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SURFACE TOPOGRAPHY OF COMPOSITE REINFORCED WITH FIBRES FROM USED TYRES

Lucia Knapčíková
Technical University of Košice, Faculty of Manufacturing Technologies with a seat in Prešov, Department of Industrial Engineering and Informatics, Bayerova 1, 080 01 Prešov, Slovak Republic, lucia.knapcikova@tuke.sk

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Abstract: This paper aims to study the surface topography of composite reinforced by fabric from used tyres. By the research was used an atomic force microscopy (AFM). AFM can drive the force between the sense probe and the surface and the in Z axis can move piezo and sensor. The composites were reinforced with fibres from used tyres. After homogenization the thermoplastics matrix and fibres from used tyres we pressed test specimens and after this technology was material tested by atomic force microscopy. Generally we can say, therefore, it provides important information about the surface of the display material and its properties that are necessary to know for the further examination, in particular for of utilization the material and displayed by using atomic force we can get a clearer idea of the investigated materials and other behavior in a various mechanical tests.

1 Introduction

Atomic force microscopy, mainly AFM operates in a constant amount or under constant force (load) and according to the required feedback between the carrier and piezo. If the feedback is week while surface capturing, amount of the piezo and sample remains unchanged and the rate of change on the surface (of relief) is bending carrier with tip. In this case, the force between the tip and the surface varies. If the feedback is strong, piezo amount varies according to the surface relief in order to bending of the carrier with the tip and thus the transmitted power were constant. [1,2,4] AFM can drive the force between the sense probe and the surface and the in Z axis can move piezo and sensor. Mostly is used just constant load mode, for example in a situation when there are not display surfaces of samples with solid periodic structures, but fragile organic samples as DNA, protein, blood cells, Langmuir-Blodgett films, and polymers adsorbed on a particular substrate. In constant height mode would be possible to result in a large radius of the carrier, formation of a major forces between the tip and the surface and thereby to infringe the organic adsorbates. Constant height mode can also cause damage to the surface with fairly rugged topography. [3,4] The rugged relief is also present in the composite materials reinforced with textiles from worn tires. Characteristics of composite material depend on various factors. Among the most important belong, characteristics of the fibers and the polymer matrix, fiber content in the composite, fiber length and orientation, the nature of interfacial interface and method for composite manufacturing. By unidirectional oriented fibers in the composite materials is obtained by far the largest improvement of properties. In terms of choice of fibers is important that the the mechanical properties of the fibers (σ f , E f) were significantly higher than the properties of the matrix (σm, Em). In the selection of the polymer matrix is important that the extension of the matrix (σm) was greater than the extension of the fibers (σf). Overall, it must therefore apply:

$$\sigma_f >> \sigma_m \quad (1)$$

$$E_f >> E_m \quad (2)$$

$$\sigma_f < \sigma_m \quad (3)$$

Impact of fibre content is as important as choosing the fibres themselves. [2-5] For the specific type of the composite is therefore essential to set the lowest content of the fibres, referred to as ($n_{f\ krit}$), that will achieve improvement of mechanical properties. It is therefore very important that $n_f > n_{krit}$. The actual orientation of the fibres in the composite is critical not only for the final application properties, but in terms of the choice of appropriate processing technology. The relevant product ion technology is the technologically intensive; the higher criteria are placed on the orientation of the fibres. Therefore, it is necessary to specify in which direction in relation to the orientation of the fibres will be products stressed when designing the resulting application properties of the composites. Longitudinal strength direction is termed an index X_1 and S_1, and depends in particular on the fibre content in the composite. Typically, S_1 is directly proportional to the fibre content in the composite. About the transverse strength (S_2, S_Y), determines the strength of the polymer matrix and the interfacial strength of the interface, while the strength of the fibre does not have effect on this quantity virtually. Shear strength depends primarily on the angle of force. [8,9] At unidirectional and face oriented fibers is a hallmark anisotropy of properties (anisotropy, i.e. the dependence of the physical properties of the substances from the direction of force). It is known that the strength of the composite in the direction of fiber orientation, i.e., longitudinal is substantially higher than in the direction perpendicular to the axis of the fiber in composites with face orientated fibers longitudinal strength is the same as transverse. In the case of the face oriented fibers (laminates) are properties of the composites isotropic in each individual level, but in the direction perpendicular to the axis of the fibers are substantially lower. Isotropic properties are achieved in composites with spatially orientated fibers. In the selection of matrix for the application, it is necessary to consider application and production properties of the polymer. In the case of a thermosetting matrix in terms of application properties of composites has important role: strength, elongation in tension, toughness, heat resistance, UV resistance, chemical resistance, moisture absorption, flammability and dielectric properties. In terms of the selection of manufacturing technology is mainly viscosity, the wettability of fibers, shelf life, rate of curing, the volatile products, by-products of the curing. A deeper study of the issue polymers is dedicated to scientific literature, for example G.H. Michler in publication “Electron Microscopy of Polymers” [13] Nielsen in "Composite materials, properties as influenced by phase geometry" [16] Thomas G. Mezger in "The Reologhy Handbook" [11].

2 Experimental Procedure

2.1 Atomic force microscope using by scanning of surface topography

AFM is used sharp point to measure surface of the sample, several tens of microns long, which is formed at the free end of the spring bar. The spikes are mainly made of silicon or Si3N4, the radius of the tip of such a spike is 2 ÷ 20 nm. The bar captures interaction force between the tip and surface of the sample [1,3,7]. Piezoelectric crystal with a bar that is fastened thereon, moves (rasterized) in the x and y plane (parallel to the surface of the sample). A sharp point follows the irregularities at the surface of the sample and according to the relief and bends in the z direction (perpendicular to the surface of the sample). Bar bending detection is based on optical principles. The beam of a laser diode is incident on the tip and it is reflected on the photodetector. It is divided in two or four sensitive parts. Before the actual scanning system is mechanically adjusted so that the beam power turned out same to all parts of the photodetector. [6,8] When measuring the deflection of the beam reflected by moving traces of the reflected beam, so energy falling on each of the detector is no longer the same and from their ratio it is possible to determine the deflection of the beam. Quadrant detector allows detecting the movement of the light spot in the horizontal direction, thus deflection of the beam (Lateral Force Microscopy - LFM). [5,9,10] The detector is able to register a change of position of the beam from one nanometer. The ratio of the beam between the bar and detector to the length of the bar causes mechanical amplification. This results in that the system can detect the vertical movement of the bar below 0.1 nm. [12,13,14] Bar deflection is recording during measurement and with the help of other program processing is generated resulting surface topography. The tip that is very close to the surface particular act short-range repulsive forces of electrostatic origin (presented as overlap of electron orbitals of the atoms or molecules of the surface of the tip and the sample) and the long-range, the attractive van der Waals forces (that is, dipole-dipole force interaction). The exact quantum-mechanical calculations of these forces for the system of atoms of the tip and the surface is quite complex. The influence of both forces can be modeled, for example as empirical Lennard-Jones potential. The interaction of tip - sample for the potential we can use relationship:

$$V(r) = 4\varepsilon\left[\left(\frac{\sigma}{r}\right)^{12} - \left(\frac{\sigma}{r}\right)^{6}\right] \tag{4}$$

Where r is the distance of the tip from the the sample, σ, ε are specific Lennard-Jones parameters. Proportional member (1 / r) 12 describes the field of repulsive force, the member proportional to (1 / r) 6 area attractive force [3].

2.2 Material characterization

Mixing of polymers produces a mixture of two or more polymers, with non-polymeric additives (e.g. fillers, plasticizers etc.). For preparation of composite was used continuous mixing of various mixtures. For continuous mixing of polymers in the modern production lines is used twin-screw mixing equipment. Thermoplastics are almost exclusively prepared on twin-screw devices less demanding mixing on single-screw devices with the inclusion of kneading and mixing elements. [3,4,8] Twin-screw extruders have two screws in the housing that can be positioned to their threads overlap one another or they are positioned without mutual overlap. For preparation of the compositions are used only twin-screw devices with mutual overlaps of threads. The direction of rotation of the screw can be affirmative or controversial. Modern twin-screw devices are constructed segmental, i.e. the elements (threads) can be exchanged on the core or rearrange [2,4,6]. In our case we used for the homogenizing the mixture twin-screw auger and the specific device for kneading polymers Plastic-Corder W 350 E, Brabender (Germany). Kneading itself was preceded by determining the weight of the sample required for the first kneading and later for pressing. In our case was total weight of the mixture 200 g. The first step was to heat the machine to the required operating temperature. The heating took 10 minutes, after the temperature reached 100 ° C was added into the feeding tube polyvinyl butyral (PVB), homogenization time was 20 minutes, after the completion of the homogenization of the first component are subsequently added to mixture the amount of fabrics, we need to used weight in order to out the whole required amount the into the kneading machine. Homogenization, already with fabric, it lasted 30 minutes. The whole kneading process was set to a time of 60 min.

For further homogenizing of the ingredients with a higher proportion of the fabrics, , the period was shortened by approximately 10 minutes, since the machine has already been heated up to working temperature. Homogenization of pure thermoplastic PVB (without addition of the fabric) was set for time of 30 minutes [4,7].

2.3 Specimen Preparation

Feed material is a composite material made from used tires, in particular from the textile component as the filler and the matrix - polyvinyl butyral (PVB), which was obtained by after the recycling of car windshields. The test sample was a material containing 10% fabrics and 90% PVB.

Table 1 Measurement characteristics [4]

Equipment	Solver Scanning Probe Microscope (Russia)
Scanning bar	NSG01 Golden Silicon Probe (NT-MDT)
Length of the bar	130 µm
Width of the bar	35 µm
Thickness of the bar	1,7-2,3 µm
Scanning frequency	115-190 kHz
Scanning strength	2,5-10 N/m
Scanning mode	semi contact, phase contact

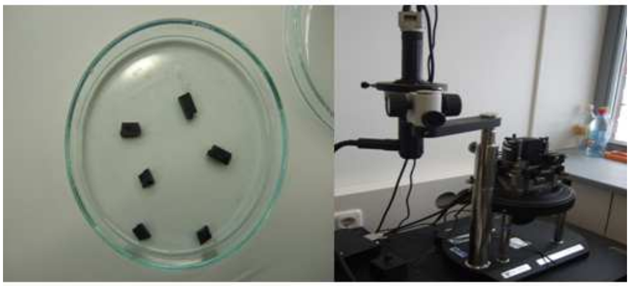

Figure 1 Specimen preparation for AFM testing (left side) and AFM equipment (right side) [4]

3 Results and discussion

Utilization of Atomic force microscopy has just an irreplaceable position for studying the surface topography of various materials and also thermoplastics. Therefore, it provides important information about the surface of the display material and its properties that is necessary to know for the further examination as to the utilization of the material [3,4]. The following figure shows the color scheme that displays the values mean in how "deep" the sample was situated at the time of measurement. More specifically, the movement of the bar across the surface of the sample, in view of the fact that for measuring is required a minimum amount of sample about 20 mg. The sample was adjusted for the preparation with a knife, for comparison was prepared also a sample by fracture after treatment with liquid nitrogen [15]. From images is visible inequality of area, textile fibers course, the presence of rubber particles and also cavities incurred after dropping the rubber particles after the breaking of the sample. The 3D view also helps to imagine how is in materials arranged fiber of fabric. Figure shows the display 50x50 microns of the height. The analyses were performed with the sample frozen with liquid nitrogen. [2,3] The figure shown cavity (black), according color scheme, we can see that the darker the color is, the deeper penetrated the scanning bar in the sample. [4,5] Figure shows a 3D view of the sample under consideration. It is seen that fracture by liquid nitrogen was broken rubber compound, giving us interesting view of the sample - cavity (crater) in view examined material. Third in the series is displayed using the form of a phase, where there is surface tracking in the direction of fibers.

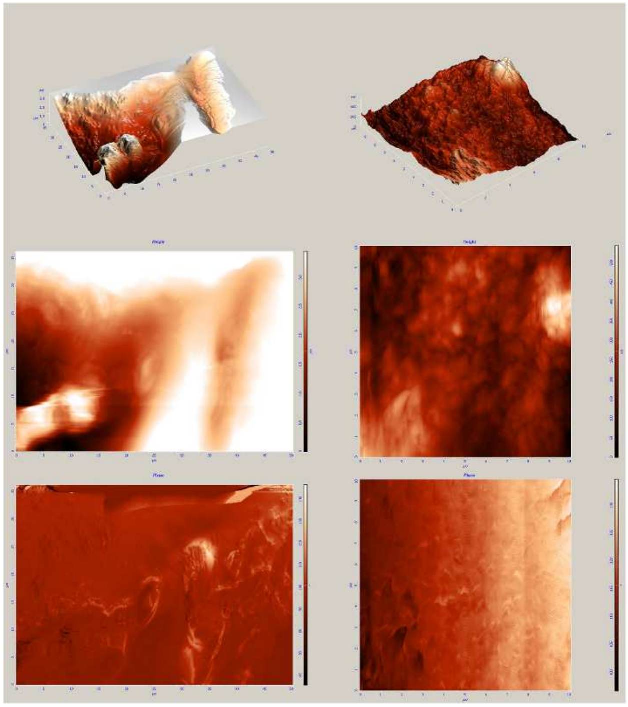

Figure 2 Results from AFM analysis (left side: 90%PVB and 10% Fabric, 50x35, 3d height, height and phase contrast. Right side: 90% PVB and 10% Fabric, 10x10, 3d height, height and phase contrast)

4 Conclusion and future direction of research

Atomic force microscopy application is primarily used for research probing of samples. Currently, has an irreplaceable position in the study of surface topography of various materials such as composite materials. We can say, therefore, it provides important information about the surface of the display material and its properties that is necessary to know for the further examination, in particular for of utilization the material. Displayed by using atomic force we can get a clearer idea of the investigated materials and other behavior in a various mechanical tests.

- For these types of composites by AFM was seen that the structure is distorted in places rubber compound, which was located in the fabrics.
- This disruption could cause defects for example in tensile test, when the material does not meet predetermined load.

References

[1] AVERY, J.: *Injection Moulding Alternatives*, Carl Hanser Verlag., 1998.
[2] ŠOLTÝS J, MARTAUS, J.: *Výskum povrchov metódou atómovej silovej mikroskopie*, Elektrotechnický ústav, SAV, Bratislava, 2009. (Original in Slovak)
[3] KNAPČÍKOVÁ, L: *Štúdium povrchu termoplastov pomocou mikroskopie atomárnych síl*, Plastics Production, 2011. (Original in Slovak)
[4] KNAPČÍKOVÁ,L: *Optimalizácia technologických procesov pri zhodnocovaní plastov*, Dissertation, Technical University of Kosice, 2011. (Original in Slovak)
[5] KLUVÁNEK P.: *Dlhá cesta k atómovému rozlíšeniu*, http://www.sav.sk/. Accessed 25 November. 2015. (Original in Slovak)
[6] HUYNH H, RAGHAVAN D, FERRARIS CF.: *Rubber particles from recycled tires in cementitious composite materials*, In: National institute of standards and technology, Washington, 1994.
[7] LIPTAKOVA, T., ALEXY, P. et al. *Polymérne technické materiály*, Vysokoškolská učebnica, Bratislava, 2009. (Original in Slovak)
[8] MADEJOVA, J.: *Infračervená spektroskopia*, SAV, Ústav anorganicke.j chémie, Bratislava, 2009. (Original in Slovak)
[9] MENGES G.: *Werkstoffkunde der Kunststoffe*, 4. Auflage, Carl Hanser Verlag, München,1998. (Original in German)
[10] MEZGER G.T.: *The Rheology Handbook*, 2nd ed.,Vincentz Network, Hannover, 2009. (Original in German)
[11] MEZGER, G.: *Das Rheologie Handbuch*, Vincentz Verlag, Hannover, 2000. (Original in German)
[12] MICHAELI, K.: *Training in Injection Moulding*, Carl Hanser Verlag, Berlin, 2001.
[13] MICHLER, G.H: *Electron Microscopy of Polymers*, Springer Verlag Berlin Heidelberg, 2008.
[14] MORTENSEN, A.: *Concise encyclopedia of composite materials*, Elsevier, London, 2007.
[15] MUELLER, M.: Hybrid composite materials on basis of reactoplastic matrix reinforced with textile fibres from process of tyres recyclation, *Agronomy Research*, Vol.13, No.3, pp. 700-708, 2015.
[16] NIELSEN L. F.: *Composite materials, properties as influenced by phase geometry*, Springer-Verlag, Berlin, 2002.

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