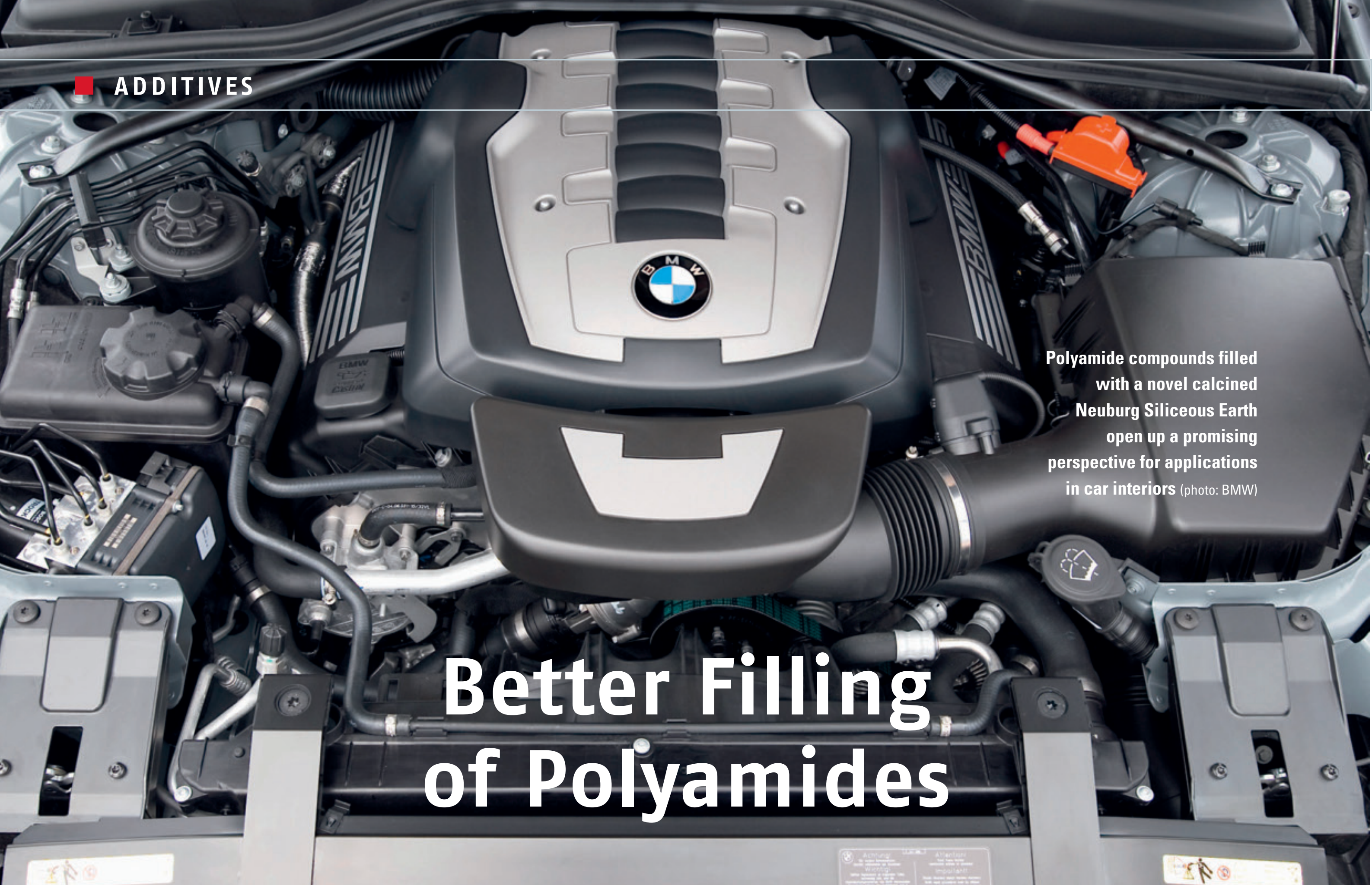

Polyamide compounds filled with a novel calcined Neuburg Siliceous Earth open up a promising perspective for applications in car interiors (photo: BMW)

# Better Filling of Polyamides

**Functional Filler.** The newly developed surface-treated calcined siliceous earth shows an outstanding performance profile in polyamide 66. Tests showed that, compared to its competitors, the filler's advantages lie in the good mechanical properties of the compound, despite the high filler content. Particularly good results were obtained for impact strength and elongation at break.

SIEGFRIED HECKL
NICOLE KNÖR

Because of their high thermal resistance, combined with very good mechanical properties, polyamides are used in many areas of daily life. Their chief applications are as synthetic fibers in textiles, in household and electrical/electronic goods, but also in automotive applications. Their resistance to lubricants and fuels at temperatures up to over 150 °C allows them to be used in air intake systems, fuel lines and engine cowls. These applications determine the requirements for polyamides: besides good processability, they must have a high surface quality, high stiffness and toughness, low warpage and good heat resistance.

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Such tailored properties can be provided by the selective use of fillers – often in combination with glass and carbon fibers.

For low warpage parts, calcined kaolins and wollastonite with a blocky particle shape are preferred as the typical fillers for polyamide. The novel calcined Neuburg Siliceous Earth (Aktifit AM, surface treated) from Hoffmann Mineral GmbH, Neuburg a.d. Donau, Germany, is predicted to open up further possibilities for the above-mentioned applications **(Title photo)**. In the study described below, Aktifit AM is presented as a functional filler for

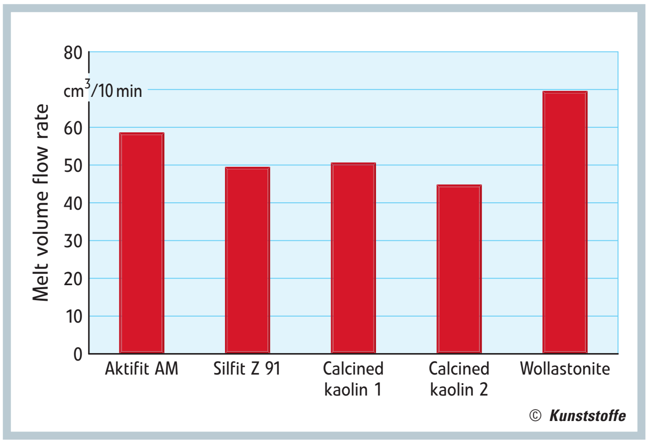


**Fig. 1. The melt flow of polyamide compound containing Aktifit AM did not quite reach the level of the compound filled with wollastonite, but was significantly above the values of the tested compounds with kaolin**

| Characteristics | Calcined kaolin | | Wollastonite | Calcined Neuburg Siliceous Earth | |
|---|---|---|---|---|---|
| | Type 1 | Type 2 | Blocky particle shape | Silfit Z 91 | Aktifit AM |
| Color value L* (Cielab) | 96 | 96 | 95 | 94 | 94 |
| Color value a* (Cielab) | 0.1 | -0.3 | -0.2 | 0.1 | 0.1 |
| Color value b* (Cielab) | 2.5 | 2.3 | 0.8 | 1.6 | 1.6 |
| Particle size $d_{50}$ [µm] | 3.6 | 2.5 | 3.5 | 2.1 | 2.1 |
| Particle size $d_{97}$ [µm] | 17 | 11 | 16 | 10 | 10 |
| Oil absorption [g/100 g] | 66 | 96 | 32 | 59 | 55 |
| Residue > 40 µm [mg/kg] | 100 | 17 | 17 | 3 | 12 |
| Surface treatment | Aminosilane | | Aminosilane | None | Aminosilane |

**Table 1. Characteristics of the fillers used**

thermoplastics – preferably polyamides. The aim is to improve the property profile, specifically the flow and mechanical properties, of polyamide 66 using Aktifit AM compared to typical competitor fillers. Silfit Z 91 is used as an example of a non-surface-treated filler grade.

## Fillers and Property Data

Silfit Z 91 is a naturally occurring mixture of amorphous and cryptocrystalline silica and lamellar kaolinite that has been thermally treated. Aktifit AM is an activated Silfit Z 91, whose surface has been modified with aminosilane. During compounding, the amino groups of Aktifit AM result in good wetting and very good dispersion in the polymer matrix. Moreover, it achieves good bonding in polyamide thanks to hydrogen bonding [1]

In the study, two different calcined kaolins and a wollastonite were used as comparative fillers. Kaolin 1 is a widely used standard grade with a somewhat coarser grain distribution. The finer kaolin 2 is particularly valued for good impact resistance, stiffness and strength. It has a similar grain spectrum to the Neuburg Siliceous Earth variants, but with significantly higher oil absorption. As an example of another filler class, a wollastonite with a blocky particle shape and a low length/diameter ratio was used. All fillers, with the exception of Silfit Z 91 had been surface treated with aminosilane **(Table 1)**.

## Compounding and Injection Molding

For the trials in polyamide 66, polyamide Ultramid A3K, a free-flowing and fast processable injection molding grade from BASF SE, Ludwigshafen, Germany, was used. To allow the typical filler properties to be assessed without the influence of additives, the compound was composed entirely of 60 wt.-% Ultramid A3K and 40 wt.-% filler. Compounding was performed on a ZSK 30 twin-screw extruder (screw diameter 30 mm, L/D 45) from Coperion GmbH, Stuttgart, Germany. The matrix material was predried for at least 8 h at 80 °C in a dry-air dryer. The fillers were processed as supplied, without preliminary drying.

During compounding, the polyamide was charged to the main stream and the filler fed into the polymer melt as a side stream. The extruded strands were pelletized by cold-face cutting and the pellets subsequently homogenized in a tumbler mixer to minimize batch fluctuations.

The test specimens were produced on an FX 75 injection molding machine from Ferromatik Milacron GmbH, Malterdingen, Germany, or on a 320A 600-170 machine from Arburg GmbH & Co. KG, Lossburg, Germany, using a test sample tool as per ISO 294 with interchangeable inserts. Before processing, the pellet stock was pre-dried (dry air 8 h/80 °C) and molded in the injection molding machine according to ISO 1874 with a melt temperature of 305 °C, a mold temperature of 80 °C and a flow-front velocity of 200 mm/s. The demolded test samples were hermetically packaged until testing.

Compounding and injection molding were performed at the German Plastics Institute (Deutsches Kunststoff-Institut, DKI) in Darmstadt, Germany. The tests were carried out at Hoffmann Mineral.

## Flow Behavior and Mechanical Properties

In this study, only freshly injection molded samples of polyamide compounds were tested. The samples had a moisture content of about 0.2 %.

For determining the melt volume flow rate, the pellet samples were taken from the homogenized and predried pellets that were provided for injection molding the test samples. The melt flow **(Fig. 1)** of the polyamide compound containing Aktifit AM did not quite reach the level of the compound with wollastonite, but was significantly above the values of the tested compounds with calcined kaolins.

The tensile test was carried out on the type 1A test sample acc. to DIN EN ISO 527 with a test velocity of 5mm/min until breakage of the samples. No conspicuous differences in the strength (approx. 90 MPa) were ascertained in the tensile test. The compound with kaolin 2 →

**i Contact**

**Hoffmann Mineral GmbH**
**Münchener Strasse 75**
**D-86633 Neuburg a. d. Donau**
**Germany**
**TEL +49 8431 53-350**
**→ www.hoffmann-mineral.com**

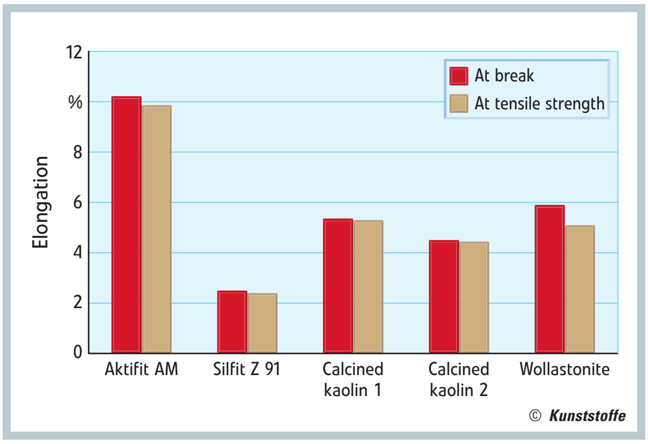


**Fig. 2. Determining the elongation in the tensile test, the samples of the compound with Aktifit AM do not break until almost double the strain is applied, compared to the compound with competitor fillers** (source of the graphics: Hoffmann Mineral)

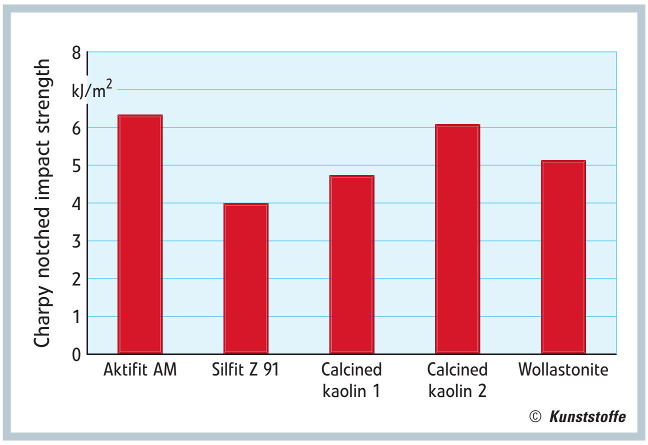


**Fig. 3. The value for the Charpy notch impact strength of polyamide compounds with Aktifit AM slightly exceeds the value for kaolin 2 compounds, but significantly surpasses that of kaolin 1 and wollastonite compounds**

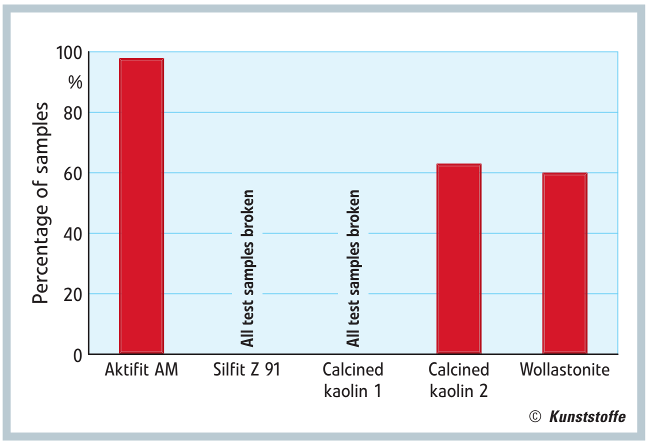


**Fig. 4. The percentage of unbroken samples for Charpy impact strength with a 4 J pendulum containing Aktifit AM, resulted in almost 100 %, thus significantly outperforming the competitor materials**

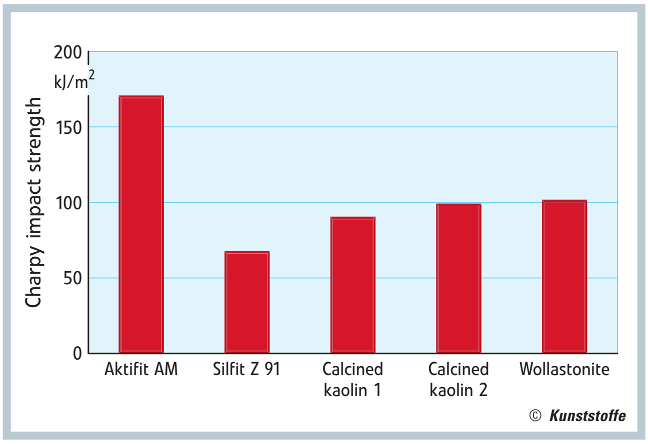


**Fig. 5. In the test for Charpy impact strength with the 15 J pendulum, too, the polyamide compound containing Aktifit AM, showed an impact strength significantly above that of the competitor fillers**

tended to achieve slightly higher results; the compound with wollastonite was at a somewhat lower level. Breakage occurred immediately after the maximum strength was reached. The samples of the Aktifit AM compound failed at almost twice the strain load compared to their competitors **(Fig. 2)**. As an indication of the material stiffness, the tensile modulus was determined at a test rate of 0.5 mm/min. The kaolin 2 compound reached a somewhat higher stiffness, at 6.7 GPa, than the compounds with the other fillers. The Aktifit AM compound, at 6.2 GPa, showed comparable values to the compounds with kaolin 1 and wollastonite.

To determine the Charpy notch impact strength, the standard test specimen, with dimensions 80 × 10 × 4 mm, was provided with a single central notch of the preferred type A (notch base radius 0.25 mm and residual base width 8.0 mm). The notch impact strength was determined according to the standard DIN EN 179-1 by edgewise impact on the unnotched side (i.e. the pendulum strikes the 4 mm side of the test sample). The compounds with Aktifit AM and kaolin 2, at approx. 6 kJ/m$^2$, reached the best values, followed by compounds with kaolin 1 and wollastonite **(Fig. 3)**.

Charpy impact strength is determined according to DIN EN 179-1 on the unnotched standard test sample in the edgewise impact direction. **Figure 4** represents the proportion of unbroken samples in the test with a 4 J pendulum. The Aktifit AM compound, with a proportion of almost 100 % unbroken test samples, was clearly superior to the competitor products.

To bring about the failure of all test samples, the test was performed using a larger pendulum with 15 J energy. The resulting values of the compounds with calcined kaolins and wollastonite was only in the range from 90 to 100 kJ/m$^2$. The Aktifit AM compound, with an impact strength of 170 kJ/m$^2$ achieved almost twice the level of the competitors, and therefore extremely high values for a functional filler in polyamide **(Fig. 5)**.

## Summary

In the tests, the novel calcined Neuburg Siliceous Earth, particularly the surface-activated Aktifit AM, showed a better melt flow behavior in the polyamide compound and comparatively high tensile strength of 90 MPa and stiffness (6.2 GPa) compared to calcined kaolins. The doubling of the values for elongation at break and impact strength was notable.

This property profile, together with the excellent processing behavior of calcined siliceous earth, characterized by a low proportion of grit, good dosability and good wetting and dispersion behavior, offers clear advantages over competitor fillers. The compound with calcined siliceous earth can be injection molded into parts with low warpage, high surface quality and high heat resistance.

This opens up future applications for Aktifit AM as filler for polyamide wherever low warpage combined with high surface quality is just as important as good melt flowability, high elongation at break and high impact strength even in the freshly molded, dry state. Compared to competitor fillers, the calcined Neuburg Siliceous Earth thus has a comprehensive portfolio of properties for better filling of polyamide 66. ■

## REFERENCES

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## THE AUTHORS

SIEGFRIED HECKL, born in 1966, is an area sales manager at Hoffmann Mineral GmbH, Neuburg a.d. Donau, with responsibility, among others, for the plastics industry in Germany;
siegfried.heckl@hoffmann-mineral.com

DR. NICOLE KNÖR, born in 1978, is an area sales manager at Hoffmann Mineral GmbH, Neuburg a. d. Donau, for Australia and Pacific;
nicole.knoer@hoffmann-mineral.com



 