



Budapest University of Technology and Economics

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Micromechanical deformation processes in polymer composites

Ph.D. Thesis

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1. Introduction

In the past decades plastics became unnoticed an essential part of our everyday life. The number of application areas is increasing continuously and today it is almost impossible to design new products without plastic components. With the increasing number of applications the requirements for the raw materials increased as well. The aerospace industry is often mentioned as the most specific and demanding field, but similar, if not stricter conditions must be fulfilled by human implants, for example hip replacements. Because the number of industrially produced polymers is limited, progress would be impossible without the modification of polymers. Polymers can be modified in many ways. Copolymerization, grafting, or most polymer analogous reactions are usually complicated and economically not feasible for industrial applications. Another possibility of modification is offered by the addition of a second component to the matrix polymer. Depending on the type of modifier the heterogeneous polymers can be classified in several ways. Arbitrarily, we divide them into three categories: polymer blends¹, particulate filled polymers² and fiber reinforced composites³. Such modified polymers are typically used as structural materials.

In the Department of Applied Polymer Chemistry and Physics at the Institute of Materials and Environmental Chemistry, HAS and the associated Laboratory of Plastics and Rubber Technology (LPRT) at the Budapest University of Technology and Economics heterogeneous polymer systems are investigated and developed for a long time. The group started with the study of particulate filled polymers and developed models describing various phenomena in

¹ Paul, D. R., Bucknall, C. B.: *Polymer Blends*, Wiley, New York, 2000.

² Rother, R. N.: *Particulate-Filled Polymer Composites*. Rapra Technology, Shrewsbury, 2003.

³ Pilato, L. A., Michno, M. J.: *Advanced Composite Materials*, Springer-Verlag, Heidelberg, 1994.

these materials. Several publications and industrial cooperations show the success of the group in this field. The investigated materials and problems changed considerably during the years. The focus of attention shifted towards multicomponent materials, carbon fiber reinforced thermosets and thermoplastics, and to layered silicate nanocomposites. Interfacial interactions are one of the most important factors determining the ultimate properties of composites. This Thesis is a further step in the line of research done in this area focusing on the factors determining deformation processes and on their effect on the final properties of composites.

The introduction of fillers or reinforcements into a polymer matrix results in a heterogeneous system. Since most of them are structural materials, during any application their load bearing capacity is crucial. Under the effect of external load heterogeneities induce stress concentration, the magnitude of which depends on the geometry of the inclusions, on the elastic properties of the components and on interfacial adhesion^{4,5}. Heterogeneous stress distribution and local stress maximums initiate local micromechanical deformations, which determine the deformation and failure behavior, as well as the overall performance of the composites.

Nanocomposites are claimed to be the structural material of the future. Polyamide nanocomposites proved to be the most successful among all polymer/layered silicate nanocomposites. Modification of the surface of the clay with an ω -amino acid assists exfoliation, on the one hand, and creates a strong bond with the matrix, on the other⁶. Such composites occasionally show largely improved properties compared to the neat matrix, including increased

⁴ J. N. Goodier, *J. Appl. Mech.*, **55**, 39 (1933).

⁵ T. Kowalewski, A. Galeski and M. Kryszeński, in *Polymer Blends. Processing, Morphology and Properties*, Plenum, New York, 1984.

stiffness and strength⁷. The excellent properties of PA nanocomposites are explained with the complete or almost complete exfoliation of the silicate supported by X-ray diffraction measurements and transmission electron microscopy⁸. In spite of the general belief in extensive exfoliation and improved properties, widely differing modulus and strength values were reported in the literature even for composites composed of very similar ingredients^{8,9}. The dissimilar properties indicate differences either in structure and/or in interfacial interaction since mainly these factors determine composite properties.

Polymers reinforced with natural fibers are used in large quantities; the production of such materials is a mature technology now¹⁰. Intensive research continues in the field with the goal of improving composite properties, and to produce better and cheaper materials¹¹. Many attempts have been made to improve the properties of natural fiber reinforced composites. Several reports are available on the effect of wood type¹², but it is still unclear if the use of soft or hard wood results in composites with better properties. Somewhat clearer picture is obtained on the effect of fiber characteristics on properties¹³. The extent of reinforcement increases with anisotropy, but the effect of coupling becomes less pronounced as the aspect ratio of the filler increases. While the effect of wood characteristics and interfacial adhesion on composite properties has been studied extensively¹⁴, much less attention has been paid to the influence of matrix characteristics.

⁶ Kojima, Y., Usuki, A., Kawasumi, M., et. al.: *J Mater Res* **8**, 1185-1189 (1993)

⁷ Reichert, P., Kressler, J., Thomann, R., et. al.: *Acta Polymerica* **49**, 116-123 (1998)

⁸ Fornes, T. D., Yoon, P. J., Hunter, D. L., et al.: *Polymer* **43**, 5915-5933 (2002)

⁹ Reichert, P., Nitz, H., Klinke, S., Brandsch, R., et. Al.: *Macromol Mater Eng* **275**, 8-17 (2000)

¹⁰ Markarian, J.: *Plast Additives Comp* **4**, 18-21 (2002)

¹¹ Bledzki, A. K., Letman, M., Viksne, A., Rence, L.: *Composites* **A36**, 789-797 (2005)

¹² Neagu, R. C., Gamstedt, E. K., Berthold, F.: *J Compos Mater* **40**, 663-699 (2000)

¹³ Coutinho, F. M. B., Costa, T. H. S., Suarez, J. C. M., Melo, D. P.: *Polym Test* **19**, 625-633 (2000)

¹⁴ Zhang, C., Li, K., Simonsen, J.: *Polym Eng Sci* **46**, 108-113 (2006)

As indicated above, a large number of papers have been published on the mechanical properties of all kind of polymer composites. However, very few of these concentrate on micromechanical deformation processes although the dominating process will determine the ultimate properties of the composites. In this Thesis we focus our attention onto these processes in order to obtain more information about the factors determining them, as well as to develop guidelines for the improvement of mechanical properties and to achieve better reinforcement. Our laboratory has been working on various heterogeneous polymer systems for a long time and the expertise developed helps considerably the identification and interpretation of these processes. The main motive of the Thesis is the study of micromechanical deformation processes and the factors influencing them.

2. Materials and methods

Composites were prepared from PP and various fillers in a wide composition range. The components were homogenized in an internal mixer then compression molded into plates. Mechanical properties of the composites were characterized by tensile testing. The adhesion of the filler to the matrix was modified in different ways depending on the investigated system. The strength of interfacial adhesion was estimated quantitatively with model calculations. Micromechanical deformation processes were followed by acoustic emission (AE) and volume strain (VOLS) measurements. The size distribution of the filler was determined by light scattering, while filler characteristics were observed with the help of scanning electron microscopy (SEM). The failure mechanism was also supported by SEM.

3. Results

Particle filled polymers were studied in the first stage of the research. PP/PMMA model composites containing particles with narrow particle size distribution were compared to composites made from PP and commercial CaCO₃ filler. Measurements of acoustic emission signals during the elongation of PP/PMMA composites allowed us to assign the debonding process, including its initiation, unambiguously to a well defined range of the stress vs. strain curve. The number and intensity of the signals detected in the matrix and the composite, respectively, differed considerably, which made possible the separation of the various micromechanical deformation processes occurring in them. At small particle content debonding occurs at relatively low stresses, which differ considerably from yield stress. Considerable plastic deformation of the matrix starts at the yield point. At larger filler content debonding and shear yielding occur simultaneously. Micromechanical deformation processes cannot be separated as clearly in composites prepared from the commercial CaCO₃ filler with a broad particle size distribution. The debonding of particles with different sizes occurs in a wide deformation range because of the particle size dependence of debonding stress. The analysis of characteristic values derived from acoustic emission experiments proved that the interacting stress fields of neighboring particles influence deformation and

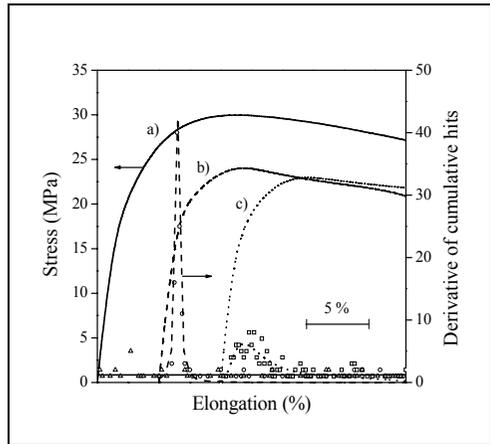


Fig.1 Determination of debonding stress for PP (a), PP/PMMA (b), and PP/CaCO₃ (c) composites. Filler content: 10 vol%.

At small particle content debonding occurs at relatively low stresses, which differ considerably from yield stress. Considerable plastic deformation of the matrix starts at the yield point. At larger filler content debonding and shear yielding occur simultaneously. Micromechanical deformation processes cannot be separated as clearly in composites prepared from the commercial CaCO₃ filler with a broad particle size distribution. The debonding of particles with different sizes occurs in a wide deformation range because of the particle size dependence of debonding stress. The analysis of characteristic values derived from acoustic emission experiments proved that the interacting stress fields of neighboring particles influence deformation and

that even large particles may aggregate or at least associate at large filler content.

Nanocomposites were prepared from polyamide and silicates with and without organophilization. The goal of the study was to investigate the effect of filler content and adhesion on the structure and properties of the composites. With the help of acoustic emission and volume strain measurements supple-

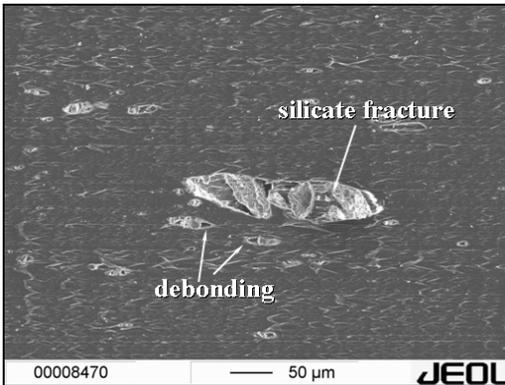


Fig. 2 Mechanism of micromechanical deformations in PA nanocomposites

mented by microscopy we tried to obtain as much information about the mechanism of deformation as possible. The results showed that the structure of the composites is more complicated than usually stated. They contain various structural entities; besides individual silicate

platelets, tactoids with different degree of intercalation and larger particles can be also found in them. The matrix polymer and the composites deform according to different mechanisms. Sound is emitted by cavitation in the former, while sound emitting processes are related to larger structural entities, i.e. to tactoids and non-exfoliated particles in the composites. Acoustic events are generated mainly by the fracture of the particles. Matrix/silicate adhesion seems to be strong, debonding rarely takes place, and volume increase is initiated primarily by particle failure. The type and amount of the surfactant used for organophilization plays an important role in the determination of deformation processes and properties, since it influences both matrix/filler interaction and the internal adhesion of silicate particles.

Experiments carried out on PP/wood composites proved that several processes take place during the deformation of failure of these composites. The matrix deforms mainly by shear yielding, debonding and fiber pull out occurs when interfacial adhesion is poor, and fiber fracture dominates in the presence of coupling agent. We concluded from these results that further improvement of composite strength is possible only by the increase of the inherent strength of wood particles. One way to do that was assumed to be the decrease of their size. The study of the deformation and failure of PP/wood composites contain-

ing wood particles of different sizes proved that micromechanical deformations change drastically with decreasing particle size. Less debonding, fiber pull out and fiber fracture occur in composites containing small particles. The apparently slight influence of particle size on composite strength results from the smaller aspect ratio of the small particles, which indicates that orientation and

orientation distribution must have a strong effect on reinforcement. Further improvement in composite strength is possible only through the optimization of particle size, aspect ratio and the inherent strength of wood.

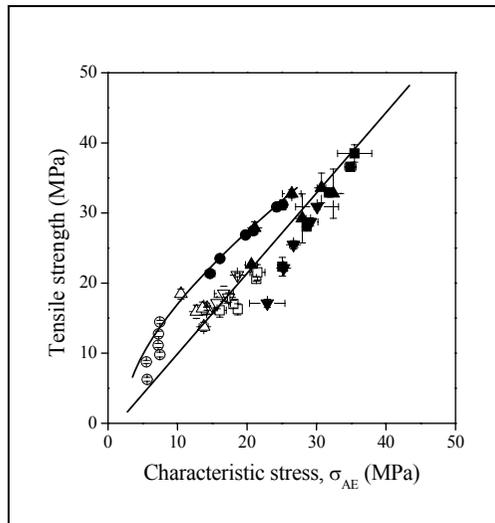


Fig.3 Correlation between the tensile strength of PP/wood composites and the characteristic stress (σ_{AE}) determined from acoustic emission measurements

A more detailed study on the factors determining the reinforcing efficiency was carried out subsequently. PP/lignocellulosic composites were pre-

pared from four different fillers. The results proved that the properties of the composites depend strongly on interfacial adhesion and on the particle characteristics of the wood. Coupling with functionalized polymer is necessary for the preparation of composites with acceptable properties if the size of the particles is large and their aspect ratio is small. The effect of adhesion is smaller for particles with large aspect ratio. Large aspect ratio and small fiber diameter result in better reinforcement. The inherent properties of the reinforcement may limit the improvement of composite strength. A failure map was presented to show the effect of particle characteristics and adhesion on the dominating deformation process.

While the effect of wood characteristics and interfacial adhesion on composite properties were studied extensively, much less attention has been paid to the influence of matrix characteristics. As a consequence, the goal of

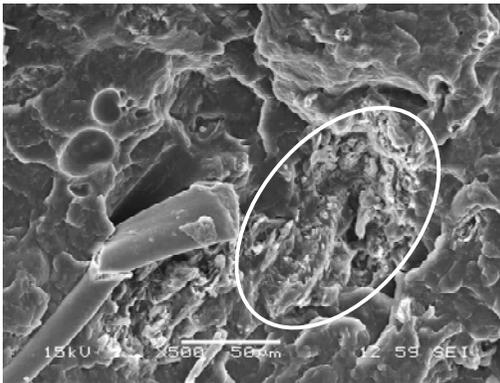


Fig. 4 *Good adhesion and high yield stress of the matrix results in the fracture of filler*

another subproject was to investigate the influence of matrix characteristics on deformation and failure in PP/natural fiber composites. The experiments carried out with three different PP matrices, a homopolymer, a random and a heterophase copolymer, proved that the dominating micromechanical

deformation process may change with matrix properties. Yield stress determined from the stress vs. strain traces may cover widely differing processes. Debonding is the dominating process when the adhesion of the components is poor, while matrix yielding and/or filler fracture dominate when adhesion is

improved by the introduction of a functionalized polymer. The dominating deformation mechanism is determined by component properties (matrix characteristics, particle strength and geometry) and adhesion. Interfacial adhesion, matrix yield stress and the inherent strength of the reinforcement can be limiting factors in the improvement of composite strength. The properties of polymer composites reinforced with lignocellulosic fibers are determined by micromechanical deformation processes, but they are independent of the mechanism of these processes.

Various models and methods were introduced in this thesis to characterize interfacial interaction and the effect of reinforcement. In the final part of the study an approach was proposed for the quantitative determination of adhesion strength in composites, in which adhesion is created by other mechanisms than secondary interactions. The approach is based upon a model, which gives debonding stress as a function of interfacial adhesion. Debonding stress is determined by acoustic emission experiments. The mechanism of deformation was checked by SEM experiments and the approach was verified on composites with known interfacial adhesion. The results obtained showed that the use of functionalized polymer in PP/CaCO₃ composites resulted in adhesion strength one order of magnitude larger than without the coupling agent. The application of various surface modification techniques in PP/glass bead composites yielded different adhesion values covering a range of about one order of magnitude. Consequently the quantitative determination of interfacial adhesion makes possible the design and optimization of most surface modification techniques in particulate filled and short fiber reinforced composites.

4. New scientific results

1. We proved by the analysis of micromechanical deformation processes in PP composites containing model and commercial fillers that debonding stress can be determined with the help of acoustic emission measurements. We pointed out that debonding occurs in a deformation range depending on the size distribution of the filler. The interacting stress fields of neighboring particles influence deformation, and even large particles may aggregate or associate at large filler content. [1]
2. We showed by the analysis of PA6/layered silicate composites that they contain various structural entities; besides individual silicate platelets, tactoids with different degree of intercalation and larger particles can be also found in them. We proved by acoustic emission and volume strain measurements that deformation processes are related to larger structural entities, i.e. to tactoids and non-exfoliated particles in the composites, and the dominant deformation process is mainly the fracture of particles. [2]
3. We analyzed the micromechanical processes taking place during the deformation of PP/wood composites containing wood particles of different sizes and showed that micromechanical deformations change drastically with decreasing particle size. Less debonding and fiber fracture occurred in composites containing the filler with smaller particle size, but thus influenced composite strength only slightly. We predicted that further improvement in composite strength is possible only through the optimization of particle size, aspect ratio and the inherent strength of wood. [3]
4. We predicted by the study of deformation processes in different PP/lignocellulosic composites that micromechanical deformation proc-

esses determine composite properties irrespectively of their mechanism. We proved this statement by the close correlation between the characteristic stress derived from acoustic emission measurements and composite strength. [4]

5. We could confirm by the study of PP/lignocellulosic composites with three different PP matrices that interfacial adhesion, matrix yield stress and the inherent strength of the reinforcement can be limiting factors in the improvement of composite strength. We proved that in the case of strong adhesion the dominating deformation process change with matrix properties, yielding occurs if the inherent strength of the filler is larger than the yield stress of the matrix, but for matrices with larger yield stress the fracture of filler particles becomes the dominant deformation process limiting the reinforcing effect. [5]
6. We developed a method for the quantitative determination of adhesion strength in composites, in which adhesion is created by other mechanisms than secondary interactions. We pointed out for the first time that the use of functionalized polymer in PP/CaCO₃ composites results in adhesion strength of one order of magnitude larger than without the coupling agent. [6]

5. Publications

5.1 The thesis is based on the following papers

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- composites: correlation of structure and properties, *Polym. Eng. Sci.* **47**, 1235-1245 (2007)
3. Renner, K., Móczó, J., Pukánszky, B: Deformation and failure of PP composites reinforced with lignocellulosic fibers: Effect of inherent strength of the particles, *Compos. Sci. Technol.* **69**, 1653-1659 (2009)
 4. Renner, K., Kenyó, Cs., Móczó, J., Pukánszky, B: Micromechanical deformation processes in PP/wood composites: particle characteristics, adhesion, mechanisms, *Composites Part A* (submitted)
 5. Renner, K., Móczó, J., Suba, P., Pukánszky, B: Micromechanical deformations in PP/Lignocellulosic filler composites: effect of matrix properties *Compos Sci Technol* (accepted)
 6. Renner, K., Móczó, J., Vörös, G., Pukánszky, B: Quantitative determination of interfacial adhesion in composites with strong bonding *Macromol Rapid Comm* (submitted)
 7. Renner, K., Móczó, J., Pukánszky, B: Micromechanical Deformations in Particulate filled Polymers: The Effect of Adhesion, *Conference proceedings of the 17th International Conference on Composite Materials*, (2009)
 8. Renner, K., Móczó, J., Pukánszky, B.: Mikromechanikai deformációs folyamatok akusztikus emissziós vizsgálata poliamid nanokompozitokban, *Műanyag Gumi*, **42**, 443-448 (2005)

5.2 Other publications

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5.3 Conference presentations

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