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Unternehmensgruppe Hoffmann

Calcined Neuburg Siliceous Earth

in thermoplastics:

Polybutylene terephthalate (PBT)

Author: Petra Zehnder
Hubert Oggermüller

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HOFFMANN MINERAL GmbH • Muenchener Strasse 75 • D-86633 Neuburg (Donau) • Phone +49 (0) 8431 53-0
www.hoffmann-mineral.com • e-mail: info@hoffmann-mineral.com

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1 Introduction

Polybutylene terephthalate (PBT) as a raw material finds extensive application in many areas predominantly in the automotive and electrical industries.

Important properties here are easy processing, high strength and stiffness, high dimensional stability, good friction and wear properties as well as a good chemical resistance against many solvents.

For low-warpage parts with attractive surfaces, frequently glass beads are added as fillers.

Compounds filled with mineral fillers have hardly been available so far, because of their unsatisfactory property profile.

In the following report, Calcined Neuburg Siliceous Earth is presented as a functional filler for PBT.

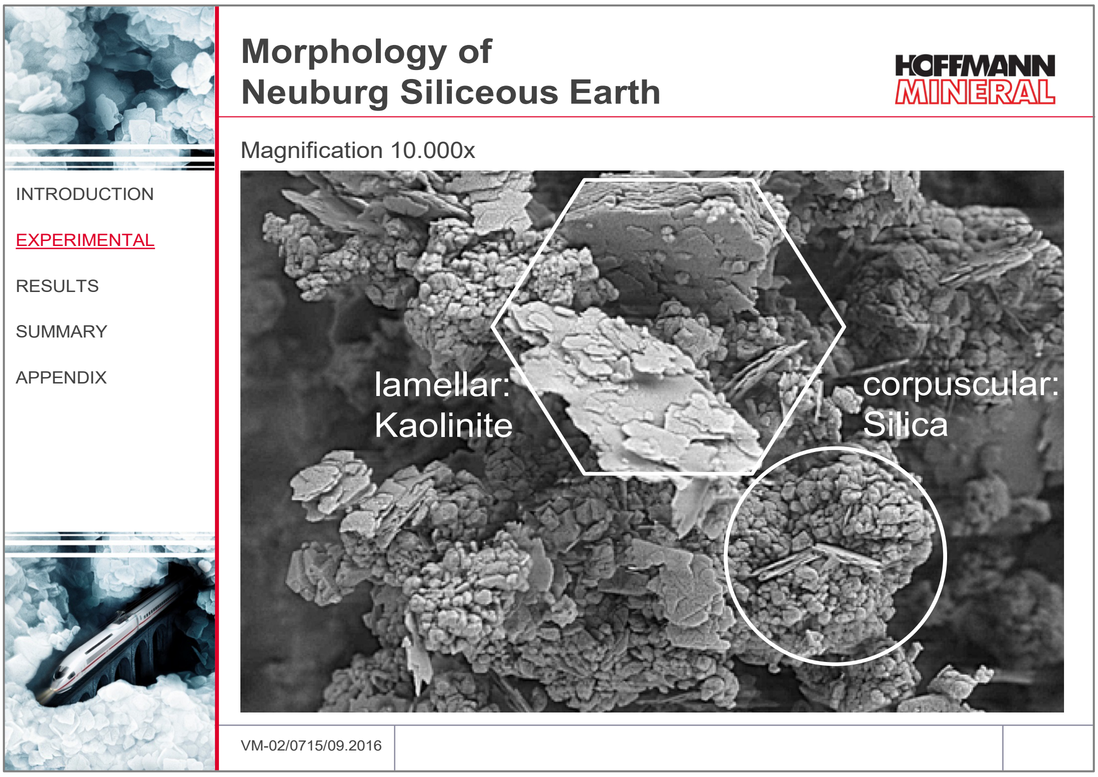
The study includes a comparison of the property profile of Aktifit VM, a calcined surface-treated Neuburg Siliceous Earth grade, with surface treated glass beads with respect to flow properties, dimensional stability at high temperatures and mechanical properties.

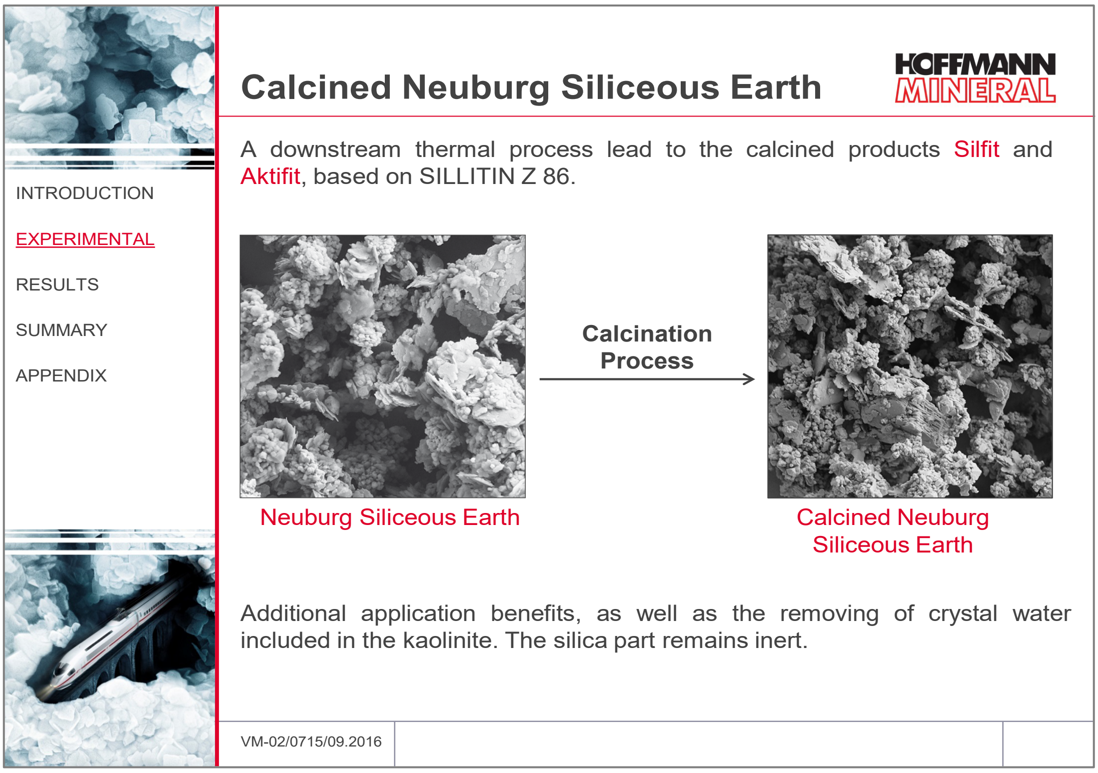
2 Experimental

2.1 Neuburg Siliceous Earth

Neuburg Siliceous Earth, extracted in the surrounding of Neuburg (Danube), is a natural combination of corpuscular Neuburg silica and lamellar kaolinite: a loose mixture impossible to separate by physical methods. As a result of natural formation, the silica portion exhibits a round grain shape and consists of aggregated, cryptocrystalline primary particles of about 200 nm diameter.

The special morphological composition of Neuburg Siliceous Earth, which represents a class of minerals on its own, is illustrated here by a SEM photograph.

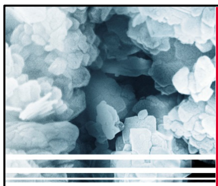




During calcination, Neuburg Siliceous Earth is subjected to a heat treatment. The components and the thermal process lead to a product that offers special performance benefits as a functional filler.

2.2 Fillers and their characteristics

The table shows a summary of the most important filler properties.



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Fillers and Characteristics

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Filler	Description	Functionalization
Glass beads	d_{50}: 15-30 µm, d_{90}: 30-80 µm	Suitable for PBT
Aktifit VM	Calcined Neuburg Siliceous Earth d_{50}: 2 µm, d_{97}: 10 µm Hydrophobic Moisture less or equal 0.1 %, even with high air humidity No pre-drying necessary	Vinyl

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Aktifit VM is an activated calcined Neuburg Siliceous Earth grade whose surface was modified by treatment with a special vinyl functional group.

For comparison purposes, a glass bead grade suitable for PBT was used.

2.3 Compounding and molding

The study was based on a stabilized medium-viscosity PBT. The compounds contained 70 weight percent of polymer and 30 weight percent of filler resp. glass beads.

The compounding was made in a Werner & Pfleiderer twin-screw extruder ZSK 30 (screw diameter 30 mm).
In the compounding operation, the PBT was introduced into the main stream, and the filler was added to the melt via side feeder. The extruded strands were pelletized by cold-face cutting.

The preparation of the test specimens was made on a screw injection molding unit from Krauss Maffei, using a specimen tool according to ISO 294 with exchangeable inserts for the individual test specimens.
Prior to processing, the pellets were pre-dried for at least 16 hours in a vacuum furnace at 80 °C (residual moisture <0.04 %).

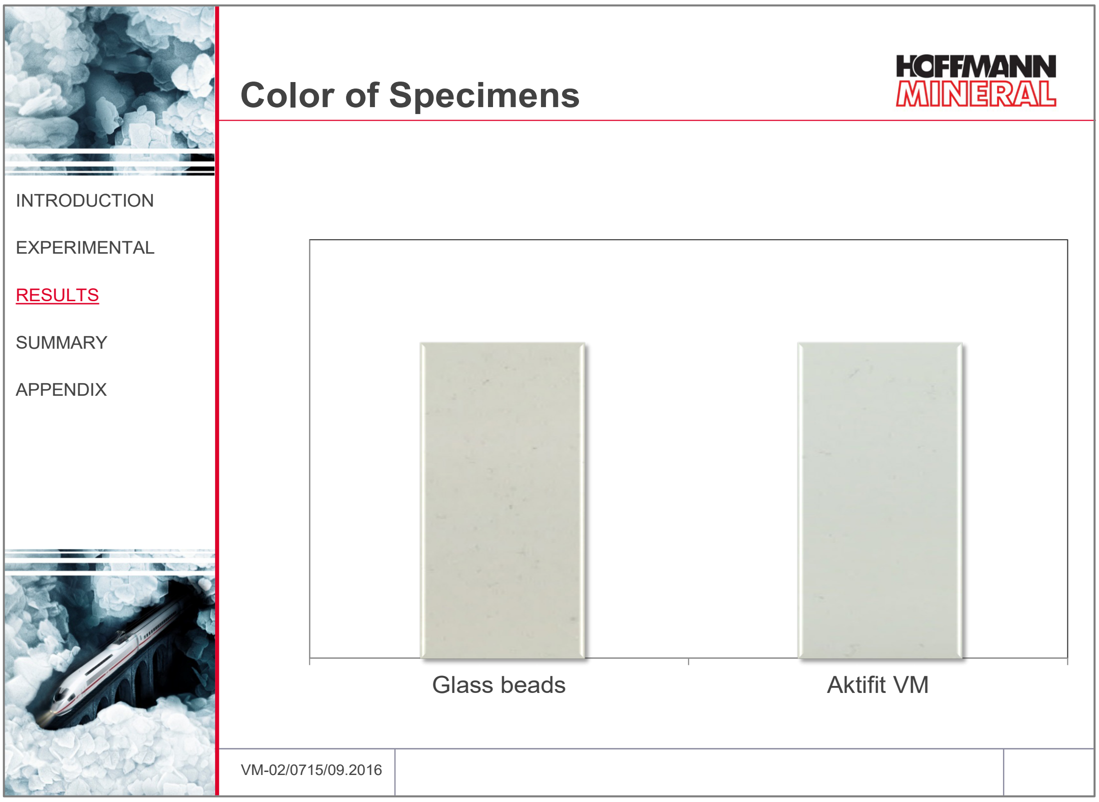
The PBT granules were injected with a melt temperature of 260 °C at a mold temperature of 80 °C.

Compounding, injection molding and subsequent tests were carried out at A. Schulman in Kerpen, Germany.

3 Results

3.1 Color of specimens

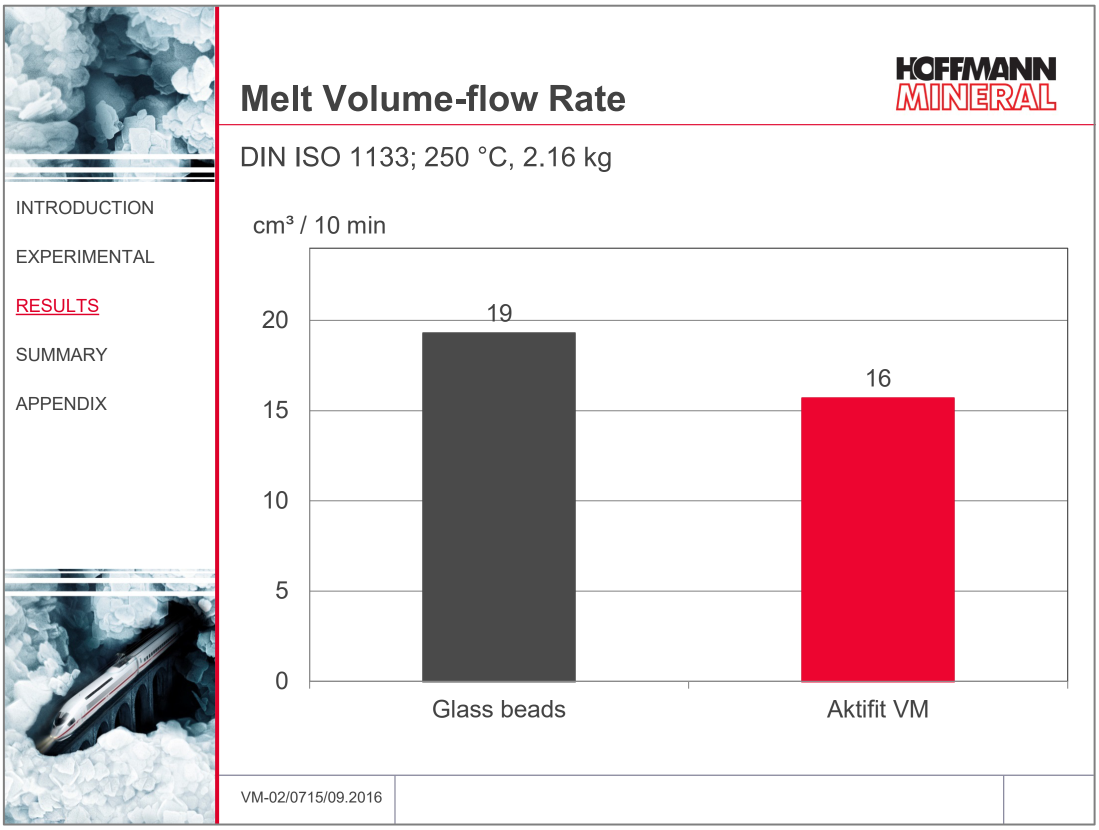
The graph shows parts of the sample sheets, photographed under the same light conditions.



Aktifit VM results in a very neutral color of the compound.

3.2 Melt volume-flow rate

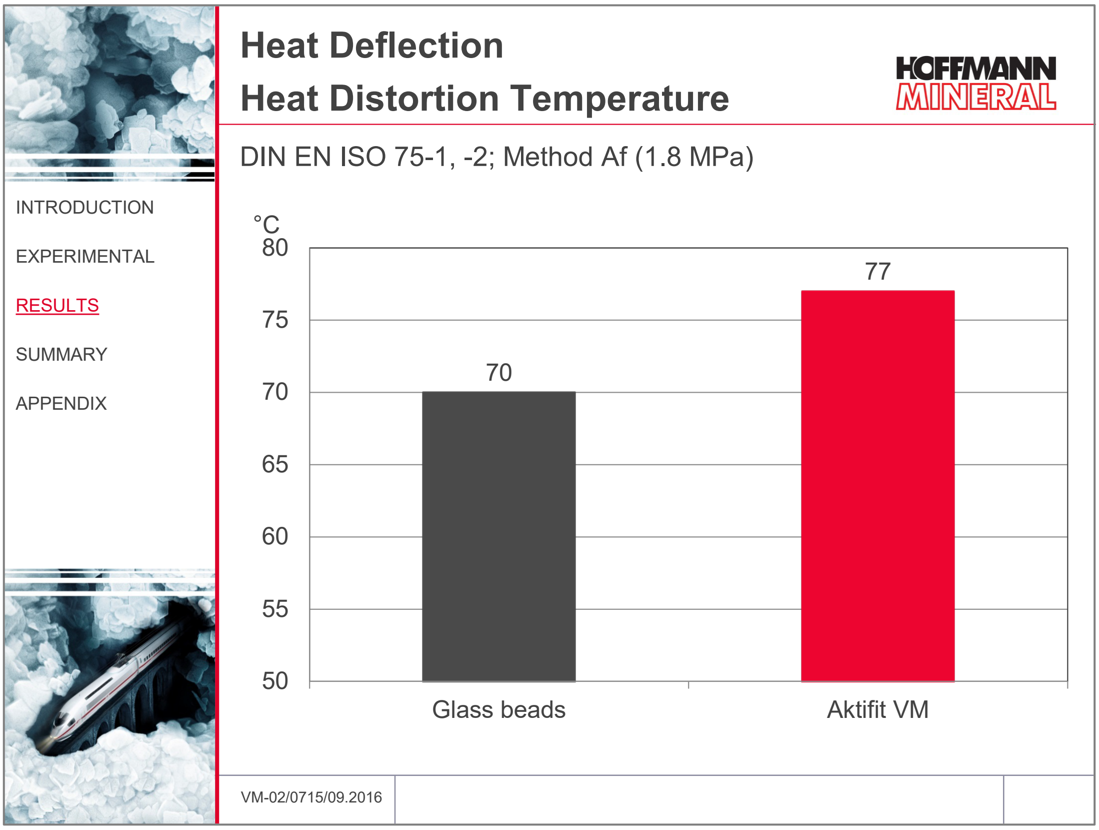
Samples for this test were taken from the homogenized and pre-dried pellets ready for injection molding.



Flow properties with Aktifit VM are somewhat poorer than with glass beads.

3.3 Heat deflection

For the determination of the Heat Distortion Temperature (HDT), the samples were bent according to the 3-Point bending principle, charged with a constant load and heated with a rate of 120 K/h. The required load as a function of the sample thickness is calculated in order to arrive at an outer fiber stress of 1.8 MPa (Method A). The HDT then is the temperature that leads to a defined standard bending corresponding to 0.2 % outer fiber strain.

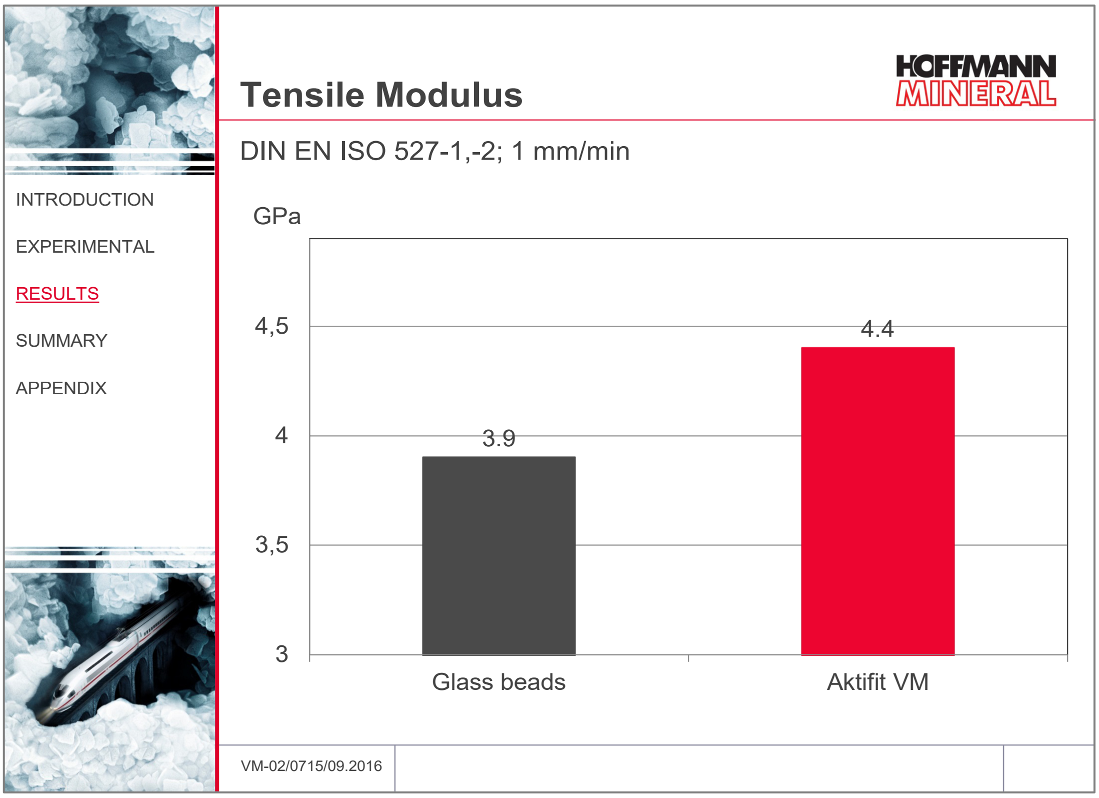


With Aktifit VM the HDT is somewhat higher than with the glass beads, and consequently the compound offers a slightly improved dimensional stability at higher temperature.

3.4 Tensile test

Tensile modulus

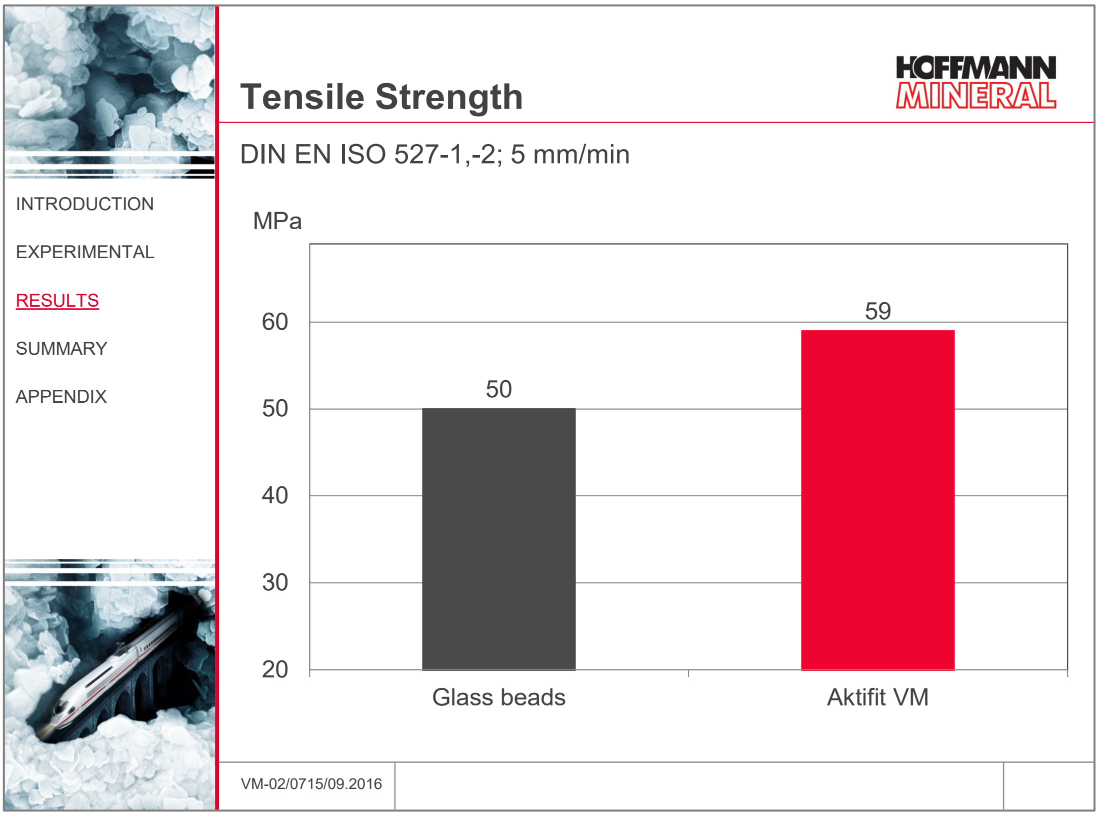
In place of the stiffness of the material, the tensile modulus was determined in a tensile test at an extension rate of 1 mm/min.



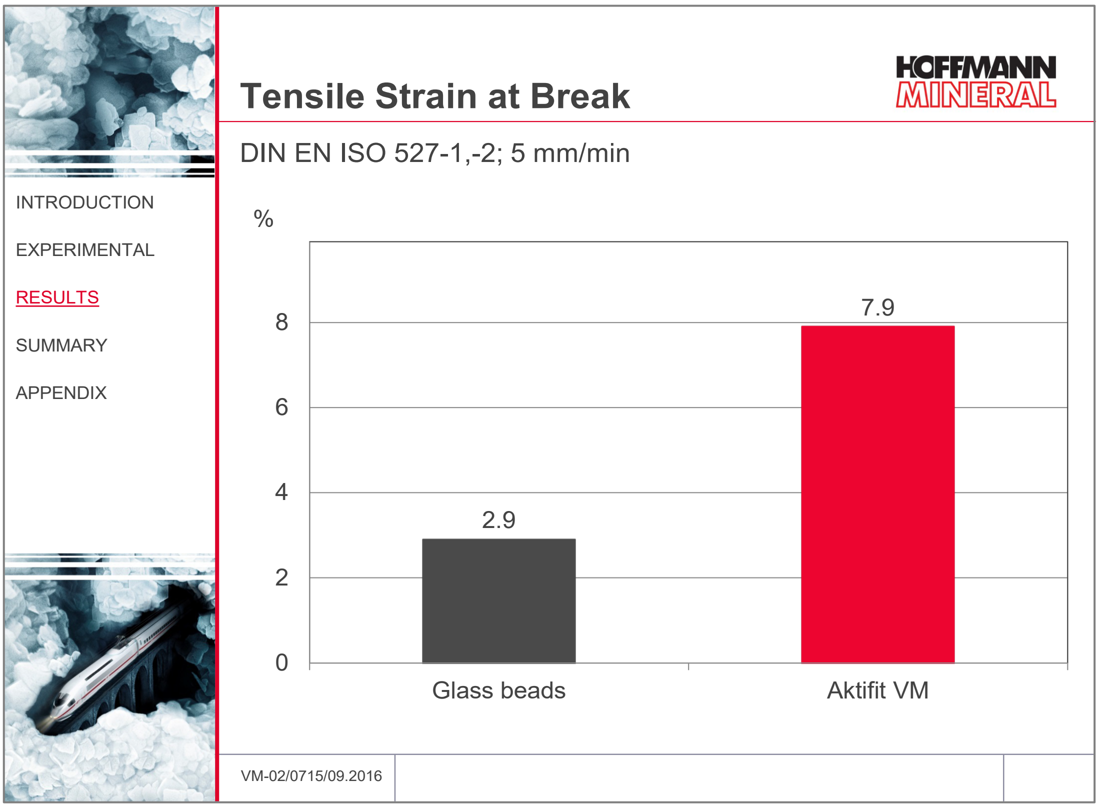
Aktifit VM imparts a stiffness higher by 12 % compared with the glass beads.

Tensile strength and tensile strain at break

The test was run with specimens type 1A at an extension rate of 5 mm/min up to break.



The tensile strength obtained with Aktifit VM is approximately 18 % higher than with the glass beads.

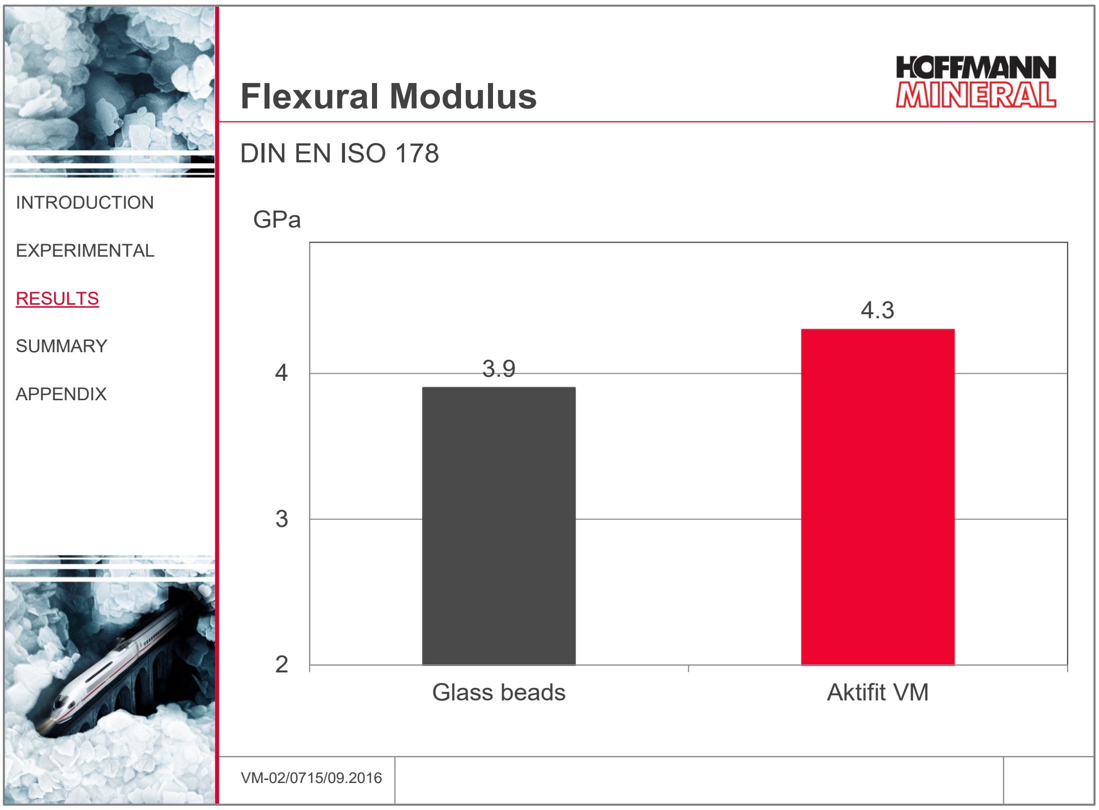


In comparison with the competitive filler, the samples filled with Aktifit VM only break at about the double strain. This effect is welcome for applications where building parts when mounted have to resist to certain deformations, as for example snap hook joints.

3.5 Flexural test

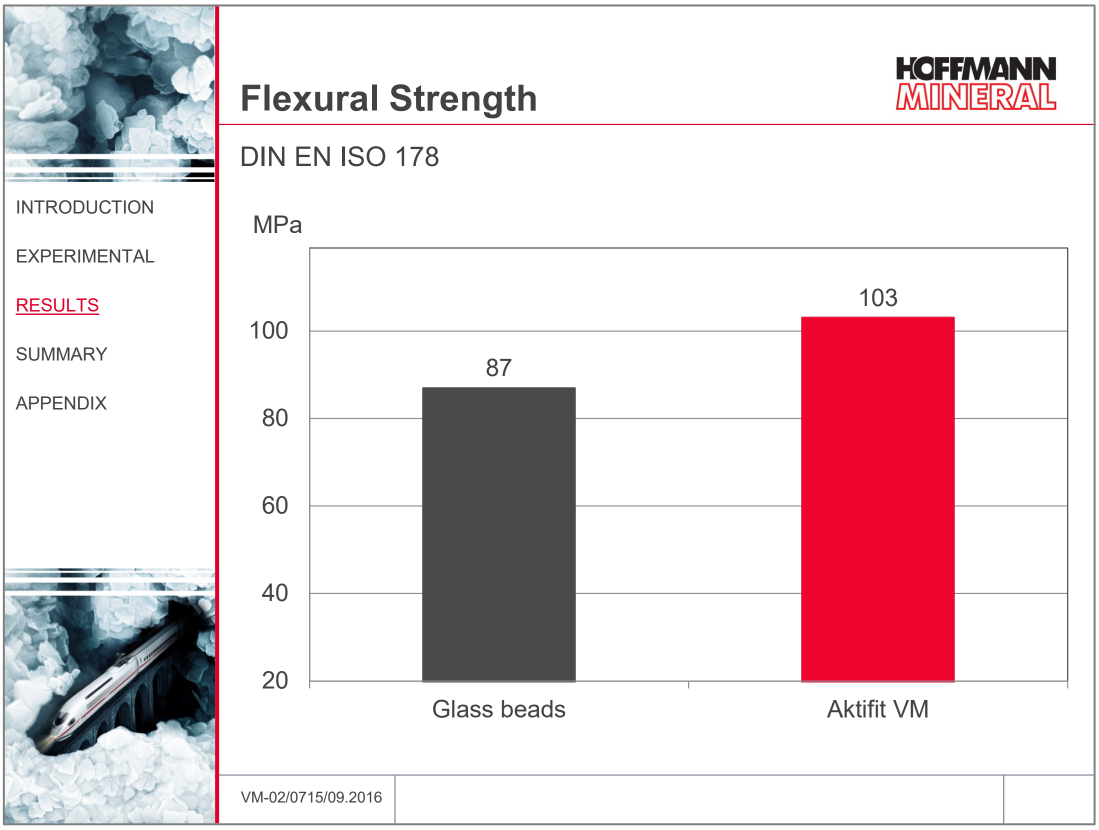
The three-point bending test was carried out in accordance with DIN EN ISO 178. Basically the bending test leads to similar results as the tensile test.

Flexural modulus

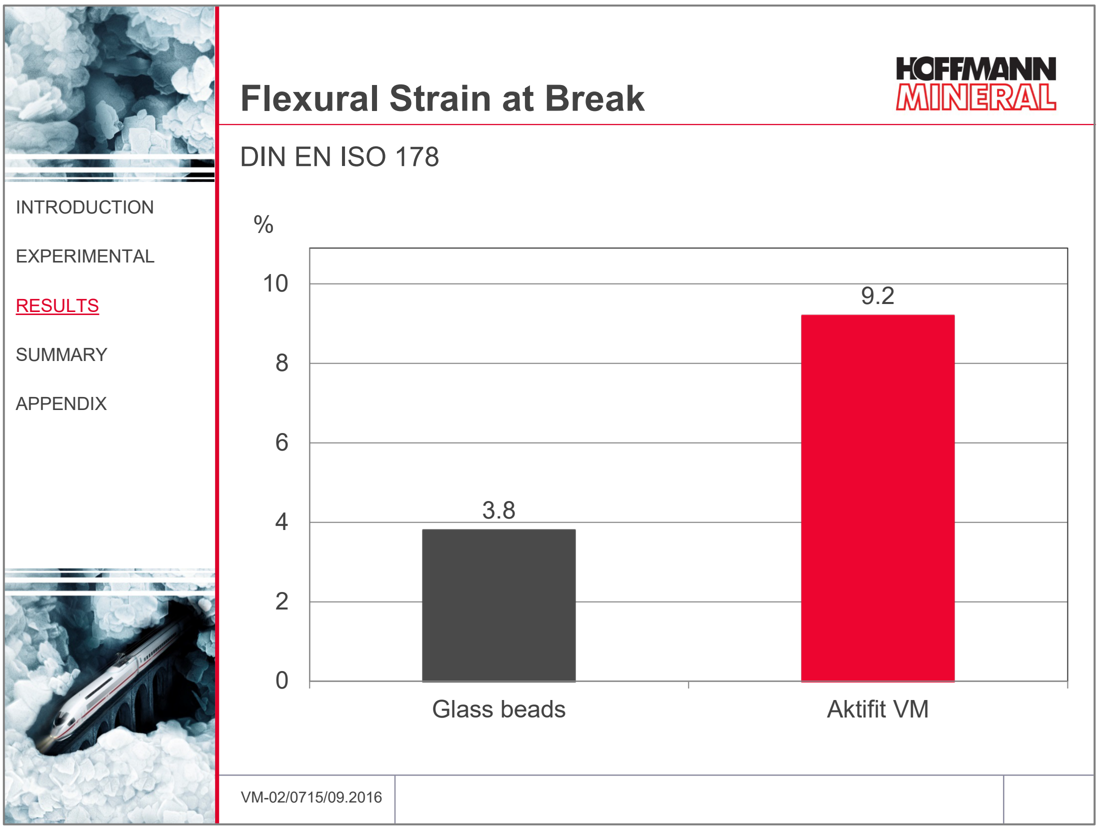


Aktifit VM gives rise to a higher bending strength compared with the glass beads. The results approximately correspond to the modulus determined in the tensile test.

Flexural strength and flexural strain at break



The flexural strength with Aktifit VM is higher by 18 % compared with the glass beads; which is in agreement with the results for the tensile strength.



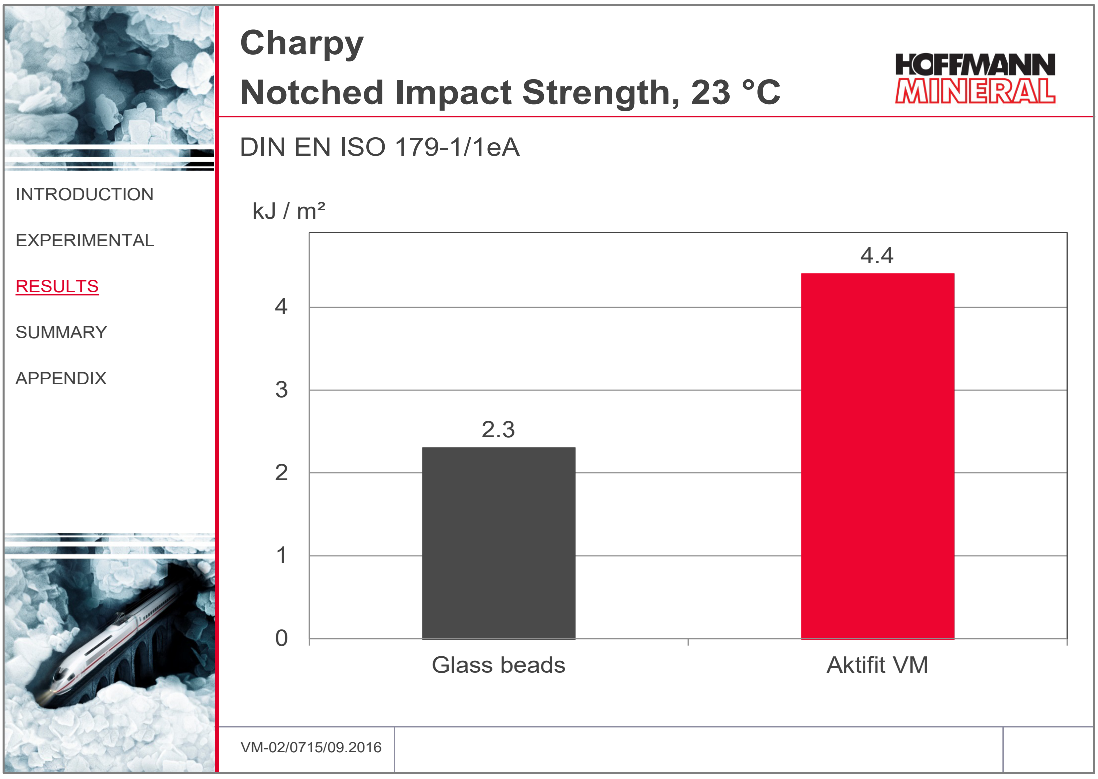
Also in the bending tests the samples with Aktifit VM break at about twice the elongation compared with the glass beads.

3.6 Impact strength Charpy

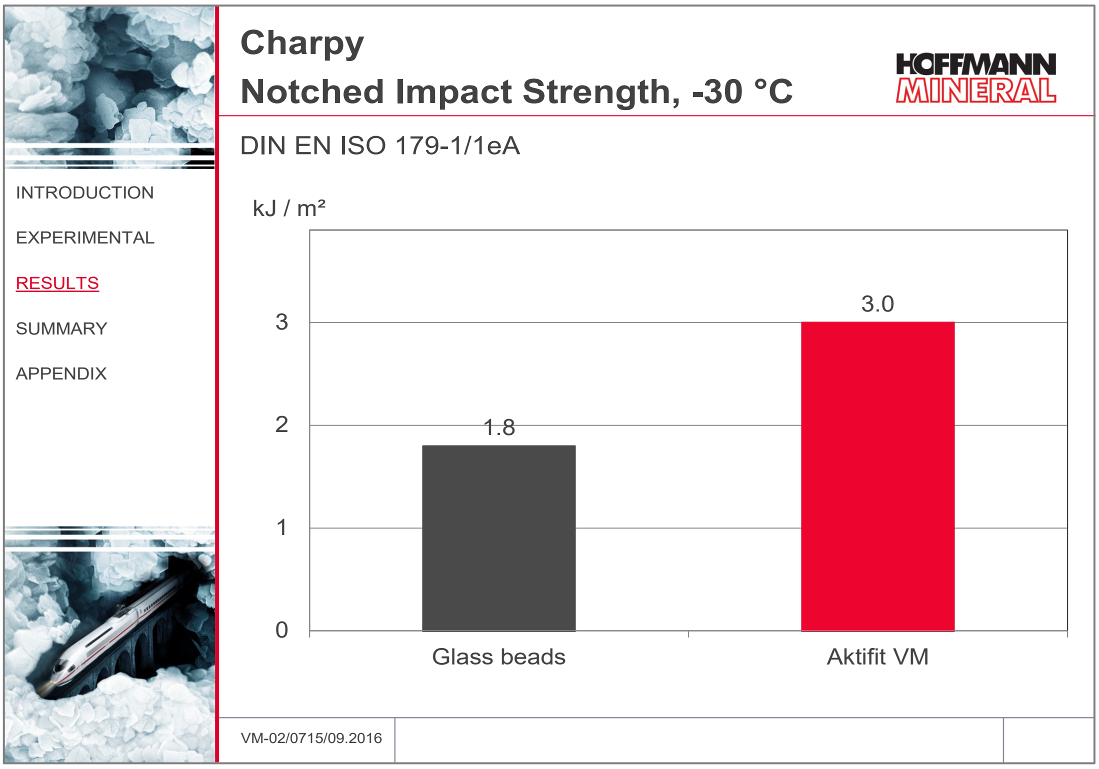
According to the Charpy method, the sample is supported unclamped at both ends and hit in the middle with a pendulum hammer.

Notched impact strength

For this test, the standard samples are provided in the middle with a single notch of the preferred kind A (notch root radius 0.25 mm, rest ground width 8.0 mm). The impact strength was determined according to the standard test with an impact on the narrow side opposite the notch (edgewise).



Compared with the glass beads, Aktifit VM gives rise to about twice the notched impact strength.

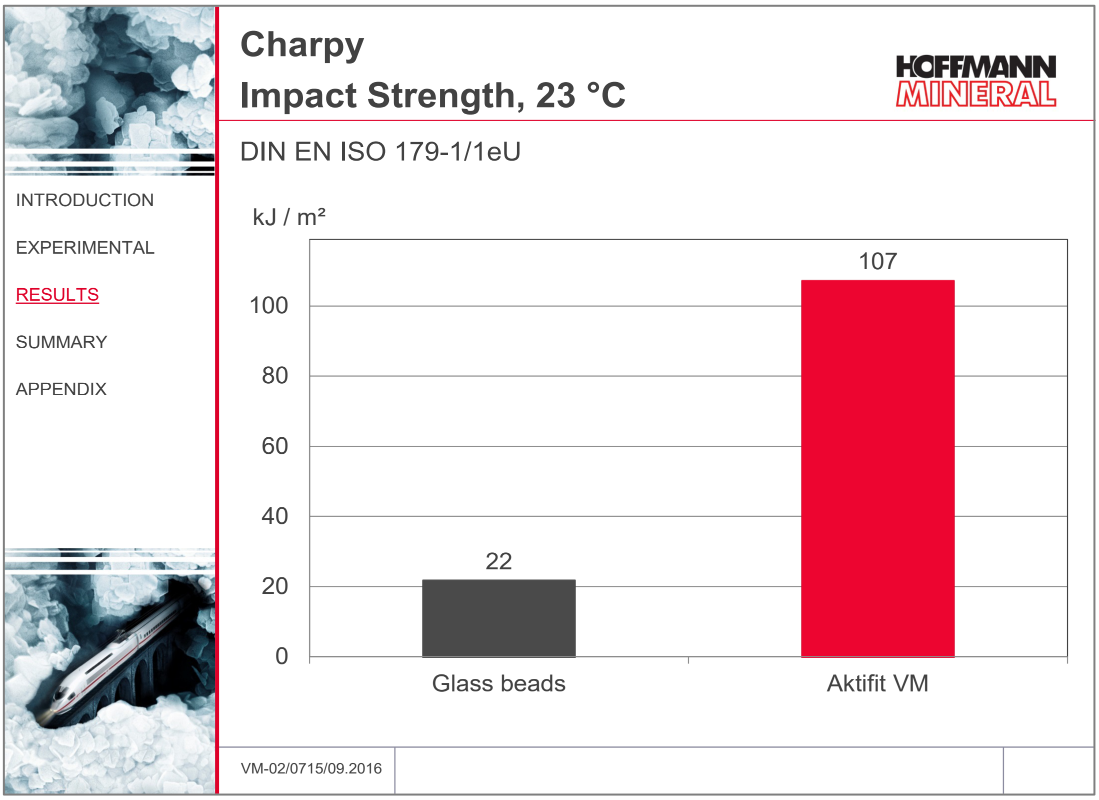


At low temperature, the general level of the notched impact strength is somewhat lower. All the same, Aktifit VM comes out markedly higher.

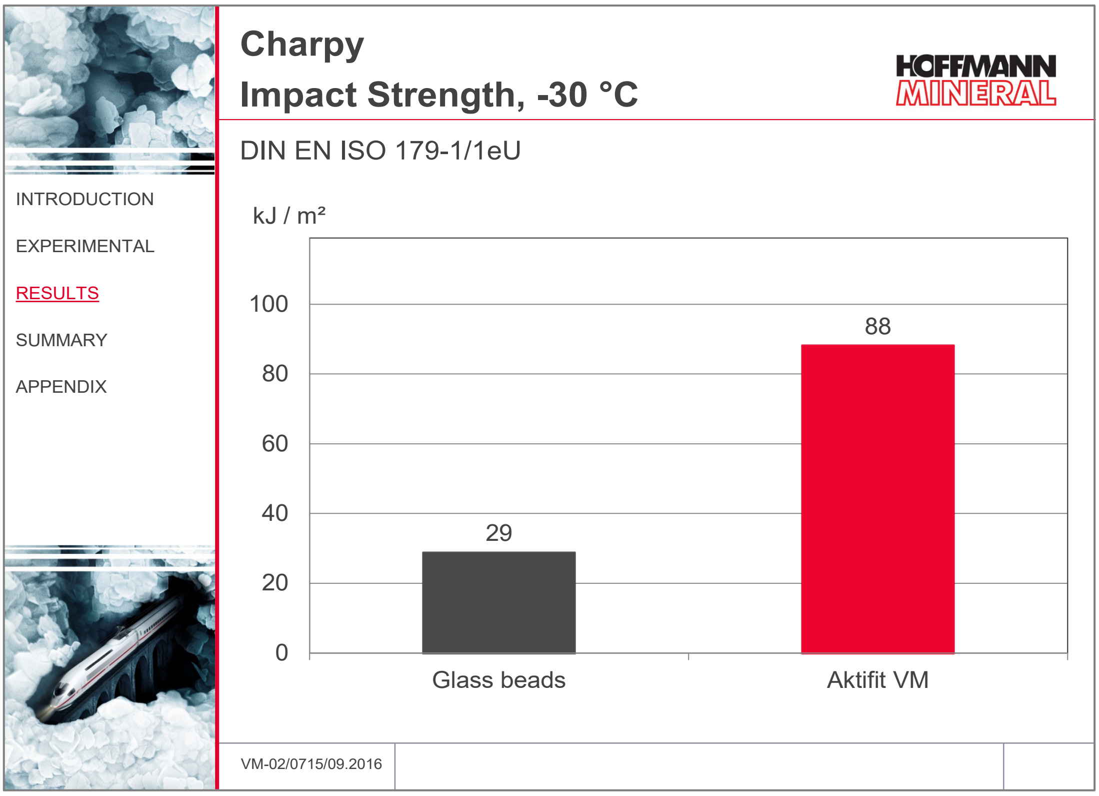
Unnotched impact strength

The test was run on unnotched standard samples 80 x 10 x 4 mm with the impact hitting the narrow side, i.e. the pendulum hits the 4 mm side of the sample.

In order to assure the breakage of all samples, an impact pendulum of an energy capacity of 7.5 J was used. When working with the usual 4 J pendulum, in view of the lower energy capacity only impact strength levels up to 100 kJ/m² max. can be differentiated. For higher levels – this way also for Aktifit VM – the result would be registered as „No Break“, and no statement about a quantification of the improvement could be given.



Here again, Aktifit VM outperforms the competition material by a five times higher result.

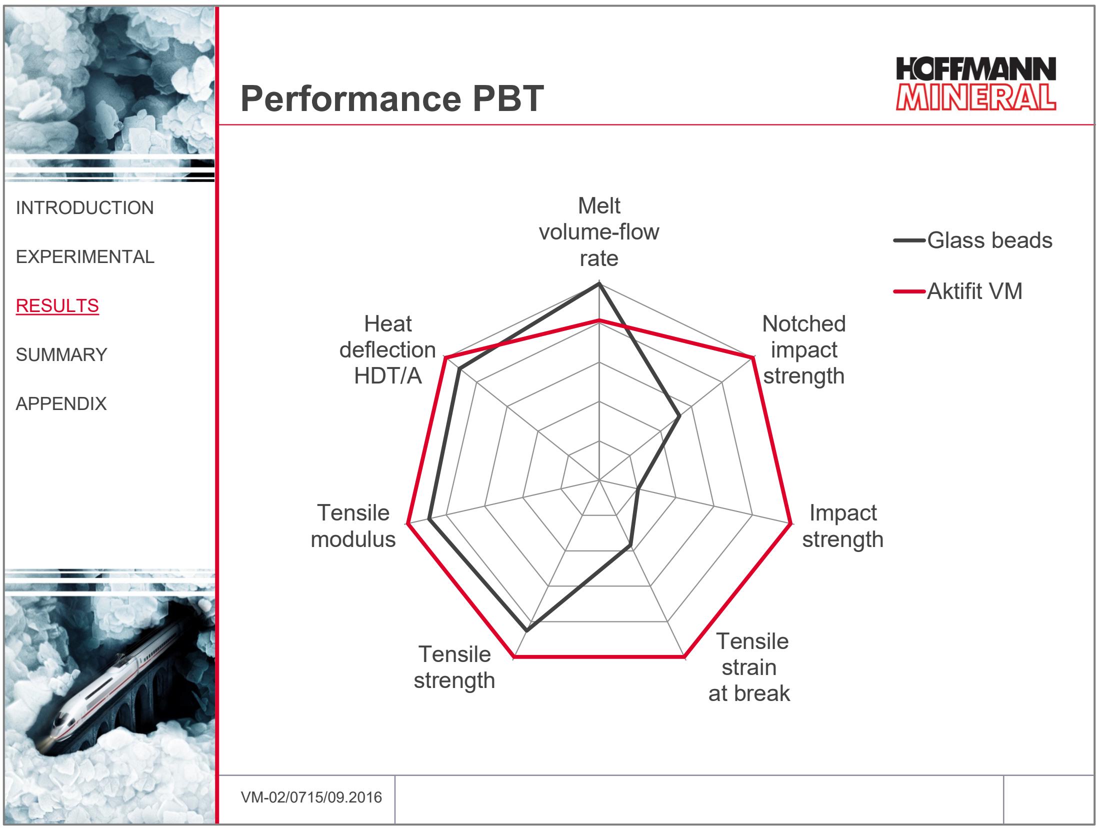


The same situation is true at low temperature (-30 °C): the compound with Aktifit VM shows an impact strength several times higher.

3.7 Black compounds

Coloring the compounds with carbon black often causes a loss of mechanical properties. Aktifit VM was therefore also compounded with a black color batch.
Aktifit VM reached results approximately comparable to the natural-colored compound, no significant difference was noted.

3.8 Overview: performance PBT



Aktifit VM outperforms the glass beads used in almost all properties.

4 Summary

Aktifit VM shows in PBT vs. surface treated glass beads:

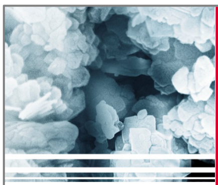
- Lighter and more neutral color of the compound
- Somewhat lower melt flow rate
- Higher heat deflection temperature
- Higher stiffness
- Higher strength
- Very high strain at break
- Excellent (notched) impact strength, even at low temperature

Compared with glass beads, Aktifit VM gives rise to an improvement of nearly all mechanical properties, in particular elongation at break and impact strength.

In another comparison with a loading of 15 % glass fibers, Aktifit VM showed the following advantages: higher dimensional stability, brighter and more neutral color, and again a markedly higher impact strength and elongation at break.

Application areas for Aktifit VM in PBT should be found where low warpage in combination with high surface quality are just as important as good processability, high elongation at break and high impact resistance.

5 Appendix: Summary table



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			Glass beads	Aktifit VM
Melt Volume-flow Rate		cm³/10 min	19	16
Heat Distortion Temp. HDT/A		°C	70	77
Tensile Modulus		GPa	3.9	4.4
Tensile Strength		MPa	50	59
Tensile Strain at Break		%	2.9	7.9
Flexural Modulus		GPa	3.9	4.3
Flexural Strength		MPa	87	103
Flexural Stress 3.5 %		MPa	87	98
Flexural Strain at Break		%	3.8	9.2
Impact Strength	23 °C	kJ/m²	22 C	107 C
Charpy, 1eU	-30 °C	kJ/m²	29 C	88 C
Notched Impact Strength	23 °C	kJ/m²	2.3 C	4.4 C
Charpy, 1eA	-30 °C	kJ/m²	1.8 C	3.0 C

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