Special reprint from Kunststoffe international 4/2008

HANSER

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Performance Enhancements for Highly Filled Compounds

Dispersing Additives

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Special reprint

Dispersing Additives. Compounds with high filler loadings are difficult to process. This results in sub-optimal dispersion of the fillers in the polymer matrix, which also has a negative influence on the mechanical properties. A new additive concept now allows processing and properties to be improved.

Through the addition of a newly developed dispersing additive the throughput of, e.g., a 60 % calcium carbonate filled PP compound is increased (left: 0 %, right: 1 % additive)

Performance Enhancements for Highly Filled Compounds

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Fillers are used in polymers in many ways to improve properties or reduce costs. A wide range of various different fillers are available to the processor. Nearly all the matrix properties (e.g. mechanical properties, flammability and thermal behavior) can be influenced in a targeted manner by the use of suitable fillers. The challenge for the compounder is that the resulting compounds, particularly those with high filler levels, are difficult to process and that the overall mechanical properties are negatively affected. The cause of this often lies in a sub-optimal dispersion of the fillers or in the use of non-optimized processing aids or other additives.

A newly developed additive concept from BYK-Chemie GmbH, Wesel, Germany, now allows compounders to produce and process highly filled compounds with excellent mechanical properties reliably and cost effectively. In this way it is possible to achieve additional added value for the end product.

Interfacial Bonding Improved

Especially for technically demanding highly filled compounds processors are currently forced to use additives, for example

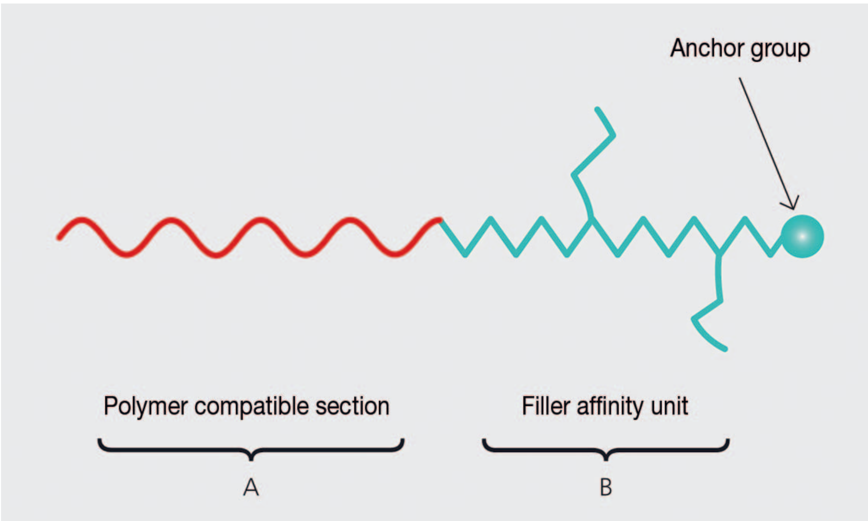

Fig. 1. Schematic representation of the BYK-P 4101 block copolymer (red: polymer compatible section, blue: filler affinity unit with anchor group)

Translated from Kunststoffe 4/2008, pp. 66–69

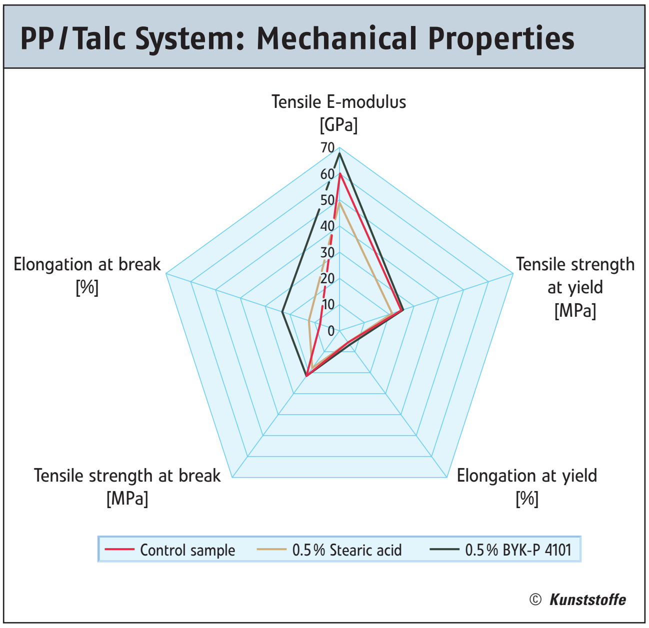

Fig. 3. Mechanical properties of the PP compound PPH 9060 with 40 % talc (output: 27.0 kg/h; temperature profile of the dies: 200 to 220°C)

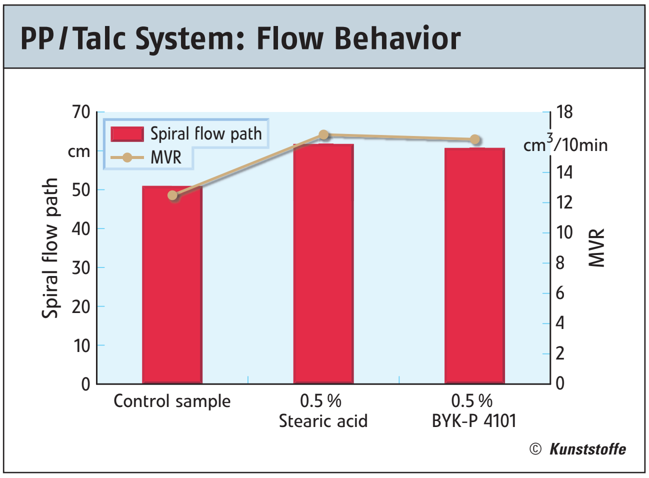

Fig. 4. Flow behavior of the PP compound PPH 9060 with 40 % talc (output: 27.0 kg/h; temperature profile of the dies: 200 to 220°C)

grafted polyolefins or silanes, in order to achieve a good adhesion at the filler/matrix interface. However, depending on the filler used it is not always possible to achieve a satisfactory result. In addition it is often necessary to add additional processing aids (e.g. waxes or stearates) in order to make processing easier. These additives however often have a negative effect on the mechanical properties.

The newly developed additive system BYK-P 4101 has been designed to simplify this complicated processing scenario. In highly filled compounds containing BYK-P 4101 the processing parameters such as viscosity, torque demand and output are improved. The mechanical characteristics, i.e. tensile strength and elastic modulus (tensile E-Modulus), are also increased.

As a block copolymer BYK-P 4101 displays a so called AB structure. The schematic in Figure 1 shows on one side a highly active wetting anchoring group (blue) which is attracted to the filler. On the other side there is a block segment (red) that is compatible with a wide range of polymers. Through its bonding mechanism BYK-P 4101 can reduce the interfacial tension at the polymer/filler boundary. Its molecular structure contributes to an improved bonding between filler particles and polymer.

Through the optimal combination of molecular weight and functionality the phase coupling of the filler to the polymer matrix is strengthened. This can be explained using the theory that in respect of the mechanical properties there is a critical entanglement molecular weight. The „critical“ entanglement molecular weight varies according to the polymer, e.g. 7,000 g/mol for polypropylene (PP), 4,000 g/mol for polyethylene (PE) and 4,000 to 5,000 g/mol for polyamide (PA) [10, 11]. This effect is supported by the affinity of the acid anchoring group for the most common filler types.

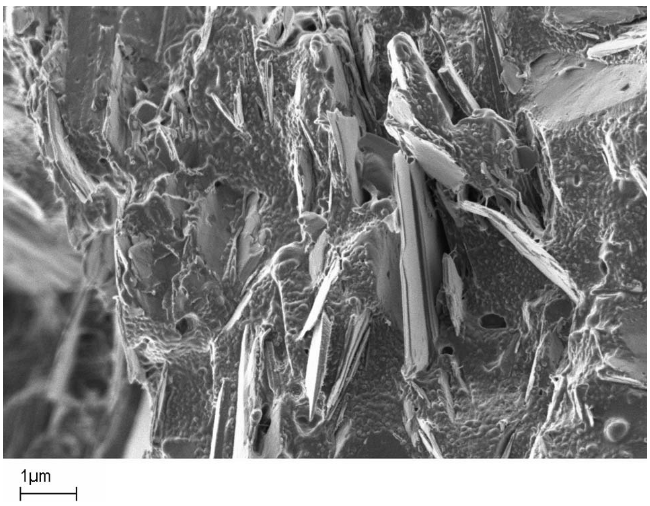

Fig. 2 left. SEM picture of the PP/talc system without additive (control): In the control sample the filler particles have been torn out of the polymer matrix due to a lack of bonding (source: Eckart)

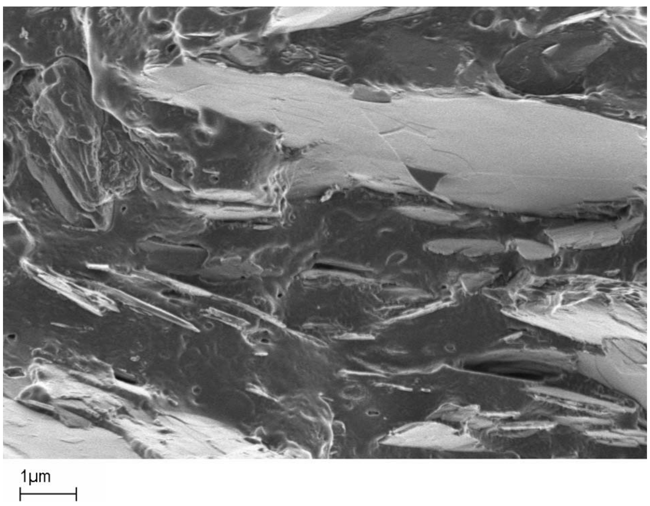

Fig. 2 right. SEM picture of the PP/talc system with additive: In the samples with additive the filler particles are so well bonded that they internally fracture (source: Eckart)

Its chemical structure allows the product to function as a classic dispersing agent, i.e. when shear processes during compounding break up agglomerates the new filler surface is immediately coated with BYK-P 4101. The surface energy of the filler is matched to that of the polymer and the viscosity is thus reduced. Through this process reagglomeration is also prevented and the filler particles are stabilized. This results in a more homogeneous polymer compound than is possible with classic processing aids.

In order to demonstrate the good filler incorporation, fracture surfaces of highly filled polyolefin compounds were investigated using a scanning electron microscope (SEM). As examples of this the fracture surfaces of a talc filled PP are shown in Figure 2. In the control sample talc platelets have been torn out of the polymer matrix due to the lack of adhesion (Fig. 2 left). The sample with additive on the right hand side of Figure 2 looks very different. Here the filler particles are so well bonded that they have internally fractured. In this case the additive performs a classic coupling agent function.

PP / $CaCO_3$ System: Mechanical Properties

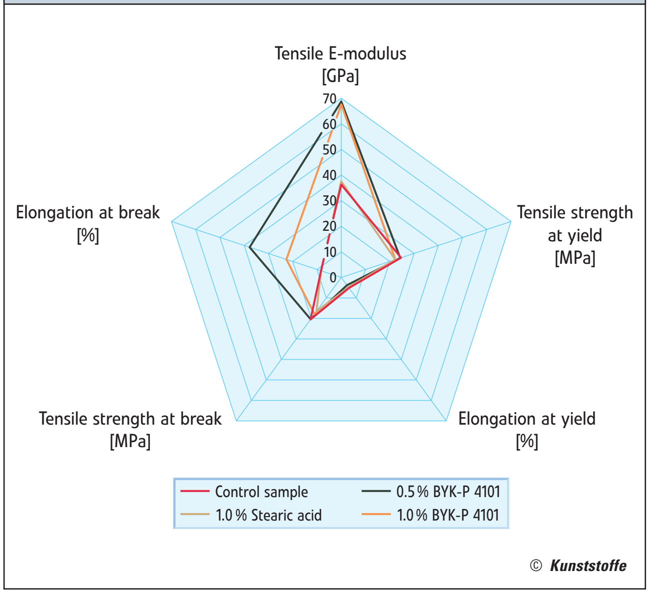

Fig. 5. Mechanical properties of the PP compound PPH 9060 with 60 % calcium carbonate (output: 27.0 kg/h; temperature profile of the dies: 200 to 220 °C)

PP / $CaCO_3$ System: Flow Behavior

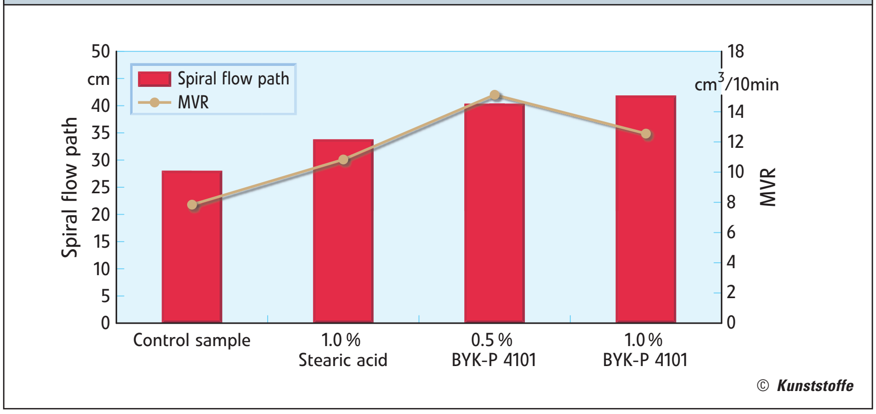

Fig. 6. Flow behavior of the PP compound PPH 9060 with 60 % calcium carbonate (output: 27.0 kg/h; temperature profile of the dies: 200 to 220 °C)

Mobility and Productivity

The molecular design of BYK-P 4101 gives it good melt mobility. This mobility and the viscosity reducing properties result in for example higher productivity during extrusion (e.g. through cycle time and increased throughput). Polymers containing these additives can also be processed at lower temperatures. This property is particularly advantageous for compounds where there is a tendency to decompose the polymer at raised temperatures.

During processing the additive reaches all the relevant interfacial layers quickly, that is the contact surface between the polymer and the extruder barrel wall as well as the polymer filler boundary. In the melt the coefficient of friction and hence the processing resistance is reduced which results in a better throughput. This property can also clearly be seen in injection molded spiral flow moldings. The control sample shows a much shorter flow path than the sample with BYK-P 4101 which clearly has a longer spiral flow, i.e. at the same pressure more substrate is generated (Title picture).

Case Studies for Fillers and Polymers

There is an increasing trend in compounding towards higher filler loadings. Through the use of BYK-P 4101 a better quality than was previously possible can be achieved. The advantages of the additive described apply not only for calcium carbonate as the standard filler, but also for aluminum trihydroxide and talc. Indeed improvements to the mechanical properties were even found with needle shaped wollastonite.

As an example, however, only the most common fillers such as calcium carbonate and talc in PE and PP homopolymers are presented here. BYK-P 4101 was tested in these basic formulations at various concentrations in order to establish the effect of the level of addition. In some instances only the results after optimization are shown. The mechanical properties were assessed using the yield and break strength, elongation at yield and break as well as the modulus of elasticity (tensile E-modulus). The values were determined using a Zwick Material Testing 1465 universal testing machine with a Zmart.Pro

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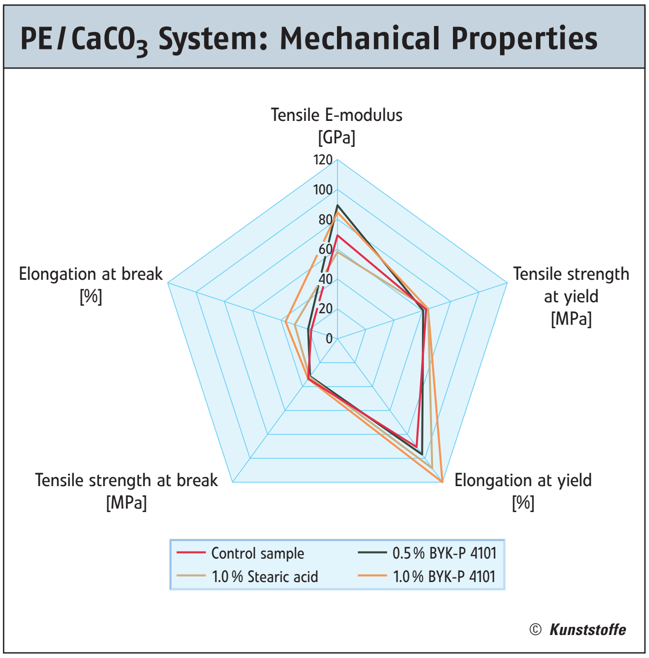

Fig. 7. Mechanical properties of the PE compound Riblene MR 10 with 60 % calcium carbonate (output: 18.0 kg/h; temperature profile of the dies: 180 to 200°C)

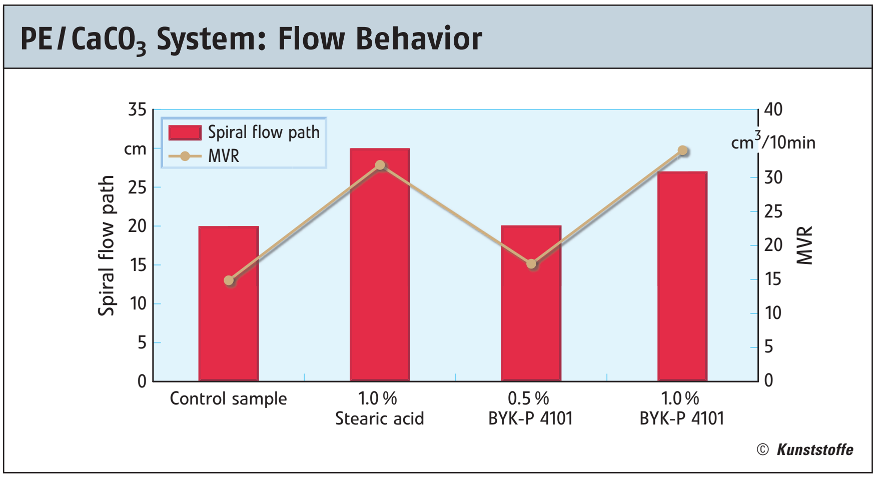

Fig. 8. Flow behavior of the PE compound Riblene MR 10 with 60 % calcium carbonate (output: 18.0 kg/h; temperature profile of the dies: 180 to 200°C)

upgrade (analysis software: testXpert; strain gauge load cell: up to 50 kN). All the compounds were prepared on a Berstdorff ZE 25 UT extruder (25 mm screw; LD 40; screw speed: 1,200 rpm; screw speed of the side feeder: 450 rpm). The test pieces were injection molded on an Engel ES 200/75 HL-V.

PP/talc system: In comparison to the control specimen the addition of BYK-P 4101 produced a higher tensile E-modulus as well as raising the elongation at yield and break whilst at the same time improving the flow properties (MVR). The use of stearic acid on the other hand clearly reduced the tensile E-modulus. The use of BYK-P 4101 in real world systems enables the adjustment of processing without loss of strength in the finished component (Figs. 3 and 4).

PP/calcium carbonate system: In comparison to talc which is a monoclinic layered silicate with an aspect ratio between 5 and 100, calcium carbonate is a fine crystalline mineral with an aspect ratio of ~1 (aspect ratio = length of the particle divided by its thickness). An improvement in the mechanical properties of the PP/calcium carbonate system is therefore an order of magnitude more difficult than for the PP/talc system. Commercially available grafted polyolefins or silanes for fillers do not show an adequate effect in this case.

Since calcium carbonate is however very cost effective it is the preferred filler in the industry. Through the use of BYK-P 4101 a significant increase in the tensile E-modulus, even with this filler, can be achieved. In tests the control samples showed a tensile E-modulus of 36 GPa. With the addition of just 0.5 % of the additive the tensile E-modulus was raised to 68 GPa. The improvements in the processing parameters (e.g. MVR from 9 to 17 cm^3/10 min) are demonstrated even more clearly when compared to stearic acid (Figs. 5 and 6).

PE/calcium carbonate system: The results for BYK-P 4101 were once again confirmed in calcium carbonate filled PE. In this case as well the additive produced a clear increase in the tensile E-modulus from 69 to 90 GPa. As before the improvements in the processing parameters were again confirmed (MVR from 15 to 34 cm^3/10 min) (Figs. 7 and 8). Particularly in the case of calcium carbonate a significant improvement in the quality of the end product can be achieved by the addition of BYK-P 4101. ■

ACKNOWLEDGMENTS

We would like to thank Mrs. Bleisteiner and Mr. Schneider, Eckart GmbH & Co. KG, plant Güntersthal, Velden, Germany, for their very active assistance in producing the SEM pictures.

REFERENCES

An extensive literature index is available from the authors – email address: baerbel.gertzen@altana.com.

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