% ATH in each case. Mass concentrations of over 20% are generally found to be critical during production. A basic requirement for the separation of the ATH agglomerates is the effective introduction of shear forces in the particle containing resin. Utilisation of the homogenizer leads to only slightly separated aggregates in the micrometer range. It can therefore be said that the homogenizer introduces only small shear forces in the dispersion. The dissolver separates the agglomerates well, so that only few agglomerates in the submicrometer range and many primary particles in the nanometer range are obtained. The best dispersion results are achieved with the basket mill. The particles produced with this equipment are predominantly in the nanometer range. This method was used for the production of all further dispersions.

Furthermore, the effect of particle content on the dispersion quality was analyzed. Figure 2 shows SEM images of nanocomposite samples made with different ATH concentrations (magnification in each case: 50×10^3). The samples were dispersed by means of the favoured basket mill and then cured.

The corresponding SEM images prove the homogeneous distribution of the spherical ATH particles in the cured nanocomposite as well as the absence of any major agglomeration of particles. The images suggest a narrow and monomodal particle size distribution with an average size below 200 nm. Obviously the variation of the particle content does not show any effect on the particle size distribution. It is evident that a good dispersion quality is retained in the nanocomposite from the liquid to the fully cured state and that primary particles are obtained.

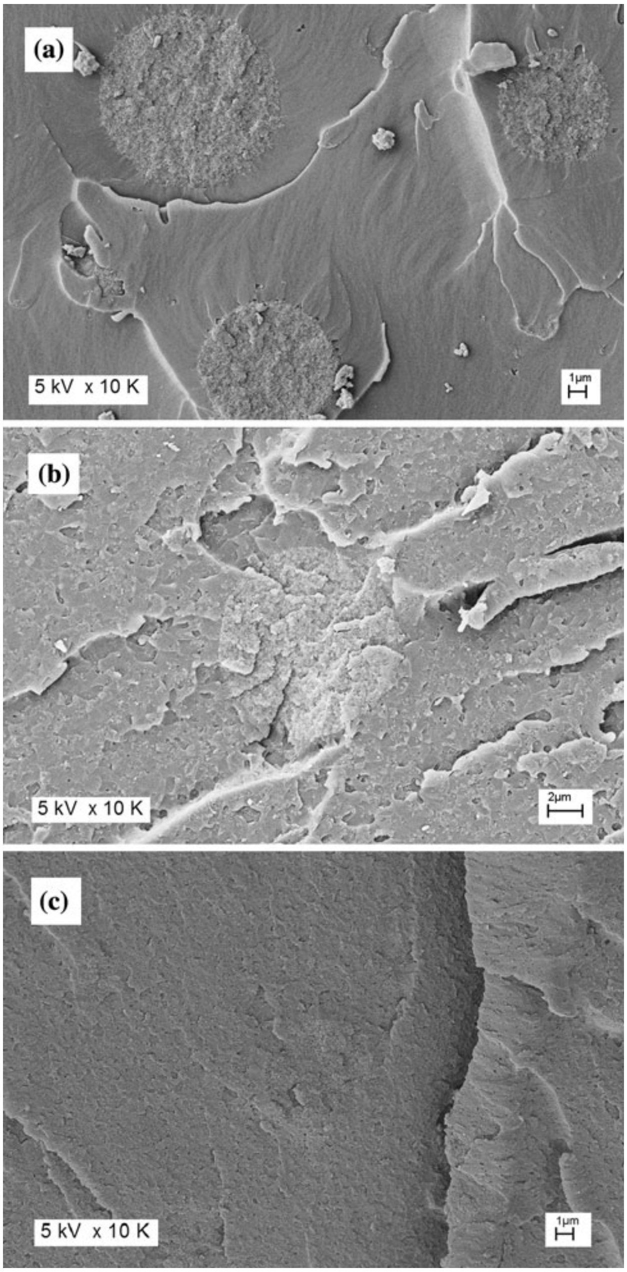


Fig. 1 SEM images (cross section) of epoxy–ATH nanocomposites with 20 wt% ATH each, which were dispersed using different methods: **a** homogenizer (Ultra Turrax; 24.000 rpm for 20 min at 70 °C); **b** dissolver (3000 rpm for 20 min at 70 °C); **c** basket mill (1200 rpm for 1 h at 70 °C)

Rheology

The viscosity of the resin and the particle size distribution are key factors for CFRP structures manufactured by the differential pressure resin transfer moulding (DP-RTM) technique [18]. Both parameters are corresponding values and determine the injectability of the dry fabric by the resin. Therefore, the flow behaviour of the nanocomposites was investigated in a series of rheological experiments, increasing the filler content up to 20 wt% ATH. The dispersions were prepared by the basket mill method and show comparable particle size distributions (<200 nm). The results are depicted in Fig. 3.

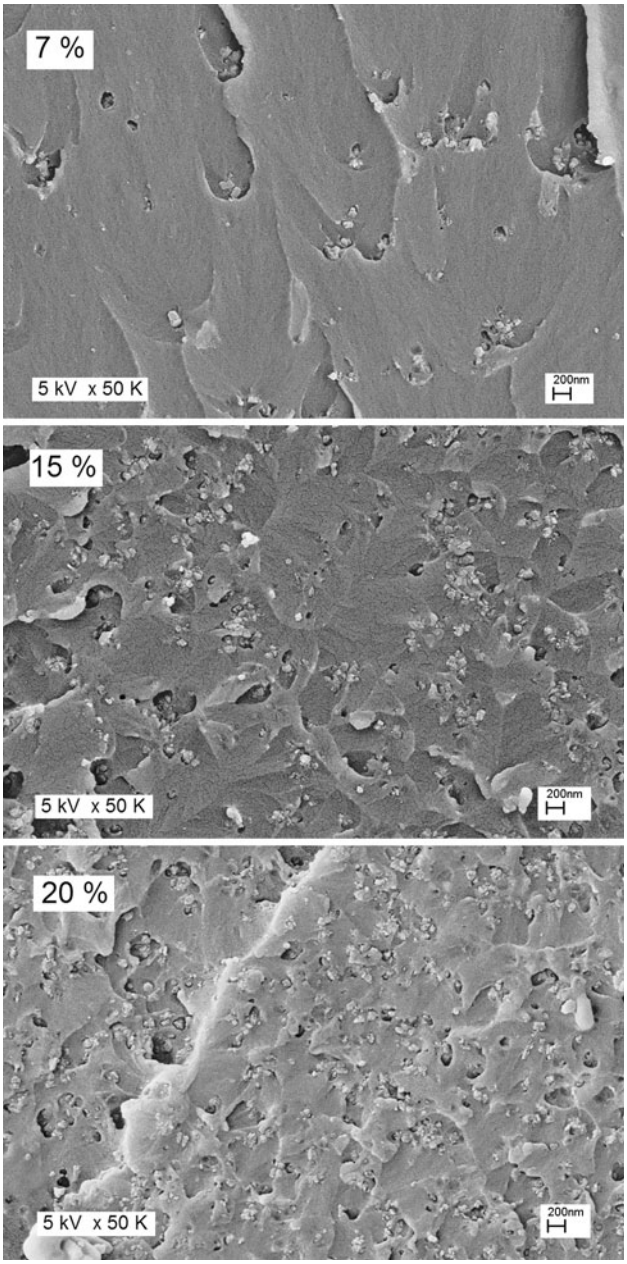


Fig. 2 SEM images (cross section) of epoxy–ATH nanocomposites with different ATH content. Dispersion technique: basket mill (1200 rpm for 1 h at 70 °C)

Obviously, the rheological behaviour of the resins modified with ATH nanoparticles is not essentially affected by the particle concentration. Thus, in a concentration range of up to 10 wt% ATH the viscosity does not increase considerably compared to reference resin. However, at an ATH concentration of 20 wt%, the viscosity at 93 °C is more than doubled compared to the reference resin. Nevertheless, the viscosity limit value for the injection process at 500 mPa*s is not exceeded for the interesting temperature range of 70 to 100 °C investigated here. With regard to the filler content studied herein, the increase of the initial viscosity is acceptable and is still in the range of processability for the injection technique (LCM). The injectability is retained for the modified resins.

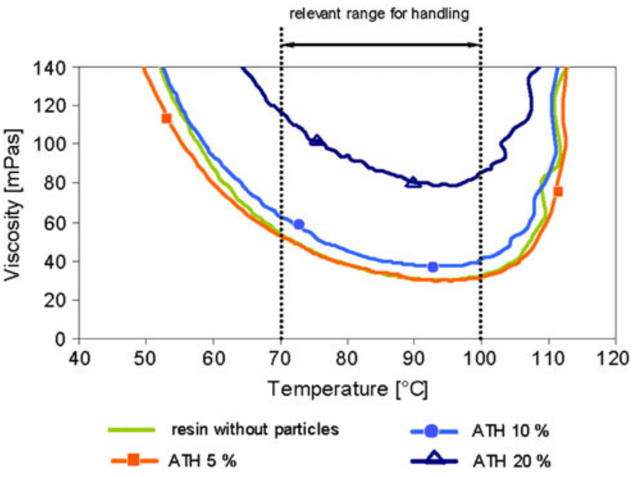


Fig. 3 Dynamic viscosity curves of epoxy–ATH nanocomposites as function of temperature and varying ATH content (0–20 wt%). Conditions: constant shear stress of 1 Pa

Thermal stability

Influence of ATH particle size in the resin

The influence of the particle size on the thermal decomposition behaviour of the polymer matrix was studied. The dispersion quality and therefore the size of the particles present in the resin are expected to have a significant influence on the thermal decomposition behaviour of the resin-particle systems [14]. Cured epoxy–ATH samples differing in ATH particle size were investigated by means of quantitative single difference thermoanalysis (QSDTA) for this purpose.

Samples with a particle size distribution predominantly in the micrometer range are obtained by stirring in commercial ATH powder manually. These samples display the same average particle size as the particle powder itself (average diameter by manufacturer specifications: 9.3 μm), since dispersions made by hand do not result in separation of the agglomerates. The samples with ATH nanoparticles were produced using a basket mill. An average particle size of ~50 to 200 nm is obtained using this method (see SEM images in Fig. 2). The influence of particle size on the thermal decomposition behaviour of epoxy–ATH resin samples is illustrated by the QSDTA curves shown in Figs. 4, 5.

The curve shape of the QSDTA measurements shows a distinct difference in the decomposition behaviour of the different dispersion qualities. In combination with the heat

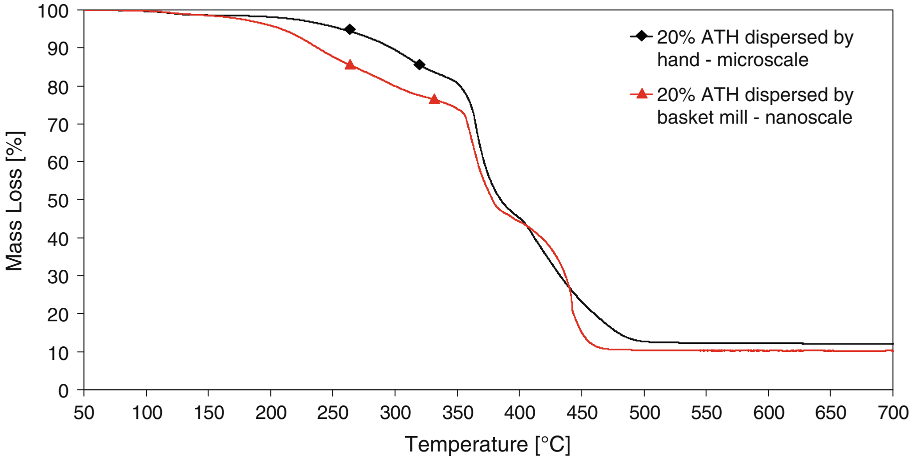


Fig. 4 Mass loss versus temperature for different particle sizes of ATH in a cured epoxy resin. Data obtained from the QSDTA

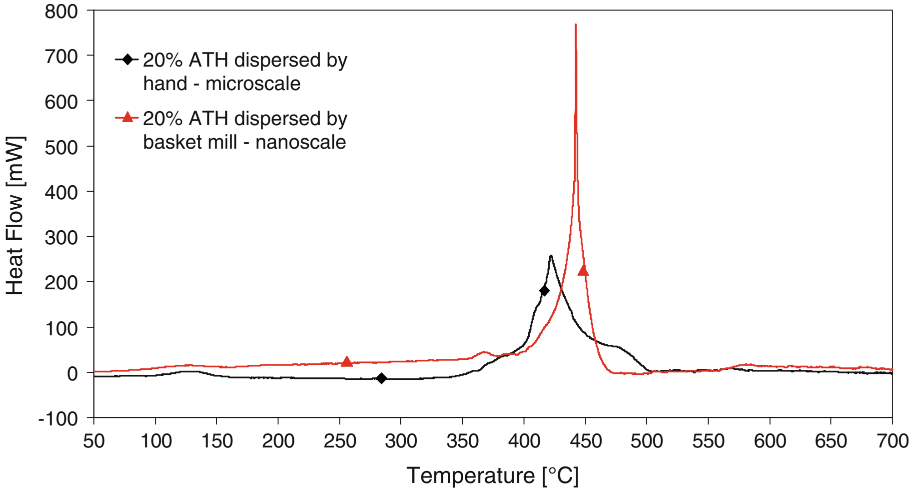


Fig. 5 Heat flow versus temperature for different particle sizes of ATH in a cured epoxy resin. Data obtained from the QSDTA

flow and peak temperature values determined (Table 1), it can be said that the particle size has a significant influence on the thermal behaviour of the modified resin system. As opposed to particles in the micrometer range, a particle size in the nanometer range results in a reduction in the average enthalpy by 11% compared to a pure resin sample (reference) and a shift of the heat peak to higher temperatures. A lower enthalpy is equivalent to a reduced fire load. The shift of the heat peak to higher temperatures indicates an increase in the thermal stability. This phenomenon can be explained by the surface effect: the larger the surface, the higher the rate of release of water. This makes the flame retardant properties more effective, which has a significant influence on the fire behaviour.

Influence of ATH particle concentration in the resin

The influence of the concentration of nanoscale ATH on the thermal stability of the epoxy resin matrix was also investigated. Cured epoxy–ATH samples differing in ATH nanoparticle concentration were investigated by means of QSDTA for this purpose. Figures 6 and 7 depict the QSDTA curves of the epoxy–ATH nanocomposites compared to a reference sample without any nanoparticles.

The decomposition of the reference sample clearly occurs in three stages, while the ATH nanocomposites decompose in four stages. A first, very indistinct stage occurs in all of the samples, starting at a temperature of about 100 °C. This is caused by the evaporation of moisture absorbed by the

Table 1 Influence of ATH particle size on the thermal decomposition behaviour of the epoxy resin matrix measured with QSDTA

Sample	ATH content (wt%)	Average enthalpy (J/g)	Average peak temperature (°C)
Pure resin (reference)	0	7298	435
ATH dispersed by hand: microscale dispersion	20	7615	415
ATH dispersed by basket mill: nanoscale dispersion	20	6490	456

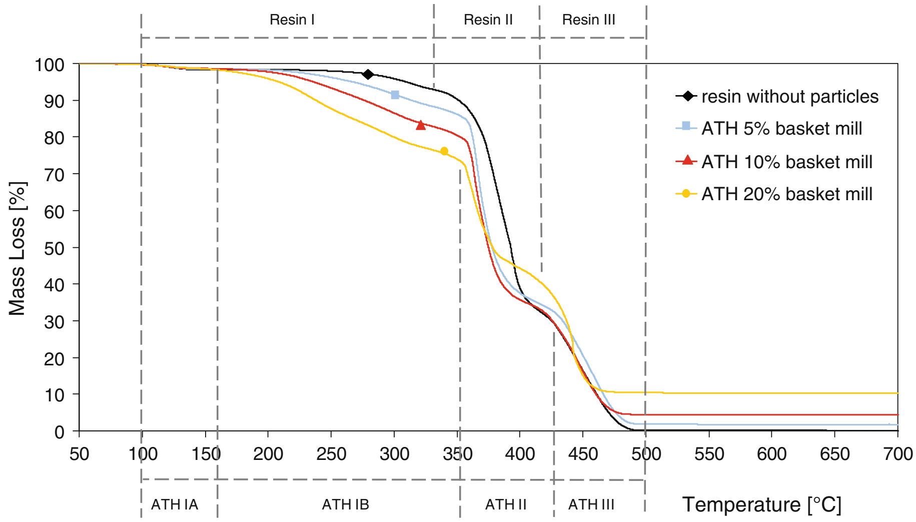


Fig. 6 Mass loss versus temperature for different concentrations of nanoscale ATH in a cured epoxy resin; data obtained from the QSDTA. Resin I–III: reference resin; ATH I–III: ATH nanocomposite

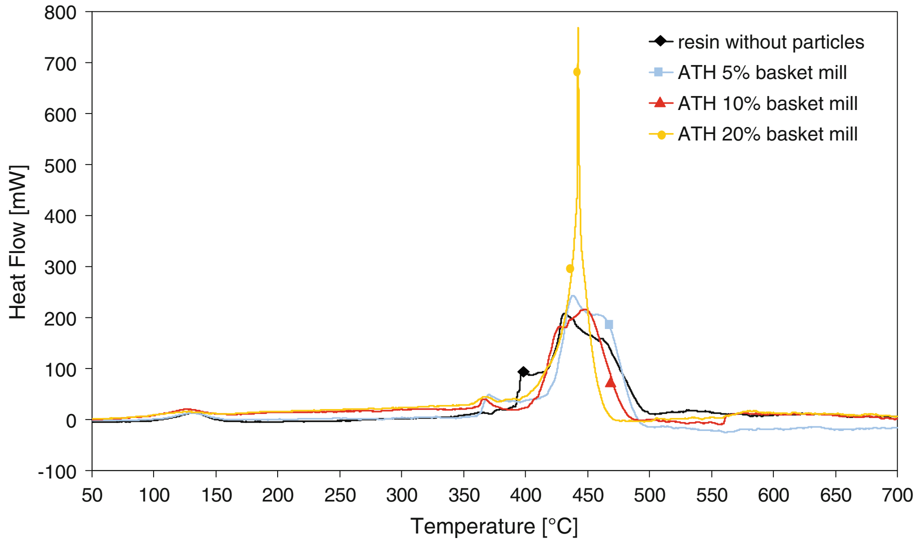


Fig. 7 Heat flow versus temperature for different concentrations of nanoscale ATH in a cured epoxy resin. Data obtained from the QSDTA

samples from the surroundings. In contrast to the reference resin, a further decomposition stage is observed in the ATH nanocomposite samples from a temperature of ~160 °C. This results from the release of bound water from the ATH nanoparticles (decomposition temperature of ATH: ~200 °C [1–3]). It is clearly evident that as the particle content increases, the decrease in mass occurs earlier and is simultaneously more pronounced. Above 330 °C, a two-stage decomposition process of the epoxy resin is observed in all of the samples. This effect is caused by the degradation of the individual components present in the resin. In the reference sample, the first stage occurs between ~330 and 420 °C, while in the ATH nanocomposites this occurs between ~350 and 430 °C. This involves a ~60% reduction in the mass of the reference resin. Only minor exothermic reactions as well as a first peak can, however, be recognised in this area in all of the heat flow curves. This first peak is shifted from approximately 395 °C (reference resin) to about 365 °C by the addition of ATH nanoparticles. The higher the particle content of the sample the flatter the curve in this section. This is directly followed by complete decomposition of the resin. Most exothermic energy is released in this region, which is indicated by a pronounced peak. Enthalpies were determined for all the measured samples by evaluation of integrals in the relevant sections of the curves; these were used to obtain an average value. Although the enthalpy of the 5 wt% samples is slightly higher compared to the reference resin, the enthalpy of the ATH modified resins decreases with increasing particle concentration on the whole. Compared to the reference samples, the enthalpy values of the samples with 20% ATH decrease by about 11% (from 7298 to 6490 J/g).

Influence of ATH particle concentration in the composite

The thermal behaviour of ATH modified fibre composite samples is illustrated in Figs. 8, 9. The curves of the ATH modified samples are approximately the same. The curve of the unmodified CFRP reference sample on the other hand, differs significantly with regard to mass loss and heat flow. The mass loss diagram shows clearly that the unmodified CFRP reference sample loses about 30% of its mass during the first decomposition stage, while the ATH-modified CFRP samples only lose ~15%. This behaviour is also reflected by the heat flow diagram. The exothermic decomposition reaction of the resin begins at a temperature of ~430 °C for the ATH modified CFRP samples, while this temperature is ~440 °C for the unmodified CFRP sample. The associated released heat flow is, however, significantly smaller. It can generally be said that the addition of 20% ATH particles causes a reduction in the quantity of heat released by the composite by ~38% (from 2250 to 1400 J/g). At a temperature of ~520 °C, the unmodified CFRP sample is subject to an apparent increase in mass. This behaviour is caused by the commencing decomposition of the fibre material, the resulting ignition of which gives rise to a recoil effect registered as an increase in mass.

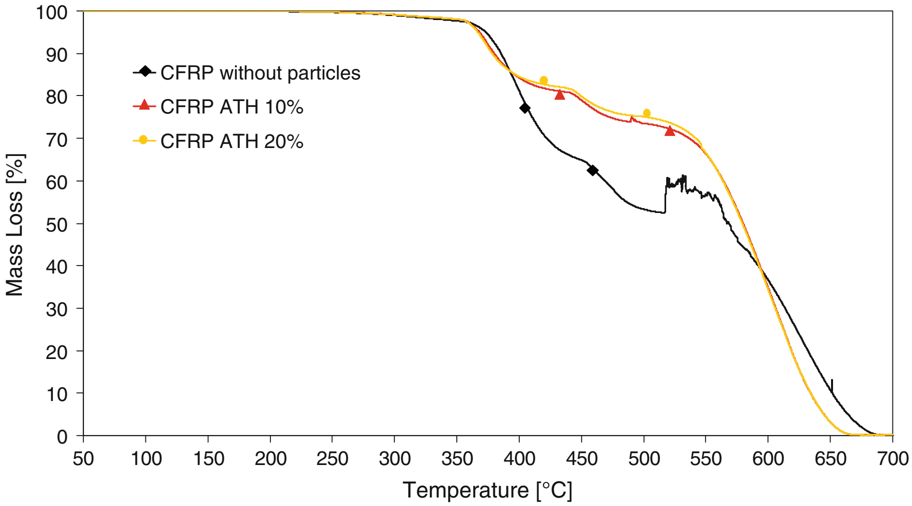
Flammability test

The fire behaviour of the ATH modified CFRP samples were studied in an OSU test chamber. The heat release rate (HRR) was used as a test criterion. An improvement in the fire protection properties can generally be expected if the HRR curves are flat, the peak values occur late and the integrals of the quantity of heat become smaller in total. These parameters appear to be fulfilled by the ATH nanoparticles, as demonstrated by the HRR curves in Fig. 10. The heat flow is slightly negative at the beginning of the test, since energy is consumed by the pyrolysis of the material. The pyrolysis gases ignite after about 30 s causing the heat flow to increase rapidly. After reaching a maximum value, the released quantity of heat drops again. A comparison of the measurements shows several differences in the fire behaviour. The increase during the ignition of the samples is steeper for the modified CFRP sample (10 wt% ATH relative to the resin) than for the unmodified sample. Furthermore, the HRR decreases immediately after reaching the maximum value in the modified sample. In the unmodified sample on the other hand, the maximum value of the HRR is reached and then remains approximately the same for a period of 95 s. Moreover, the curve of the flame retarded sample is flatter. This can be accounted for by the endothermic water release from the ATH nanoparticles, causing a removal of energy from the system. The use of ATH nanoparticles made it possible to reduce the quantity of heat released (heat integral) by about 12% (from 138 to 121 kW/m^2) after 2 min and by about 58% (from 474 to 199 kW/m^2) after 5 min, compared to the reference sample. The use of ATH nanoparticles clearly leads to an unexpectedly large decrease in the heat release rate. A combined fire protection mechanism is presumed, i.e. a tight barrier layer (vitrified layer) [20] which prevents the fire from progressing is simultaneously formed in addition to the endothermic elimination of water. Both fire protection effects should be favoured by the nanoscale size of the ATH particles due to the higher specific surface area and are not observed in the case of the reference samples.

Summary and conclusion

In this work, the fire behaviour of nanoscale ATH particles in epoxy resins and the corresponding CFRP laminates has been investigated. The thermal decomposition behaviour has been analysed with respect to the particle size, i.e. by using micro and nanoparticles, as well as the variation of particle concentration. The use of nanoparticles instead of microparticles leads to a decrease of enthalpy and a shift

Fig. 8 Mass loss versus temperature for different concentrations of nanoscale ATH in CFRP. Data obtained from the QSDTA



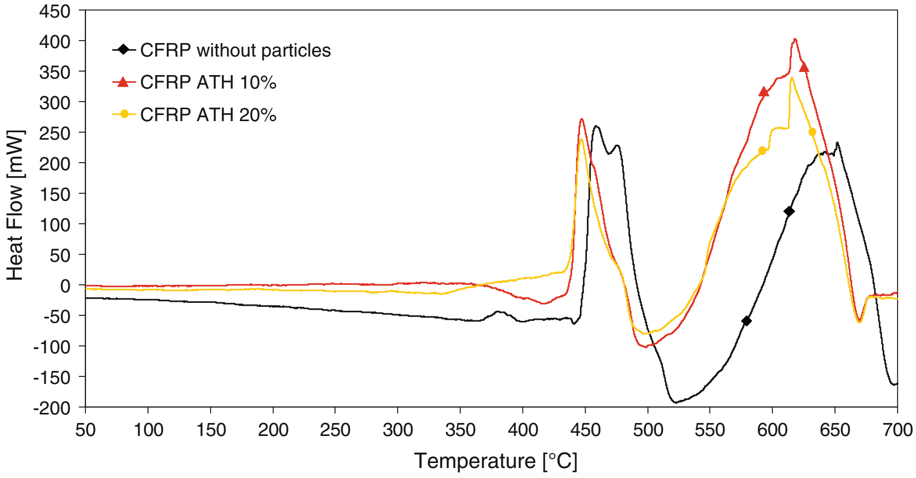


Fig. 9 Heat flow versus temperature for different concentrations of nanoscale ATH in CFRP. Data obtained from the QSDTA

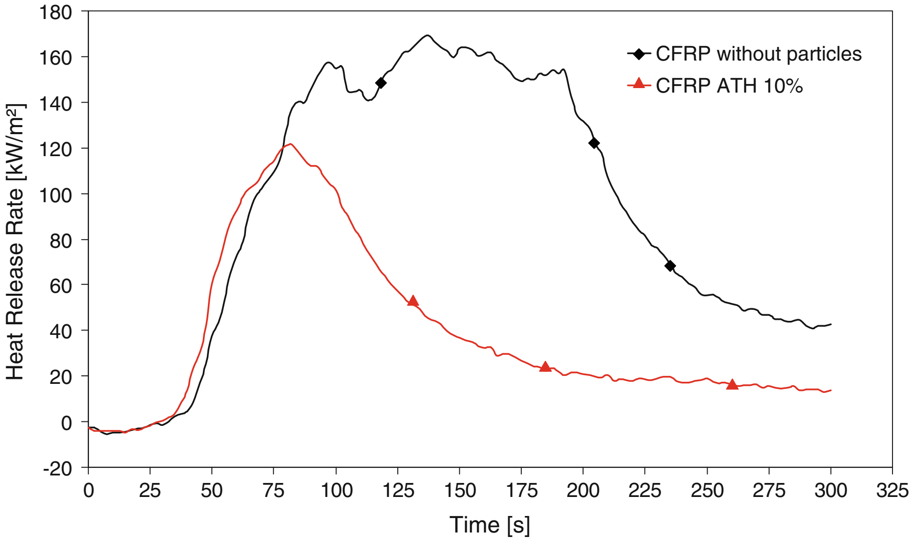


Fig. 10 HRR curves for the modified CFRP (10 wt% ATH nanoparticles) and the unmodified reference system obtained from the OSU calorimeter

of the heat flow peak to higher temperatures as seen by QSDTA curves suggesting a higher thermal stabilization of the polymer matrix by nanoparticles. Furthermore the average enthalpy decreases with the amount of particles in the resin. With 20 wt% of ATH nanoparticles, the enthalpy is reduced by about 11% in comparison to the pure resin. The same effects are observed for the CFRP laminates, i.e. a nanoparticle concentration of 20 wt% ATH in the resin reduces the average enthalpy by about 38%. Flammability tests with an OSU calorimeter show that a CFRP with 10 wt% ATH nanoparticles in the resin induce a significantly lower heat integral in comparison to a CFRP without a flame retardant. Obviously, the flame retardant properties of ATH nanoparticles can be efficiently transferred from resin to the CFRP laminates and the nanoscale ATH seems to be an even more successful flame retardant than the corresponding microparticles. In addition, nanoscale ATH particles as a new environmental-friendly flame retardant might also improve the fundamentally mechanical values of the CFRP as well as the structural integrity of the part in case of fire (lower deformation during fire) which have to be investigated in forthcoming papers.

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