Fibers Skillfully Concealed

Controlled Crystallization Improves Surface Appearance

Under the generic term “surface-improved polyamides”, several compounds based on engineering thermoplastics have been developed that combine the typical properties of this material group with an optimized surface appearance. Processing conditions and their effect on surface appearance are an important consideration here.

The backrest frame for the MOVYis3 office swivel chair from Interstuhl is made from Ultramid SI surface-improved polyamide

(figures: BASF)

The polyamide (PA) family is characterized by an exceptionally favorable combination of strength, stiffness and toughness along with chemical and thermal stability and is therefore the ideal material group for highly stressed components in all industry sectors (**Title figure**). The inherently very good mechanical properties of polyamide are generally further enhanced by the addition of fibers. **Figure 1 left** shows the stiffness and strength of conditioned polyamide 6 as a function of short glass fiber content. As expected, both these material properties are markedly improved compared with the unfilled product. This increase in strength and stiffness is not however accompanied by embrittlement of the plastic. **Figure 1 right** makes this clear. Although the incorporation of small quantities of glass fibers initially reduces impact strength, larger quantities lead to increased toughness.

A disadvantage of using fibers is the fact that they show up as streaks on the component surface and create a rough, uneven appearance. Particularly with high fiber volume contents and dark colors, the pale fibers form a marked contrast to the polymer matrix. **Figure 2** shows a test component made from standard PA6 filled with 50 % glass fibers. The marks caused by the glass fibers can clearly be seen. For many “visible” applications such a surface appearance is unacceptable and must be concealed by downstream processes such as coating or painting.

The reason for the rough, uneven surface lies in crystallization and solidification behavior. During the injection phase, the glass fibers arrange themselves at the melt front. In components with long flow paths, crystallization of the material starts during the injection phase (**Fig. 3 top**). The associated increase in viscosity means that the polymer is no longer able to coat the glass fibers on the mold wall and they lie exposed on the molded part surface. This can result in an uneven surface appearance (**Fig. 3 below left**). Furthermore, the increased viscosity due to the onset of crystallization makes the weld lines highly visible. These weld lines are then extremely difficult to conceal, even by painting.

With this knowledge, it is now possible to develop polyamides with improved surface appearance without compromising on the glass fiber reinforcement and therefore on the associated good mechanical properties. Through controlled crystallization, the start of crystallization is slightly delayed. As a result, the viscosity of the molding material remains longer at a low level. In this way, the fibers on the mold wall are coated with polymer and so become significantly less visible. The weld lines are also considerably less obvious (**Fig. 3 below right**). On this basis, a portfolio of surface-optimized polyamides has been developed. The Ultramid SI (SI = Surface Improved) product group currently comprises four materials (**Table 1**).

Surface characterization by various methods is discussed below.

Measuring Methods and Test Procedure

Many different methods are suitable for evaluating surface appearance and all have their advantages and disadvantages. The most important criterion is often visual evaluation. Microscopic methods as well as color and gloss measurements can be used for characterization, too. Since the different surface appearance properties are associated with different roughness, tactile roughness measurements are also employed. Another interesting test method that has proved

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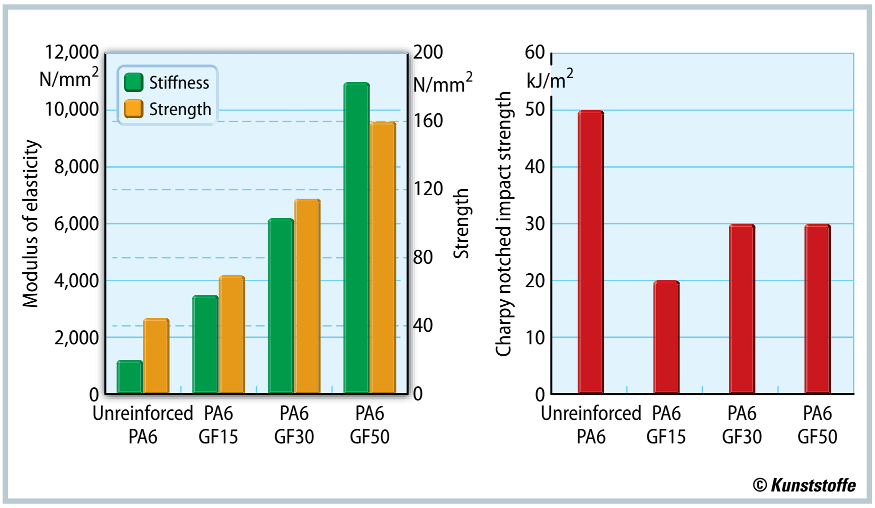

Fig. 1. Stiffness, strength, and toughness of PA6 as functions of glass fiber content

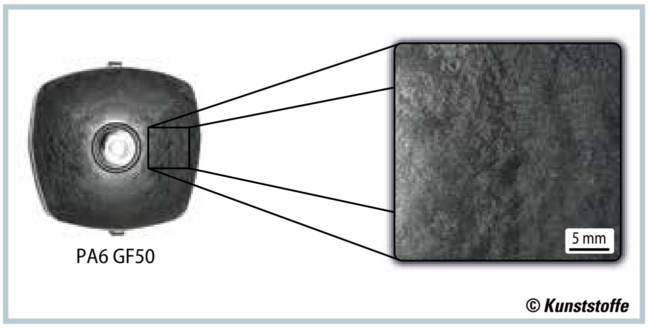

Fig. 2. Test component made from PA6 GF50: the rough surface and exposed glass fibers are clearly evident

suitable is white light interferometry (WLI), which will be briefly discussed below.

White light interferometry (WLI) is a rapid, optical, i.e. non-tactile, 3-D surface measuring technology with the highest vertical resolution (sub-nanometer range) of all optical methods. Unlike in tactile measuring methods, the test specimen is not influenced or damaged by WLI, since it is a contactless method. Other advantages are fast measuring times, low-noise data, and the possibility of characterizing the surface all the way through with transparent media. In contrast to individual profile measurements, the method is designed for 3-D imaging.

In this method, a broadband light source – generally white light – with low coherence is used. The incident light is separated by a beam splitter into a sample beam and a reference beam. The reference beam is reflected by a reference mirror that can be moved in the sub-nanometer range by piezo elements. On the way to a CCD camera, this beam interferes with the sample beam reflected by the sample. Through the phase difference between the two beams, which can be adjusted via the reference mirror, constructive/destructive interference or coherent/non-coherent superposition takes place, resulting in intensity modulation of the reflected light (**Fig. 4**). Using this light intensity modulation with changing reference mirror position, a three-dimensional image of the surface can be generated, as demonstrated (**Fig. 4 right**) by the oak twig on the obverse side of a German one cent euro coin. Further information on this measuring method is given in [1].

To characterize the surfaces, three-dimensional test components were produced by injection molding (**Fig. 2**). The materials used were a standard PA6 with 50 % glass fiber reinforcement and Ultramid B3EG10 SI (surface-optimized PA6 with 50 % glass fibers). Different processing temperatures and injection rates were used. Measurement was carried out at the same position of each component.

Results

Figure 5 shows the results of the WLI tests. The height profiles of the surfaces are represented three-dimensionally. Light-colored zones indicate raised areas, while dark to black regions denote depressions. The two pictures on the left show standard PA6, while the pictures on the right depict surface-optimized Ultramid SI. At the top are samples produced at medium injection rate and below at high injection rate.

It was found that the standard PA6 produced at medium injection rate (**Fig. 5 top left**) leads to a very uneven and rough surface. Individual glass fibers stand out and the viscosity of the polymer matrix during the filling operation is not low enough to fill the voids between the fibers. The resultant protruding glass fibers form a marked optical contrast to the plastic. This effect can be reduced, even with the standard polymer, by reducing the filling time, i.e. increasing the injection rate. The image in **Figure 5 bottom left** shows a very much smoother surface. However, it should be noted that such high injection rates are technically demanding and in some cases not feasible at all.

Material	Product
PA6 GF20	Ultramid B3EG4 SI
PA6 GF30	Ultramid B3EG6 SI
PA6 GF50	Ultramid B3EG10 SI
PA6 GF20 flame retardant	Ultramid B3U40G4 SI

Table 1. Range of surface-improved polyamides (Ultramid SI)

Mechanism	Effects	Countermeasures
Oxidative degradation of the polymer matrix	Discoloration (yellowish brown) Surface erosion Mechanical damage	Stabilizers UV absorbers
Degradation of the pigment/dye	Fading Discoloration	If possible, select lightfast pigments
Surface cracking	Loss of gloss Graying	Stabilizers UV absorbers

Table 2. Damage mechanisms resulting from the action of UV radiation on polymer materials

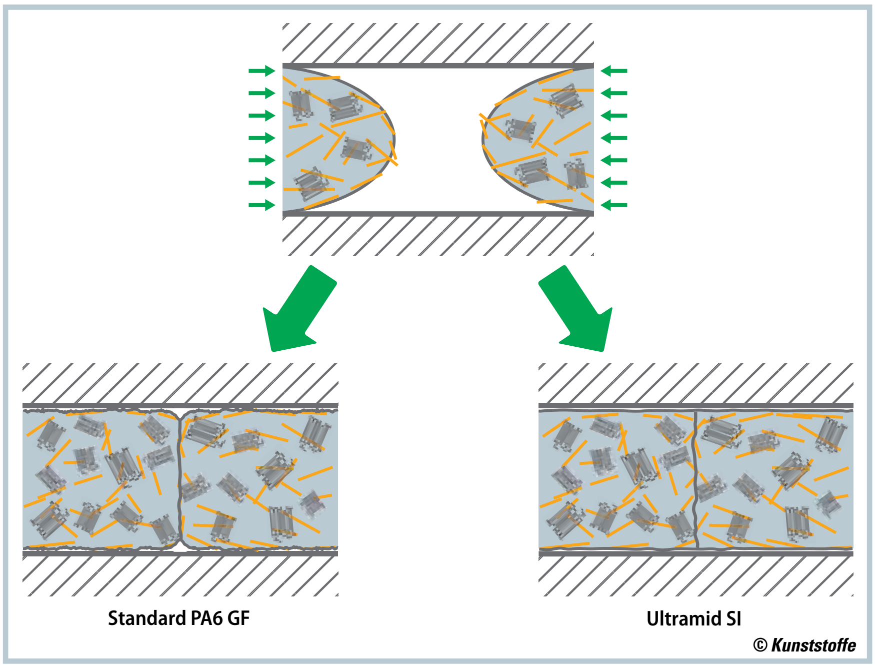

Fig. 3. With standard PA, the mold wall is unsatisfactorily replicated due to the onset of crystallization and a clearly visible weld line is produced. With surface-improved Ultramid SI, these effects are significantly reduced

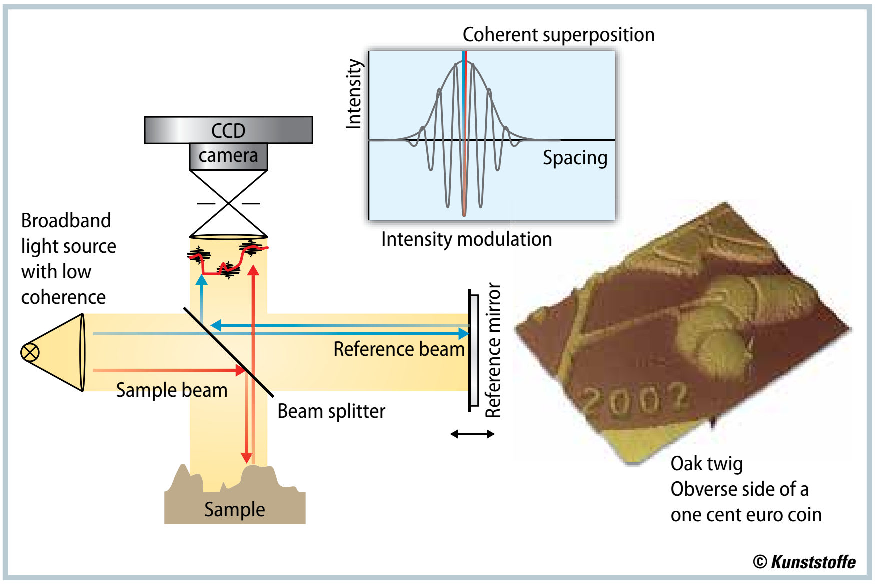

Fig. 4. Principle of white light interferometry

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In the tests with surface-optimized Ultramid SI, a very high-quality surface appearance is universally achieved, even under average processing conditions (**Fig. 5 top right**). Despite the low injection rate, hardly any defects occur. Thanks to the delayed crystallization, the viscosity of the polymer is low enough to fill the voids between the fibers almost completely and so replicate the surface of the mold wall. An increase in injection rate leads to further improvement, with the surface here being entirely free from defects (**Fig. 5 bottom right**).

So, with surface-optimized polyamides, materials are available which, despite high glass fiber content, permit very good surface appearance to be obtained over a wide processing window.

UV Weathering of Reinforced Polyamide

The ability to produce surfaces with a high-quality appearance from reinforced engineering thermoplastics by injection molding opens up the possibility of manufacturing “visible” structural parts with very good mechanical properties without additional post-treatment. It should be noted that painting not only improves surface appearance but, with suitable modification, the paint can also provide protection for the underlying thermoplastic substrate. While eliminating the need for post-painting cuts out a costly production operation, it means that stabilization of the plastic compound against external effects becomes crucial to ensure that the original surface quality and color of the component are maintained long term.

It must be remembered that “visible“ structural parts such as chair backrest frames are exposed, even indoors, to UV radiation attack. Although window glass filters out some of the short-wave, high-energy UVB radiation from sunlight, the part of the UV spectrum that penetrates the window glass is still sufficient to damage polymer materials on prolonged exposure. The mechanisms of this damage, its effects, and possible countermeasures are show in **Table 2**.

Through a combination of suitable light stabilizers and UV absorbers, the surface-improved polyamides have been very successfully stabilized against the

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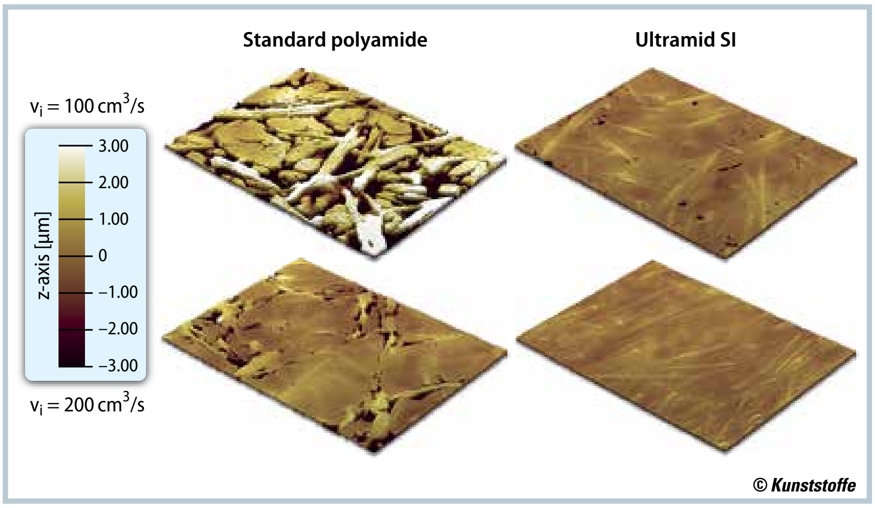

Fig. 5. Three-dimensional representation of the surface topology by white light interferometry; v_i: injection rate

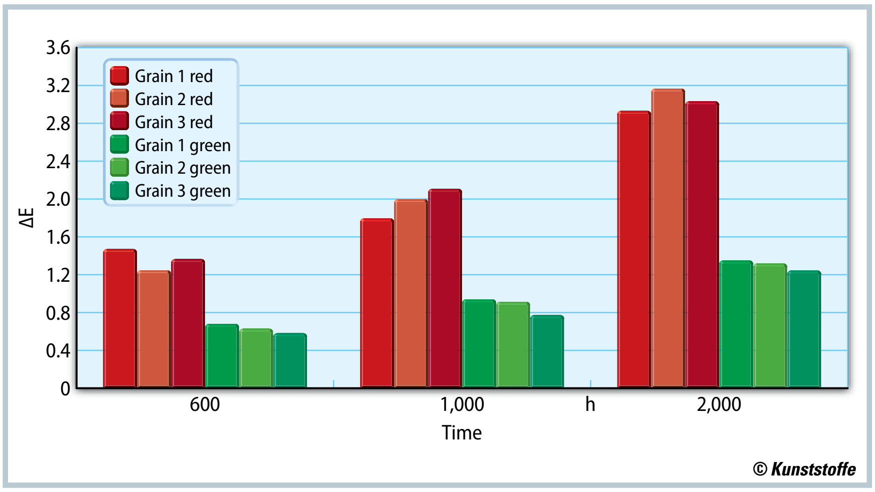

Fig. 6. Color change in sample plaques with different grain depths during accelerated UV weathering (Suntest CPS+ from Atlas Material Testing Technology) (DIN EN ISO 105 B02). The roughness of the grain increases from 1 to 3

damaging effects of UV radiation behind window glass. In conjunction with the color masterbatches specially prepared for the surface-improved polyamides by BASF Color Solutions, various durability requirements can be met. A broad database on the UV resistance of different compound-masterbatch combinations makes it possible to offer tailored solutions.

As **Figure 6** shows, different colors naturally vary in their sensitivity to UV radiation. In addition, bright shades, such as a strong red shades, show up UV damage far more than pale shades. Even without pigment fading, changes due to UV are more evident solely as a result of surface cracking and the greater contrast with glass fibers. However, thanks to optimum stabilization, it has been possible, even with red colors, to limit the color change after 2,000 hours of accelerated UV aging (Suntest) to a ΔE value of 3. Both colors shown, red and green, retain an excellent value of 4 to 5 in visual gray scale assessment after 2,000 hours (the scale ranges from 5 = no change to 1 = very marked perceptible change). In the case of compounds without a specific light stabilizer package, a change in color location at least twice as great can be observed, associated with gray scale values of less than 3. The influence of various depths of grain on the UV resistance of the color, on the other hand, is almost negligible.

Conclusion and Outlook

With the development of surface-improved polyamides, it is now possible to offer the market a range of short glass fiber-reinforced polyamide compounds suitable for the production of structural components that can meet very high mechanical and optical property requirements. Thanks to controlled crystallization behavior, the mold surface can be very accurately replicated. This results in homogeneous, glossy surfaces without any visible glass fiber swirls. At the same time, weld lines on the surface are also significantly reduced compared to results obtained with conventional injection molding materials. In many cases, users are able to dispense with costly post-painting. In combination with suitably tailored color masterbatches, very high UV stability can also be achieved, which ensures optimum color retention, even after many years' exposure to direct sunlight behind glass. ■