

Report

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From:

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For mobility program:

06.06.2006 – 30.09.2006

hosted in Institut für Verbundwerkstoffe GmbH (IVW),

DE – Kaiserslautern

Topic:

Improvement of mechanical properties of nanocomposites made of
thermoplastic material and ceramic nanoparticles

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30.09.2006

Table of contents

I.	Motivation	3
II.	Information about IVW	3
III.	Activities in the training period	3
1.	Manufacturing of nanocomposites of thermoplastic matrix and nanoparticles by varying the particle content and the process parameters	3
2.	Preparing the samples for the fracture tests	5
3.	Determination of the mechanical properties of the nanocomposites (tensile, flexural and impact testing)	5
1)	Tensile test	6
2)	Flexural Tests	6
3)	Impact tests	7
4.	Analysis of the fracture surfaces by scanning electron microscopy in order to evaluate the dispersion quality and the failure mechanisms of the samples	7
5.	Interpretation of the results gained by the tests	7
IV.	Feedback	8

I. Motivation

I participated in 3,75-months mobility program in Institut für Verbundwerkstoffe GmbH (IVW) because of the relation between the task and studies in my university. That allowed me to get deeper in the nanocomposite technologies and to compare them with the Bulgarian style of doing the same.

II. Information about IVW

Institut für Verbundwerkstoffe GmbH (IVW) is a non-profit research institute of the state Rhineland-Palatinate, Germany, exploring and advancing applications and potential applications of composite materials, based on polymer matrix systems. In 1990 the institute was founded on the campus of the University of Kaiserslautern and extended in 2002. IVW has three divisions: Design and Analysis, Materials Science Division and Manufacturing Science Division.

The missions of IVW are:

- to develop the technical applications and potential applications of composites and provides material, process, and product development up to the support of pilot series from one source.
- to maintain interdisciplinary, scientific co-operations with local, national and international partners – one of the reasons why I could participate in this mobility program.
- to transfer its findings and expertise into industry and scientific public, and, in particular, into the education of engineers at the University of Kaiserslautern.

III. Activities in the training period

By the insertion of nanoparticles in a polymer matrix, relevant mechanical properties like the flexural strength, the Young's modulus, the fracture toughness and the impact strength can be improved simultaneously. The key to gain an effective reinforcement of the polymer is to break up the agglomerates and to distribute the nanoparticles homogeneously within the matrix. The object of the proposed work is to incorporate various amounts of nanoparticles systematically into a liquid thermoplastic matrix by using extrusion processing. To achieve high dispersion and small particles process parameters are variegated with differing material and particles. The mechanical properties of the nanocomposites are examined by means of tensile, flexural and impact testing. A fracture mechanics approach may provide information about the material's toughness and also the reinforcing mechanisms of the nanoparticles. To evaluate the quality of the dispersion the fracture surfaces of the sample are observed with a scanning electron microscope.

1. Manufacturing of nanocomposites of thermoplastic matrix and nanoparticles by varying the particle content and the process parameters

The manufacture of nanocomposites of thermoplastic matrix and nanoparticles was obtained in a molten state using two apparatuses – extruder and injection molding machine.

The process of extrusion is nowadays one of the most important processing methods in the

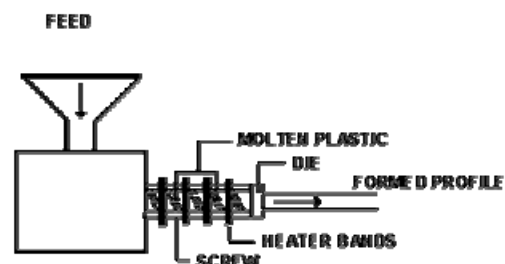


Fig. 1 – Scheme of extruder machine

plastic industry. About 60% of the thermoplastics manufactured world-wide are processed by means of this technology.



Fig. 3 – Extruder “Berstorff ZE 25A”

The plastic is fed in pellet form into the machines hopper (this machine is known as an Extruder). The material is conveyed continuously forward by two rotating screws inside a heated barrel, being softened by both friction and heat. The softened plastic is then forced out through a die and directly into cool water where the product solidifies. From here it is conveyed onwards into the take-off rollers, which actually do the pulling of the softened plastic from the die. You could see the

scheme at Fig. 1.

For our task we used Berstorff twin-screw extruder (Fig. 3). The temperatures in the tenth zones were set in dependence of the softening temperature and the melting temperature of the produced nanocomposites from TiO_2 (15 nm) particles and polyamides, polycarbonates. Thereby the particle volume amounts were varied systematically. The extrudate, when coming out of the 2 mm die, was immediately drawn by a drawing device, which resulted in a final diameter of about 0,6 – 1mm. The draw ratio was calculated as the ratio between the cross section areas of the die and the drawn bristles. The continuous bristles were pelletized by a pelletizing system (Fig. 2). The scheme of the processes of extrusion, drawing and pelletizing are shown in Fig. 4.



Fig. 2 – Pelletizer Scheer SGS 50 E

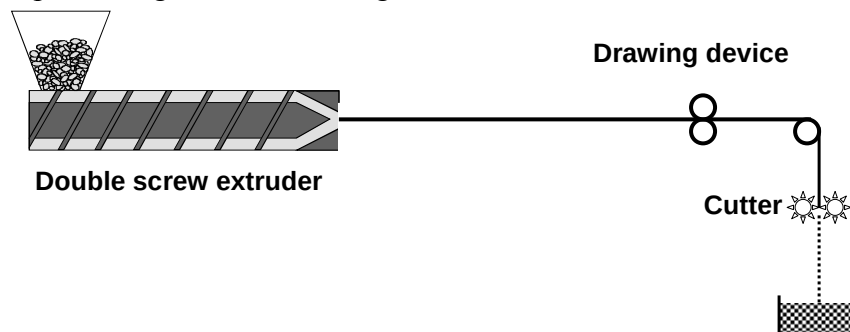


Fig. 4 – Extrusion -> drawing -> pelletizing

Injection molding (IM): An injection molding machine consists of three basic parts; the mold plus the clamping and injection units. The clamping unit is what holds the mold under pressure during the injection and cooling. Basically, it holds the two halves of the injection mold together. During the injection phase, plastic materials in the form of pellets are loaded into a hopper on top of the injection unit. The pellets feed into the cylinder where they are

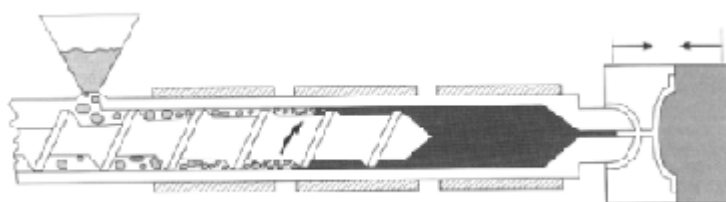


Fig. 5 – Injection molding machine cylinder

heated until they reach molten form. Within the heating cylinder there is a motorized screw that mixes the molten pellets and forces them to the end of the cylinder (Fig. 5). Once enough material has accumulated in front of the

screw, the injection process begins. The molten plastic is inserted into the mold through a sprue, while the pressure and speed are controlled by the screw. Next phase – the dwelling phase consists of a pause in the injection process. The molten plastic has been injected into the mold and the pressure is applied to make sure all of the mold cavities are filled. The plastic is allowed to cool to its solid form within the mold.



Fig. 6 – Injection molding machine “Arburg 270 U”

For this purpose the pellets were dried for at least 24 hours at 80°C (PA) and 120°C (PC). In order to preserve the PA fibrils, the materials were injection molded at specific temperatures. An injection molding machine of the type Allrounder 270 U 500-150 of the company Arburg was used (Fig. 6). The machine possesses 5 partitioned heating zones, a snail of 35 mm in diameter and an effective length of $L/D = 17$. Hereby a maximum

injection pressure of 1450 bar and an injecting stream of max. 180 cm³/s are possible.

2. Preparing the samples for the fracture tests



Fig. 7 – IM sample

Most fracture tests use symmetric specimens.

The injection-molded samples are not ready for testing (Fig. 7).

In order to prepare appropriate samples for the different tests it is necessary to cut them. For this operation a cutting machine is used (Fig. 8). It is important to use then appropriate band for

thermoplastic materials. At the same machine the samples for flexural and impact testing are cutted. For the test that we made they had to be with an accurate size: 80 x 10 x 4 mm. Then we take some of these specimens in order to make notches for Charpy test.



Fig. 8 – HEMA Bandsaw SR 500

3. Determination of the mechanical properties of the nanocomposites (tensile, flexural and impact testing)



Fig. 9 – Zwick 1485

Tensile and flexural tests are made on the machine Zwick 1485 (Fig. 9). Since the physical properties of many materials (especially thermoplastics) can vary depending on ambient temperature, it is sometimes appropriate to test materials at temperatures that simulate the intended end use environment. We made tests at ambient temperature as also at 80°C.

For 80°C tests a thermal chamber is installed on the Zwick test machine. The chamber is designed to allow the test mounts from the base and crosshead of the Zwick to pass through the top and bottom of the chamber. Standard test fixtures are installed inside the chamber, and testing is conducted inside the controlled thermal environment the same as it would be at ambient temperature. The chamber has internal electric heaters for elevated temperatures and

uses external carbon dioxide gas as a coolant for reduced temperatures.

1) Tensile test

A tensile test, also known as tension test, is probably the most fundamental type of mechanical test you can perform on material (Fig. 10). Tensile tests are simple, relatively inexpensive, and fully standardized. By pulling on something, you will very quickly determine how the material will react to forces being applied in tension. As the material is being pulled, you will find its strength along with how much it will elongate.

You can learn a lot about a substance from tensile testing. As you continue to pull on the material until it breaks, you will obtain a good, complete tensile profile. The result is a curve showing how it reacted to the forces being applied. The point of failure is of much interest and is typically called its "Ultimate Strength" or UTS.

In the initial portion of the test, the relationship between the applied force, or load, and the elongation the specimen exhibits is linear. In this linear region, the line obeys the relationship defined as "Hooke's Law" where the ratio of stress to strain is

a constant, or $E = \frac{\sigma}{\epsilon}$. E is the slope of the line in this region

where stress (σ) is proportional to strain (ϵ) and is called the "Modulus of Elasticity" or "Young's Modulus".

The modulus of elasticity is a measure of the stiffness of the material, but it only applies in the linear region of the curve. If a specimen is loaded within this linear region, the material will return to its exact same condition if the load is removed.

At the point that the curve is no longer linear and it deviates from the straight-line relationship, Hooke's Law no longer applies and some permanent deformation occurs in the specimen (Fig. 11). This point is called the "elastic, or proportional, limit". From this point on in the tensile test, the material reacts plastically to any further increase in load or stress. It will return only partly to its original, unstressed condition if the load were removed.



Fig. 10 – Tensile test



Fig. 11 – Tensile test break

2) Flexural Tests

The Flexural test measures the force required to bend a beam under 3 point loading conditions. The data is often used to select materials for parts that will support loads without flexing. Flexural modulus is used as an indication of a material's stiffness when flexed.

The specimen lies on a support span and the load is applied to the center by the loading nose producing three point bending at a specified rate.

The parameters for this test are the support span; the speed of the loading; and the maximum deflection for the test. These parameters are based on the test specimen thickness, and are defined differently by ASTM and ISO.

Flexural strength, flexural stress at specified strain levels, and flexural modulus can be calculated.

These tests were done using a tensile testing machine, Zwick 1485, at a loading speed of 2 mm/min. The principle scheme is shown in Fig. 12.

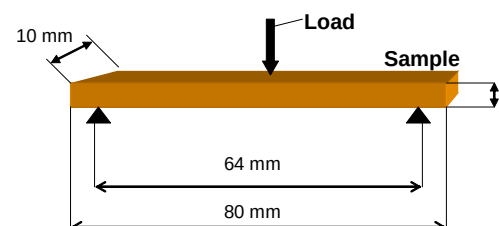


Fig. 12 – Three point bending test scheme

3) Impact tests

These experiments were done on a Charpy impact-testing machine (CEAST pendulum). The principle scheme is shown in Fig. 13. The samples had the same shape as those used for the three point bending tests. The clamp distance was 60 mm, the energy = 4 J, and the speed was 3.7 m/s.

The Charpy impact test measures the energy absorbed by the high strain rate fracture of a standard notched specimen. The specimen is broken by the impact of a heavy pendulum hammer, falling through a fixed distance (constant potential energy) to strike the specimen at a fixed velocity (constant kinetic energy). Tough materials absorb a lot of energy when fractured and brittle materials absorb very little energy. The impact energy measured by the Charpy test is the work done to fracture the specimen.

On impact, the specimen deforms elastically until yielding takes place (plastic deformation), and a plastic zone develops at the notch. As the test specimen continues to be deformed by the impact, the plastic zone work hardens. This increases the stress and strain in the plastic zone until the specimen fractures.

The Charpy impact energy therefore includes the elastic strain energy, the plastic work done during yielding and the work done to create the fracture surface. The elastic energy is usually not a significant fraction of the total energy, which is dominated by the plastic work. The total impact energy depends on the size of the test specimen, and a standard specimen size is used to allow comparison between different materials.

The notch in the test specimen has two effects. Both can decrease the impact energy. First, the stress concentration of the notch causes yielding or plastic deformation to occur at the notch. A plastic hinge can develop at the notch, which reduces the total amount of plastic deformation in the test specimen. This reduces the work done by plastic deformation before fracture. Secondly, the constraint of deformation at the notch increases the tensile stress in the plastic zone. The degree of constraint depends on the severity of the notch (depth and sharpness). The increased tensile stress encourages fracture and reduces the work done by plastic deformation before fracture occurs.

Some materials are more sensitive to notches than others, and a standard notch tip radius and notch depth are therefore used to enable comparison between different materials. The Charpy impact test therefore indicates the notch sensitivity of a material.

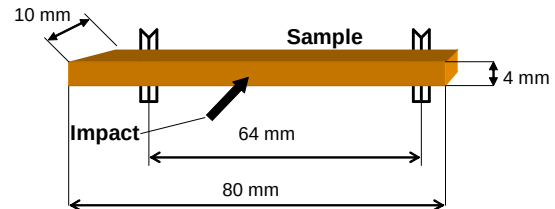


Fig. 13 – Charpy test scheme

4. Analysis of the fracture surfaces by scanning electron microscopy in order to evaluate the dispersion quality and the failure mechanisms of the samples

The scanning electron microscope (hereinafter abbreviated to SEM) is utilized not only in medical science and biology, but also in such diverse fields as materials development, metallic materials, ceramics, and semiconductors. The scanning electron microscope is a type of electron microscope capable of producing high resolution images of a sample surface. Due to the manner in which the image is created, SEM images have a characteristic three-dimensional appearance and are useful for judging the surface structure of the sample.

5. Interpretation of the results gained by the tests

The final testing results are private property of IVW and Nicole Knör and I couldn't share them before article publishing.

IV. Feedback

As a result of 3,75 internship I was able to gain experience working with all the mentioned machines above. I received practically the knowledge how to produce nanocomposite materials, how to investigate their properties and how to analyze the test results.

My stay in Kaiserslautern was with fixed daily-work time from 8 to 17 with one-hour lunch brake. During that time I had the opportunity to receive a lot of new practical and theoretical knowledge thanks to my supervisor Nicole Knör. She gave me a lot of tasks concerning the topic of my mobility program.

It was a great pleasure to work and meet a lot of scientists from a different countries working over the topics of nanocomposite materials. I made a lot of friendships that I hope will keep and develop in further. The mobility program came for me exactly after I have listened a course for nanocomposite and plastic materials. After I spent this mobility term in IVW I hope that I will have the opportunity to continue here with my master or PhD degree.

I really appreciate the time that I spent in Kaiserslautern because it gave me the possibility to advance in the language. I didn't know even a word in the beginning, but now I'm able to request some simple things (I'm doing all in order to improve my German). That is why I think that a language course during the whole mobility stay would be a better solution. I estimate highly the willingness of my tutor that spoke with me in German, but the problem was from my side that she was obliged to speak as slowly and as simple as possible.

Concerning the cultural exchange – I had a great possibility to meet and visit in the weekends a lot of German towns, to make a lot of friendships. Unfortunately the provided scholarship is not enough for a normal stay in the country and it was necessary to loan a lot of money and to spend almost all that I had saved. You could think over the question for increasing the scholarship, because it will allow better cultural exchange.

Even there was a long work days and very sunny days I'm really satisfied that I had the opportunity to participate in this mobility program. I hope that it was not my last chance.

At the end I would like to thank to all the people that were concerned with my stay here in Kaiserslautern and generally to Nicole Knor and Minko Kostov.