



Budapest University of Technology and Economy
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Development and analysis of hybrid composites

Thesis booklet

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Budapest, 2018

The referees' opinion and the minutes of the PhD examination can be inspected at the
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1. INTRODUCTION

Polymers are widely used in daily and technical life, and their market has started to grow again after the economic crisis that began in the last decade. Beside the unreinforced polymers, the composites are also gaining popularity thanks to the relatively high strength compared to their low density. The growing market not only affects the thermoset composites, but also the thermoplastic composites, since parts from these materials can easily be produced in large quantities with relatively low energy investment with the technology of injection molding.

Due to the variety of materials available to the engineers, there are many opportunities to choose the right material or to create materials with special functions (heat and electric conductivity, gas closure ...) which increase also the number of possibilities. These materials need to stand up against the new requirements and it is also important to create high quality technical materials that not only allow cost savings, but bear relative high temperatures and long lasting stresses. These requirements can be fulfilled by polyamides and their composites. Thanks to their good mechanical properties, the family of polyamides can be found in many areas of life.

In the case of composites produced by using traditional reinforcements, the research was intensive in the past and still it is. The discovery of nanoscale reinforcements has given new dynamism to the development that focuses on mechanical properties. The strength properties of the currently available nanocomposites are far below those that theoretically are available with nano or micro sized reinforcements. One of the reasons is the difficulty in distributing the particulates in the polymer matrix which is still unsolved for some reasons. Fine distribution has been already done not only in laboratory conditions and in small quantities that are not applicable in industrial processes or just with very difficult and costly technologies.

Many uses of nanoparticles are known and are emerging in new applications. A new potential application environment can be the production of hybrid composites, and the addition of the nanoparticles to conventional fibres in the polymer matrix. Nanoparticles may exhibit their positive effects in hybrid compositions in a number of ways and may provide solution for some difficulties. On one hand, the nanoparticles in the composite may be suitable for better distribution of the load in the matrix and on the other hand the particles may appear in the fibre-matrix boundary phase and can change their relationship and improve the load transfer. In this case, when using nanoparticles in hybrid systems, it is expected that

besides increasing the strength properties, a more durable material could be created and it could bear better the repetitive stresses.

2. SUMMARY OF THE LITERATURE, OBJECTIVE

In the field of composites, we have deeper knowledge mainly in those that containing traditional fibres. This wide knowledge stands for both to the material structure, to the mechanical properties and to the failure processes of these materials. In the field of traditional composites, the most challenging thing to make these materials though while retaining the strength properties.

With the application of nanoparticles these and many other difficulties can be solved with numerous advantages in the solution, but processing technologies do not allow yet their large industrial spread. Some studies have shown that nanoparticles can be used to increase the strength of a composite however, its toughness did not decrease relative to the matrix. Nevertheless, due to the size of the particles, the composites formed therefrom, are very sensitive to the processing parameters (which further complicates their wide spread), resulting that even in the same matrices we can observe very different phenomena. The size of the applied reinforcing material in some cases can be comparable to the size of the polymeric chain, therefore this may have a significant effect locally and globally on the structure and properties of the produced composite. The broad and really accurate examination and explanation of the systems always poses a challenge to the researchers, but our knowledge in this area is continuously getting deeper.

Using nanoparticles with another micro-size enhancer can help relieve sensitivity to production. The use of the two different sizes of reinforcing materials can give the created hybrid system unique attributes. Research and application of hybrid composites containing reinforcing materials that have different properties are increasingly being investigated and applied however, their use extends almost entirely to composites containing fiber reinforcement and thermoset matrix. The application of nanoparticles is more commonly used besides fiber-shaped reinforcements in thermoset matrices. The main reason for this is that the price of nanoparticles is still relatively high, despite of the increasing demand and usage, and therefore their use in thermoplastic matrix composites is still hampered because the price-sensitivity of the high-production industries. However, the research that has been carried out so far suggests the appearance of particles in these industries. For a small increase in cost, the strength of the composite can be further increased, its viscoelastic behavior can be reduced, and the resistance of these composites to long-term loads too. Therefore in my research I use

polyamides that are widely used in the industry and I produce different hybrid composites and show the benefits of their applications while examining the structure of the created systems and explaining the experienced phenomena.

Objectives:

- Development and comparison of carbon fiber and basalt fiber reinforced hybrid composites.
- Presentation of the effect of nanoparticles and examination of the mechanical properties of the created hybrid composites.
- Testing of the structure of hybrid composites, showing the combined effect of reinforcing materials
- Evaluation of the loadability of composites by quasi-static tests, especially the dependence of the carbon content on deformation components.
- Exploring the long-term behavior of carbon fiber and carbon nanotube hybrid composites for constant (creeping) and cyclical loads (tiring wear).

3. USED MATERIALS, METHODS AND EQUIPMENTS

Materials

Schulamid 6 MV 13 type polyamide 6 (PA 6) from A. Schulman GmbH (Germany) was used as matrix material. BCS KV02 type basalt fiber (BF) from Kamenny Vek Ltd. (Russia) and Panex 35 type 95 carbon fiber (CF) from Zoltek Zrt (Hungary) was applied as micro-sized reinforcement. The initial length of both fibers was 6 mm. The average diameter of basalt fibers was $15.6 \pm 1.9 \mu\text{m}$ and that of carbon fibers was $8.3 \pm 1.0 \mu\text{m}$. NANOCYL NC7000 carbon nanotubes supplied by Nanocyl SA. (Belgium) were used as nano-size reinforcement. Nanotubes had an average length of $1.5 \mu\text{m}$ and an average diameter of 9.5 nm. The minimal carbon purity of nanotubes was 90% and nanotubes had no surface treatment.

Manufacturing of composites and specimens

Before the melt mixing the PA6 was dried at $80 \text{ }^\circ\text{C}$ for 4 hours in a hot air dryer (Heraus UT-20). The components of the composites were mechanically mixed in a sealed vessel (shaking), and every 1 minute they were remixed to avoid settling. A Labtech Scientific LTE 26-44 type twin screw extruder was used for continuous melt mixing, the screw had a length/diameter ratio of 44. The rotational speed of the screws was 25 rpm. The

temperatures of the zones from spout to the tool were: 225-230-230-235-235-240-240-245-245-245-250-250 °C. The names and compositions of the composites can be found in Table 1.

Name	PA6 [weight%]	BF [weight%]	CF [weight%]	CNT [weight%]	PA6 [volume%]	BF [volume%]	CF [volume%]	CNT [volume%]
PA6	100	0	0	0	100	0	0	0
PA6 / 0,25 CNT	99,75	0	0	0,25	99,8	0	0	0,2
PA6 / 0,5 CNT	99,5	0	0	0,5	99,6	0	0	0,4
PA6 / 0,75 CNT	99,25	0	0	0,75	99,39	0	0	0,61
PA6 / 1 CNT	99	0	0	1	99,19	0	0	0,81
PA6 / 30BF	70	30	0	0	84,65	15,35	0	0
PA6 / 30BF / 0,25 CNT	69,75	30	0	0,25	84,39	15,36	0	0,24
PA6 / 30BF / 0,5 CNT	69,5	30	0	0,5	84,14	15,37	0	0,49
PA6 / 30BF / 0,75 CNT	69,25	30	0	0,75	83,89	15,38	0	0,73
PA6 / 30BF / 1 CNT	69	30	0	1	83,63	15,39	0	0,98
PA6 / 30CF	70	0	30	0	78,89	0	21,11	0
PA6 / 30CF / 0,25 CNT	69,75	0	30	0,25	78,65	0	21,12	0,23
PA6 / 30CF / 0,5 CNT	69,5	0	30	0,5	78,41	0	21,13	0,46
PA6 / 30CF / 0,75 CNT	69,25	0	30	0,75	78,17	0	21,14	0,68
PA6 / 30CF / 1 CNT	69	0	30	1	77,93	0	21,15	0,91

Table 1: Composition and name of the produced composites

Dumbbell specimens with a nominal cross-section of 4x10 mm according to MSZ EN ISO 527-2-1A were produced with injection molding. Before the molding, the materials were also dried at 80 °C for 4 hours. For injection molding, Arburg Allrounder Advance 370S 700-290 injection molding machine was used. The temperature of the injection molding zones (from the spout to the tool) were 255-260-265-270-275 °C. The tool temperature was 80 °C, injection molding speed was 40 cm³/sec, value and time of packing was 400bar and 20sec, and maximum molding pressure was 800 bar.

To measure the thermal conductivity of the composites, after extrusion, 4 mm thick sheets with a surface area of 80 x 80 mm were fabricated with a Collin Tech-Line Press Plate 200E hydraulic press. The pressing was carried out at 250 °C with a pressure of 150 bar.

Characterization methods

Before the mechanical tests, the specimens were conditioned at 50% relative humidity at room temperature for a month (magnesium nitrate, 25 °C - RH 52.9 ± 0.22). The tensile tests were performed on Zwick Z020 universal testing machine according to MSZ EN ISO 527-2:2012. During the tensile tests the force-displacement graph was registered. Young's

modulus was determined on the steepest part of the tensile curve. The tests were performed at 5 mm/min speed. In all cases, the measurements were performed on at least 5 test specimens.

During the quasi static cyclic tests, a stepwise increasing loading was used. It was raised 100 N in every cycle (Figure 1. a). Between two cycle, there was a 30 seconds relaxation step (0 N). In all cases, the tests were performed on at least five specimens. During the test, the displacement-time diagraph (Figure 1. b) and the force-displacement diagraph were registered for evaluation.

During the evaluation, the residual deformation (ϵ_m) and the total elastic deformation (ϵ_r) components were determined in every cycle. Based on the test results, the maximum (Δl_{max}) and minimum deformation (Δl_{min}) were measured, than the value of residual elongation in the actual cycle was calculated:

$$\epsilon_{m\ n} = \frac{\Delta l_{min\ n} - \Delta l_{min(n-1)}}{l_0 + \Delta l_{min(n-1)}}, \quad (3.1.)$$

The ratio of elastic recovery was determined with following equation:

$$\epsilon_{r\% \ n} = \frac{\Delta l_{max\ n} - \Delta l_{min\ n}}{\Delta l_{max\ n} - \Delta l_{min(n-1)}} \cdot 100. \quad (3.2)$$

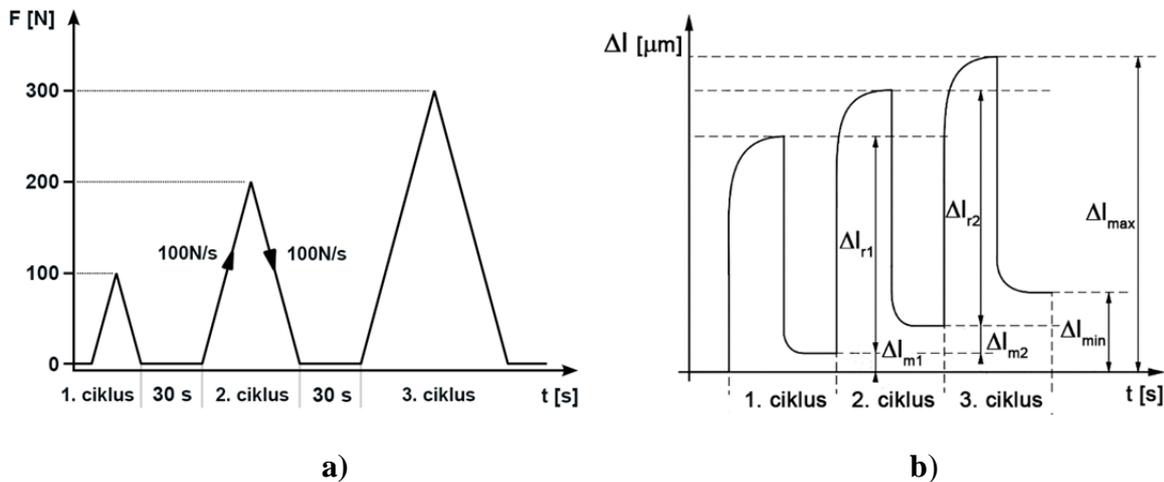


Figure 1. Excitation of the quasi static cyclic test during the time (a) and the theoretical deformation-time diagraph with the measurable values (b)

These test were performed until the failure of the specimen (fracture), or if a significant creep of the test specimens was occurred during the test (1% elongation without force increment).

The creep tests were performed on Zwick Z005 universal testing machine. Before testing, the specimens were also conditioned in exsiccator. In the first period of the test the

creep behaviour of the material was investigated on a constant load for a half hour, than the relaxation of the specimen was observed for another half hour. The tests were carried out at 3 different load levels for all composites (5, 12.5 and 25 MPa load were applied for fiber-reinforced composites and 12.5 for 25 and 50 MPa for composite fibers). The tests were repeated on at least one test specimen.

The DMA (Dynamic Mechanical Analysis) tests were performed on TA Instruments Q800. Samples with nominal sizes 56 x 10 x 4 mm were manufactured from the middle of the injection molded specimens (Dumbbells). The DMA tests were carried out in 3-point bending configuration (three repetitions per material). The measured temperature range was -50 °C to 100 °C. The heating rate during the test was 3 °C/minute and a frequency was 1 Hz.

The temperature-time superposition measurements were also carried out in DMA equipment in a three-point bending configuration (50 mm support). The temperature range was -30 °C to 60 °C. Before each load step the temperature was increased 10 °C and equilibrated for 10 minutes to reach uniform temperature in the whole specimen. At each temperature 15 minute creep test followed by 15 minutes relaxation. For polyamide and nanocomposites the load was 5 MPa, while the fiber reinforced composites and hybrids measured at 8 MPa. For each material, the measurement was repeated once.

Tensile-tensile fatigue tests were performed on an INSTRON 8872 hydraulic fatigue load system. The machine was equipped with 30 kN hydraulic grips. The composites tested in tensile-tensile mode (Figure 3.), which means continuously tensile load. In this type of specimens the compression mode is forbidden to avoid buckling. The load factor was 0.1 ($R_t = \sigma_{\min}/\sigma_{\max}$, where σ_{\max} was maximum applied load and σ_{\min} was the minimum). During the preliminary experiments, the appropriate load frequency was determined with frequency loop tool (PID control setting were tuned in this session). During the tuning the maximum load frequency was described in 2 Hz. At least 5 test pieces were tested at each load level and at least 3 load level were applied to describe load-cycle number (S-N) curves. During the testing, the temperature of the specimens was examined by FLIR A325sc thermal camera. Temperature values were recorded and evaluated using Flir R & D software. Standard sponge-like specimens were fractured at 200 mm / min to get information the strength of the materials at high speed and specify loads for fatigue tests.

The melt flow index (MFI) was measured on a CEAST 7027.000 type computer controlled capillary plastometer according to MSZ EN ISO 1133. The measurements were

made after extrusion. Before the tests the materials were dried at 80 °C for 4 hours. Capillary length was $L = 8$ mm, radius $r = 1.0475$ mm and the piston diameter was $D = 9$ mm. The tests were performed at 260 °C. The total weight of the applied load disks was 2.16 kg.

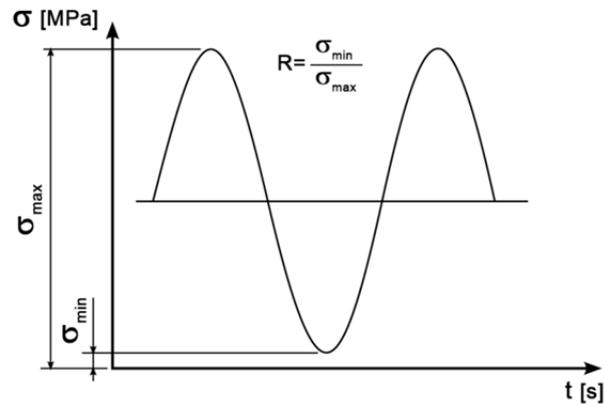


Figure 3.: The applied sinusoidal load during fatigue test

To determine the crystallinity of matrix in the composites, differential scanning calorimetry was performed on TA Instruments Q2000 DSC. From every composites 5 specimens were investigated, this shards were manufactured out from the middle of tensile test specimens. During the measurements the heating and cooling rate was 10 °C/min. The measurements were carried out in an inert atmosphere (N₂, 50 mL / min flow rate). Polyamide 6 has two stable (α and γ) crystalline modification, these variants having different melting temperatures and melting heat. This means that a different melting temperature belongs to the theoretically 100% α and 100% γ crystalline material, but in practice these melting heat values are nearly the same, therefore, an average value is used which is 230 J / g (± 20 J / g). For the determination of the crystalline fraction, the relation of 3.4 was used.

$$X_{kr} = \frac{\Delta H_m}{\Delta H_c \cdot (1 - \varphi)} [\%], \quad (3.4.)$$

where ΔH_m is the measured melting heat, ΔH_c is the average crystal melting heat for polyamide 6 and φ is the fiber content of the composite in weight%.

To determine the fiber content, different part of the tensile test specimens was investigated. The matrix material was burnt out at 500 °C for 1 hour (air atmosphere) in a laboratory furnace, after the specimens cooled down the mass of residual fibers was measured to determine the fiber content of the composites. These fibers also investigated with Olympus BX 51M optical microscope and a Stream Motion program to get information of the fibers length after processing. The Stream Motion software was used to manually measure the fiber

length. In order to determine the fiber orientation, section made from the dumbbell specimens. These sections were polished with Struers LaboPol-5 polishing machine to prepare the appropriate surface. From these surfaces panoramic footage was prepared, and it was analysed in ImageJ image processor.

The fracture surfaces were analyzed in JEOL JSM 6380LA scanning electron microscope (SEM). Before the test, the surface of the samples was coated with a thin gold layer using the JEOL JFC-1200 coater. The purpose of coating was to make a conductive surface to avoid the charging of the sample. In order to determine the length of the protruding fibers, pictures were also taken from a perpendicular view. In this investigation only fibers with a deviation of less than 5 ° were measured (at least 250 pieces for each composite).

In order to reveal the surface crystallinity of the samples, X-ray diffraction was carried out on X'pert Pro MPD (PANalytical) X-ray diffractometer (Department of Inorganic and Analytical Chemistry). The equipment was equipped with Emyrean Cu X-ray tube (40 kV, 30 mA) and Ni filter. The test range (θ) was 4-44 °.

The thermal conductivity of the produced composites was measured on asymmetrical hotplate device which developed by the Department of Polymer Engineering. The surface of the pressed plates was coated with T-Silox silicone-based heat conducting paste. The measurements were carried out on two different sheets .With the results of the measurement the thermal conductivity was calculated from the following equation:

$$\dot{Q} = -\lambda \cdot A \frac{dT}{dx}, \quad (3.6.)$$

where \dot{Q} [W] is the heat flux, λ [W/mK] thermal conductivity, A [m²] is surface of the plate and dT/dx [K/m] is the heat gradient.

4. SUMMARY

During my work, nanocomposites containing carbon nanotube (CNT), microfiber reinforced composites containing basal fiber (BF) or carbon fiber (CF) and hybrid composites were produced. During preliminary experiments, the fiber content was chosen as 10, 20 and 30% by weight. This value was constant in my later studies at 30% by weight, while the rate of CNT content was varied between 0 and 1% by weight. In order to make the research results easier to use in industry, I used commercially available materials, and the composites were made by melting mixing (twin screw extruder). While I was doing my research, my aim was

to reveal the effect of CNT content on hybrid composites based on different fibers. Introducing their behaviour during different excitations and searching for answers to the material structure responses.

My preliminary experiments and more detailed material testing showed that the production of hybrid composites improves the distribution of nanoparticles. The improved dispersion is due to the presence of fibers that during processing facilitate separation of the particles in the melt, preventing the aggregates from being stuck together. This effect is the more significant the more fibers are found in the composite, so I chose 30% by weight of the fiber content to perform further tests. There is no sign of aggregates on this reinforcement content, despite that in composites containing only nanoparticles, large aggregates can be observed.

Thanks to the separation of the particles, the untreated carbon nanotubes, which do not have any treatment on the surface that improves the adhesion, have been able to exert a strength-enhancing effect. This beneficial effect has not only affected the strength of the hybrid composites, but also their modulus. The maximum strength and modulus values were demonstrated in both basalt fiber and carbon fiber composites when using 0,5 weight percent nanoparticles. Further increase in CNT content has already resulted in stagnation or a slight decrease in strength values. After I measured the average fiber length in the composites, I could examine indirectly the protruding fiber length. This showed that the carbon nanotubes did not have any detectable effect on the fiber-matrix linkage in the produced hybrid composites. According to this result, the increase in mechanical properties can be explained by the presence of the nanotubes as reinforcement and its load homogenizing effect. Nanoparticles may, as a result of their size, affect the crystallinity of the matrix. Therefore, I examined the crystalline structure of the composites by calorimetry tests and experienced that the carbon nanotubes does not have a significant effect on the crystalline ratio of the matrix, but it does on the crystalline form ratio. With the dissolution of the DSC curves, I demonstrated that due to the presence of the nanotube, the ratio of the more instable γ crystals in the polyamide 6 matrix decreased, leaving space for the more stable α crystals. These results were confirmed by X-ray diffraction. This change in the ratio of crystalline forms may help to slightly increase strength. While separating the curves, I applied the asymmetric double sigmoid function that was not applied before to the thermoplastic composites. I have shown that this function can be matched well to melting curves with multiple peaks where different peaks can be detected during the tests.

Besides the classical mechanical studies, the load capacity of the composites and the ratio of the flexible and residual deformation at different loads with a special quasi-static cyclic test were examined. According to the tests, it can be stated that the carbon nanotube did not only increase the strength of the hybrid composite but also reduce the residual deformation at all loads by its presence. The large amount of carbon nanotubes (high in numerosity) significantly reduces the relative displacement of the polymer chains, thereby helping to properly disperse the loading force. The resulting network structure increases the force that needed for moving the chains, which appears in a more rigid behaviour. The same behaviour can be observed in nanocomposites which may be caused by the partially dispersed particles. Changes in the ratio of deformation components can be seen during creep tests thus it can be said that with the use of CNT, the creep susceptibility of nano and hybrid composites decreased. The deformation components determined by the cyclic studies overestimate the creep experienced during tests in any case, but the effects of the reinforcing agents can be traced well during cyclic studies. Based on the study of the models that describe creep, it is perceivable that Findley's power law is well suited to the creep of the hybrid composites that I have created. Deliberatively to this I have determined a suitable connection for design tasks for the compositions I have created.

During the fatigue testing of the composites I switched to a higher speed range. The nanocomposites showed significant drop in the number of cycles associated with failure at dynamic loads compared to pure polyamide. At cyclic loads, the aggregates' stress concentrating nature showed up and therefore accelerates the failure process. The fatigue of composites that containing basalt fiber fatigued sooner than the carbon nanotube. This phenomena is connected to the smaller crystalline ratio, the decreasing fiber length and the presence of fragments resulting from the formation of basalt fibers. However, the addition of carbon nanotubes to carbon fibers was successful. Based on the many tests carried out on a different loads, it can be said that the CNT always increases the cycle number associated with the failure at the same load level or the load was higher than before the hybridization at same number of cycles. This positive effect of the carbon nanotubes is getting more and more significant as the load decreases. While the composite's temperature was tested, at lower loads, the steady state can be observed by increasing temperature, which is the consequence of the process slowed down. The examination of the apparent modulus of the composites has confirmed this, as its decline in cyclic fatigue slowed down too, most likely due to the presence of carbon black. After breakage the specimens' break surface was analysed. When the fatigue test was stopped after a certain number of load cycles the specimens were

cryogenic cracked in order to analyse the breakage process. At this point I found smaller cracks in the carbon-containing samples. It can be stated that the CNT slows down the cracking process by fatigue, thereby delaying the failure of the specimens. As a consequence, it can be said that irrespectively from the speed of the test and the applied load force, the hybrid composites are more resistant to the various loads due to the added carbon nanotubes. Therefore, not only more durable, but also increased lifetime could be achieved by components using carbon fiber and carbon nanotube reinforced polyamide matrix hybrid composites.

5. THESIS

1st By adding multi-walled carbon nanotube to the polyamide 6 matrix composites with 30 weight% fibre content, the tensile strength and Young's modulus of the hybrids were significantly increased (maximum 1 weight% of MWCNT), while the created composites do not behave more rigid. This effect is independent of the fact that the microfiber reinforcement is a basalt or carbon fiber. Maximum strength increment (10%) was detected on 0.5 weight% CNT use. The effect is independent from the type of microfibre because the carbon nanotube role is small in the matrix-fiber adhesion. The carbon nanotube exerts its effect mainly by changing the properties of the matrix. Strength growth is a result of the proper dispersion of the particles melt, which is the result of the fiber shear rate increasing effect in the melt during melt mixing.[1–3]

2nd Melting curves of polyamide 6 and its composites with two stable crystal form can be decomposed with asymmetric double sigmoid function. This special function follows the shape of the real crystalline melting curves:

$$y = y_0 + A \frac{1}{1 + e^{-\frac{x-x_c+w_1/2}{w_2}}} \left(1 - \frac{1}{1 + e^{-\frac{x-x_c-w_1/2}{w_3}}} \right)$$

Dissolved carbon nanotubes up to 1 weight% in polyamide 6 matrix hybrid composites, just like the nanoparticles composite, behave as a nucleus, the presence of microfibrils has no significant influence on this kind of behavior of nanoparticles. Using this function, it can be shown that the carbon nanotube increases the ratio of the more stable α crystalline form.

- 3rd** Up to 1 weight% carbon nanotube content, CNT reduce the deformation of nano composites and hybrid composites (basalt or carbon fiber) at each load level and nanoparticle vary the ratio of deformation components during tensile test. This can be explained by the presence of partially dispersed particles which are partly impregnated by the matrix. In hybrid composite it can be explained with the physical crosslink formed by properly dispersed particles that reduces the ability of relative displacement of the chains and the stress homogenizing effect of the carbon nanotubes. Due to the more flexible behaviour and the decrement of the residual deformations, the creeping behaviour of the composites also decreased. [1, 4, 5]
- 4th** Addition of carbon nanotube in to carbon fiber reinforced polyamide 6 composites increase the cycle number connected to the fracture during fatigue tests. It can be explained with the fracture propagation speed decreasing effect of carbon nanotubes, and with the decreased loss factor of the composites. Due to this phenomena the heat consumption decrease during fatigue loads. [3, 6]

6. REFERENCES

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