FIRST INTERNATIONAL CONFERENCE ON ELECTRON MICROSCOPY OF NANOSTRUCTURES



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Morphology of Poly(urethane-siloxane)/Montmorillonite Nanocomposites

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Segmented polyurethanes (SPU) are multiblock copolymers consisted of hard (HS) and soft segments (SS). HS usually form crystalline domains that are responsible for good mechanical properties, while SS represent amorphous matrix that contributes to good elastomeric properties, hydrophobicity and inertness of the SPUs [1]. Furthermore, the morphology of the SPUs is one of the main factors that determine their physical and chemical properties. Moreover, thermodynamic incompatibility between HS and SS leads to the formation of microphase separation within the SPUs [2]. Properties of SPUs mostly come from their different composition and microphase separation between segments.

Further improvement of the properties of these materials is the main goal of the research lately. This often includes the addition of different inorganic nano-fillers, such as clay minerals, into a polymer matrix [3]. Dispersion and degree of the delamination of clay particles inside polymer matrix determine which type of polyurethane nanocomposite (PUNC) is obtained. Addition of low quantities of clay nano-fillers usually leads to the improvement of the thermal, mechanical, surface and barrier properties of the SPUs [4].

In this work series of PUNCs was obtained with the *in situ* polymerization method in solvent mixture. The PUNCs were based on α, ω -dihydroxy-poly(propylene oxide)-*b*-poly(dimethylsiloxane)-*b*-poly(propylene oxide) macrodiol (PPO-PDMS-PPO) that represents SS and 4,4'-diphenylmethane diisocyanate (MDI) and 1,4-butanediol (BD) which were selected as the HS. PUNCs were synthesized with different content of HS (from 10 to 60 wt%), with the addition of organomodified montmorillonite clay (Cloisite 30B[®]) as the nano-filler, in an amount of 1 wt%. The PUNCs are marked so that the last two numbers in title indicate mass.% of HS in samples [5]. For nanostructural examinations, SEM and TEM analysis were performed on the obtained polymer films. SEM analysis was performed on JEOL JSM-6460LV instrument on the cross-section surface of the samples, at a magnification of $3k \times$. TEM analysis was performed on the JEM-1400 Plus Electron microscope, at a magnification of $60k \times$.

The morphology of the obtained PUNCs, i.e. dispersion of the clay nanoparticles inside polymer matrix, was investigated by SEM analysis. SEM micrographs show the presence of brighter parts (,,points") in the investigated samples, which originate from the addition of clay nanoparticles (Figure 1). In addition, it is observed that the clay nanoparticles are homogeneously dispersed in the polymer matrix and do not form aggregates in the tested samples. This type of morphology increased the degree of the microphase separtion, that led to the better mechanical properties of the investigated samples.

In order to determine clay morphology inside PUNCs, TEM analysis was performed (Figure 2). On the obtained TEM micrographs darker lines represent individual layers of the clay nanoparticles, which are homogeneously dispersed within the lighter polymer matrix. Moreover, clay nanoparticles had predominantly exfoliated morphology with a small number of those that had intercalated morphology in the PUNCs. This indicates mainly complete delamination of the clay nanoparticles within polymer matrix. This occurred due to the reaction between organomodifier from clay and isocyanate groups in prepolymer. Within the polymer matrix, dispersed clay nanoparticles have the length between 50 and 250 nm and the distance between layers greater than 5 nm [5].

Series of the PUNCs based on PPO-PDMS-PPO, MDI, BD and with addition of montmorillonite clay as a nano-filler was successfully synthesized. SEM and TEM analysis confirmed homogeneous dispersion and obtained mixed exfoliated/interca-lated morphology of the clay nanoparticles within the polyurethane matrix [6].

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Figure 1. SEM micrographs of cross-section surface of the selected PUNCs with 10, 30 and 60 wt.% of HS (magnification $3k \times$).



Figure 2. TEM micrographs of the selected PUNCs with 20 and 60 wt.% of HS (magnification 60k ×).