ESRF	**Morphology and properties of injection-molded polypropylene / silica composites prepared via in situ sol-gel technology**	**Experiment number**: **SC-2615**
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Report:

Incorporation of silica particles into polymer matrices is a well-known approach for the improvement of various material properties. Solution or melt mixing is the standard procedure to obtain such composites. But this method have some disadvantages that limits its application in industry – aggragation of particles, low compatibility, etc. A new route for the preparation of polymer/silica composites based on a solvent free in situ sol-gel technology by using a new silica precursor polymer - hyperbranched polyalkoxysiloxane (PAOS) has been developed (Fig. 1, top).[1,2] The iPP/silica composites were prepared with mini twin-screw extruder. After a reactive extrusion process the composite melts were transferred directly to an injection molding machine (Fig. 1, bottom). A series of composites with silica content up to 10 wt.-% were prepared. TEM study showed that the particles were homogeneously distributed in the PP matrix with the particle size much smaller than 100 nm. To study the morphology of the injection-molded films with different PAOS content space-resolved WAXS studies were perfomed. The films were sectioned (approx. 1mm thick) in the direction perpendicular to the injection

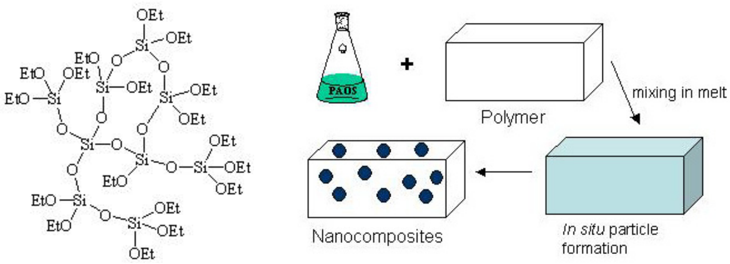

Fig.1: Chemical structure of hyperbranched polyalkoxysiloxane (**left**); Schematic representation of the composite formation (**right**).

molding. Then, the sections were scanned from top to bottom surfaces in the direction perpendicular to the flowing direction with step of 100µm. The beam size was 300µm in diameter.

The WAXS patterns of all materials measured in the center of the section show well-pronounced uniaxial "fiber" orientation in the direction of molding. 2D diffraction patterns reveal the presence of two crystalline polymorphs - α- (marked in red) and β-phases (marked in blue). In addition to the main orientation, the second fraction of crystals was found for which (110) peak is located at 15° from the meridian. Such so-called "cross-hatch" morphology of the α-phase is typical for injection-molding process. However, it was found that for pure iPP (Fig. 2, top left) the peaks are much less oriented thn for the composites (Fig. 2, bottom left). In addition, the peak (110) shows up only on the meridian, which shows the absense of "mother"crystals in the center of pure iPP film. The experimental Lorentz-corrected 1D curves were fitted with set of Gaussian functions. To calculate the relative content of each phase, the total area of most intense peaks for α- and β-modification and the amorphous halo were normalized by the total intensity. It was found that the total crystallinity of composites consequently decreases with PAOS content from 0.58 (pure iPP) to 0.39 for iPP + 20% of PAOS. The amount of β-phase shows a maximum value at 5% of PAOS - 0.06 and then slightly decreases with PAOS content. It should be mentioned that the found contents are rather low and therefore the filler particles cannot be considered as β-nucleating agents. The positional scan shows sighnificant changes of the crystal structure through the film thickness (Fig. 2, right). In contrast to the central region, the X-ray patterns of pure iPP indicate the presence of highly-oriented layer of approx. 500µm (Fig. 2, top right). In this layer both "mother" and "daughter" lamellae are found. For the composite with 7.5% of PAOS the surface layer also shows high orientation but the thickness is slightly decreased to 400µm (Fig. 2, bottom right). In conclusion, the presence of PAOS nanoparticles improves the crystal orientation in the iPP/PAOS composite and changes their morphology. The *in situ* generated silica particles play a role of lubricant for the iPP chains during the molding process. Spatially-resolved WAXS studies show the differences in the structure of injection-molded films on the surface and in the center, which is important for understanding the mechanical properties of the composites.

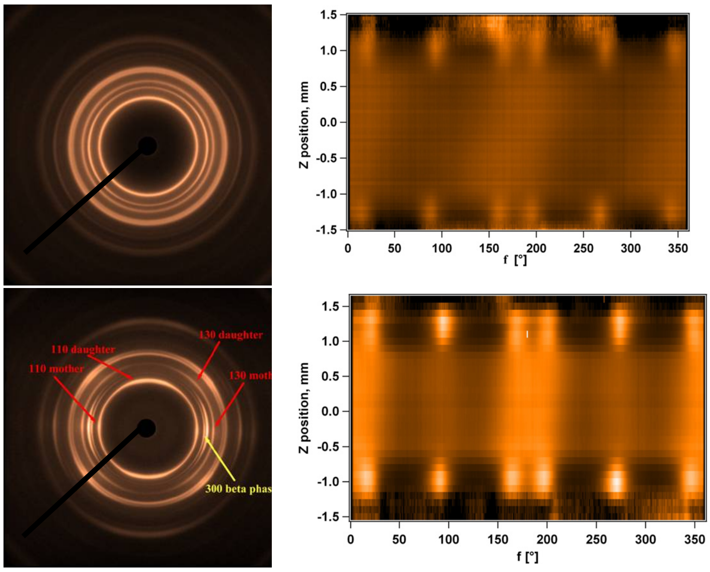

Fig.2 **Left**: 2D WAXS patterns of pure iPP (top) and iPP with 7.5% of PAOS (bottom); Molding direction is vertical. **Right:** Intensity angular distribution of (110) peak as a function of z-position for pure iPP (top) and iPP with 7.5% of PAOS (bottom). Z=0 corresponds to the center of the film.

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