PROCESSING CONTROLLED PROPERTIES OF THERMOPLASTIC-BASED NANOCOMPOSITES

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Abstract
In the present work, polypropylene based nanocomposites were compounded using a twin-screw extruder followed by injection molding. The related plaques were joined by linear vibration welding. The mechanical performances were examined by using Charpy impact testing. It was found that the incorporation of rigid particles improved the impact strength of polypropylene. The maximal impact strength was achieved at 1 vol.% TiO\(_2\) filled polypropylene. By contrast, the impact strength of the welds decreased with increased nanoparticle contents. The highest weld strength was achieved at low welding pressure without nanoparticles.

1 Introduction
In industrial series production, vibration welding is a commonly used welding method to join thermoplastics. It is well known that the processing history has a decisive influence on the structure and properties of thermoplastic materials. In particular, welding processes lead to high gradients in stress and therefore in the material morphology and properties. In the last 30 years, the basic relationships between welding process, morphology and properties are well understood for unreinforced thermoplastics; however, there is a lack of knowledge especially in the field of welding of a new class of composites such as thermoplastics reinforced with nano-sized fillers, so-called nanocomposites.

At the beginning of vibration welding, the parts to be welded are clamped into upper and lower tools, and then they are pressed together under defined pressure. After this, one of these parts is brought to vibration at adjusted frequency of 80-300 Hz over an amplitude between 0.25-2.5 mm (Figure 1). Energy is initiated by frictional forces at the contact surface, which causes the melting of the material at the interface. As a result, a molten layer appears between the welding parts. The melt squeezes out of the interface under externally applied normal pressure and a welding bead is formed outside of the contact surface. After cooling, two parts are joined together. The relative movement of the weld parts is defined as weld penetration s [1].
As a new class of materials, polymer nanocomposites (PNC) achieve more and more attentions in the last decade. The extremely high surface areas of nanoparticles can result in a great amount of interphase in the composite and thereby a strong interaction between filler and matrix is created [2, 3]. The incorporation of inorganic nanoparticles into polymer matrices has been proved to be one of the effective ways for improving the mechanical properties of the matrix [4-8]. The improvement of the properties is dependent on the type, geometry and location of particles to the direction of stress in the component and the particle/matrix interphase as well as the matrix material itself. Therefore, it is of great interest to check whether or not the high mechanical performance can be exploited in welded joints. In the present study, polypropylene based nanocomposites were used as the welding parts, in order to examine the relationship between processing parameters, morphology and mechanical properties.

2 Experimental
2.1 Materials and sample preparation
Commercial polypropylene homo-polymer (HD120MO) was provided by Borealis group. The melt flow rate and the density of this product are 8 g/10 min (230 °C/2.16 kg) and 0.908 g/cm$^3$, respectively. Hombitec RM 130 F was used as nanofiller, which was supplied by Sachtleben Chemie GmbH. This type of TiO$_2$-nanoparticles exhibits an acicular form and has a mean diameter of about 15 nm. All of the materials were used as received.

Polypropylene nanocomposites with 5 vol.% content of TiO$_2$ particles were first extruded by a Theysohn co-rotating twin-screw extruder under the screw speed of 160 rpm. The temperatures were set from 190 °C near the hopper to 210 °C at the die. The obtained PP/TiO$_2$ nanocomposites were then diluted to 0.5, 1 and 4 vol.% TiO$_2$ particle content using the same extruder under the identical conditions. Then neat PP and PP/TiO$_2$ nanocomposites were injection molded to 50x50x4 mm$^3$ sheets, which were used as welding components in the vibration welding experiments.

2.2 Welding experiment
Welding experiments were performed on a fully automatic Branson Ultraschall Lab.-Vibration welding machine M112H. The friction force, amplitude and penetration of the parts were recorded during the whole welding process. The welding pressure varied from 0.4 to 4.0 MPa, while the amplitude and frequency of vibration were kept at constant values of 0.7 mm and 240 Hz as well a constant welding time of 8 s in the whole experiments, respectively.
2.3 Impact strength
The Charpy impact strength of the specimens (50x6x4 mm$^3$) was tested according to DIN 53453 by using an impact pendulum at following conditions: room temperature, incident impact speed: 2.9 m/s, incident energy of the hammer: 4 J. Prior to the Charpy impact tests, the welding bead on the impact side was removed. At least 10 samples were tested. The samples were sawed perpendicular to the welding plane (xy-plane) from the welds.

2.4 Morphology
The morphology of the joints was analyzed on a light microscope (Zeiss AxioSkop A1. M) using microtomed thin sections of approximately 10 μm thickness cut from the yz-plane of the welded plaque (Figure 1).

3 Results and Discussion
3.1 Impact strength
The results of Charpy impact strength of unwelded (notched) and welded specimens are illustrated in Figure 2. Considering the Charpy-notched impact strengths of the parent materials, one can recognize that the incorporation of nanoparticles into PP slightly improves the impact strength of the basic matrix. The maximum of impact strength was obtained at 1 vol.% TiO$_2$ reinforcement, which is improved by approximately 7% compared to the neat matrix.

By contrast, the nanoparticles affect the Charpy impact strength of welds markedly. The addition of nanoparticles into PP reduces the impact strength at any given welding pressure, as shown in Figure 2b. The impact strength decreases with increased particle concentration up to 4 vol.%. A comparison of their impact strengths shows that the impact strength of PP/TiO$_2$ nanocomposite with 1 vol.% TiO$_2$ content drops by about 43% compared to that of neat PP at a welding pressure of 2.0 MPa. High impact strengths of PP and PP/TiO$_2$ nanocomposites were achieved, when the parts were welded at lower pressure. For example, neat polypropylene welded at 0.4 MPa exhibits an outstanding impact strength value about 27 kJ/m$^2$.

![Figure 2](image)

3.2 Microstructure of welds
The microstructure of the joints depends on the actual thermal, mechanical and flow cond-
tions in the welding seam, which all affect the crystallization behavior of the molten layer during welding and consequently the mechanical properties. Figure 3 shows, representatively, the polarization micrographs of PP and PP/TiO$_2$ nanocomposites. It is obvious that the incorporation of nanofillers into polymer matrix leads to a decrease of molten-film thickness at any given welding pressure in the studied range. The reason might be the reduction of viscosity after addition of nanoparticle. Similar to addition of nanoparticles, an increase in welding pressure also causes a decrease in molten-film thickness.

Figure 3. Light microscopic photographs of various welds: (a) neat PP welded at 0.4 MPa, (b) PP with 0.5 vol.% TiO$_2$ welded at 0.4 MPa, (c) neat PP welded at 4.0 MPa and (d) PP with 0.5 vol.% TiO$_2$ welded at 4.0 MPa.

Figure 4a depicts the dependence of molten-film thickness on the welding pressure and filler content, high welding pressure as well filler content lead to a decrease in molten-film thickness. As a consequence, lower impact strength is obtained at small molten-film thickness for all the studied materials (Figure 4b). The possible reason may be that small weld area restricts the dissipation of stress imposed during the mechanical testing due to different structure compared to bulk materials, and therefore reduces the load bearing capacity of the joints.

Figure 4. (a) Dependence of molten-film thickness on welding pressure and filler content and (b) correlation between impact strength and molten-film thickness.
4 Conclusions
In this work, polypropylene based nanocomposites were prepared by melt compounding, and then injection molded to flat plates, finally joined by vibration welding. The mechanical properties and morphologies of the welds were examined. Following conclusions can be drawn:

- The incorporation of Nano-TiO$_2$ particles into polypropylene matrix slightly increases the impact strength of bulk material
- The impact strength of the welds significantly decreases with addition of nanofillers
- The incorporation of nanoparticle into polypropylene reduces the molten-film thickness at given welding pressure
- The reason for the decrease of impact strength may be the reduction of molten-film thickness and therefore higher stress concentrations due to a different morphology in this area

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