Calcined Neuburg Siliceous Earth

in thermoplastics:

Polyketone (PK)

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VM-2/0416/02.2019

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1 Introduction

Polyketone is a versatile high performance plastic for technical parts with high requirements concerning mechanical properties, chemical resistance and tribological properties. The product has again become available in 2014.

Along with outstanding wear and chemical resistance it offers good mechanical properties and a low water uptake.

In view of the polymer-inherent tendency towards an increase of the viscosity during processing, compounds loaded with mineral fillers so far have been hardly available.

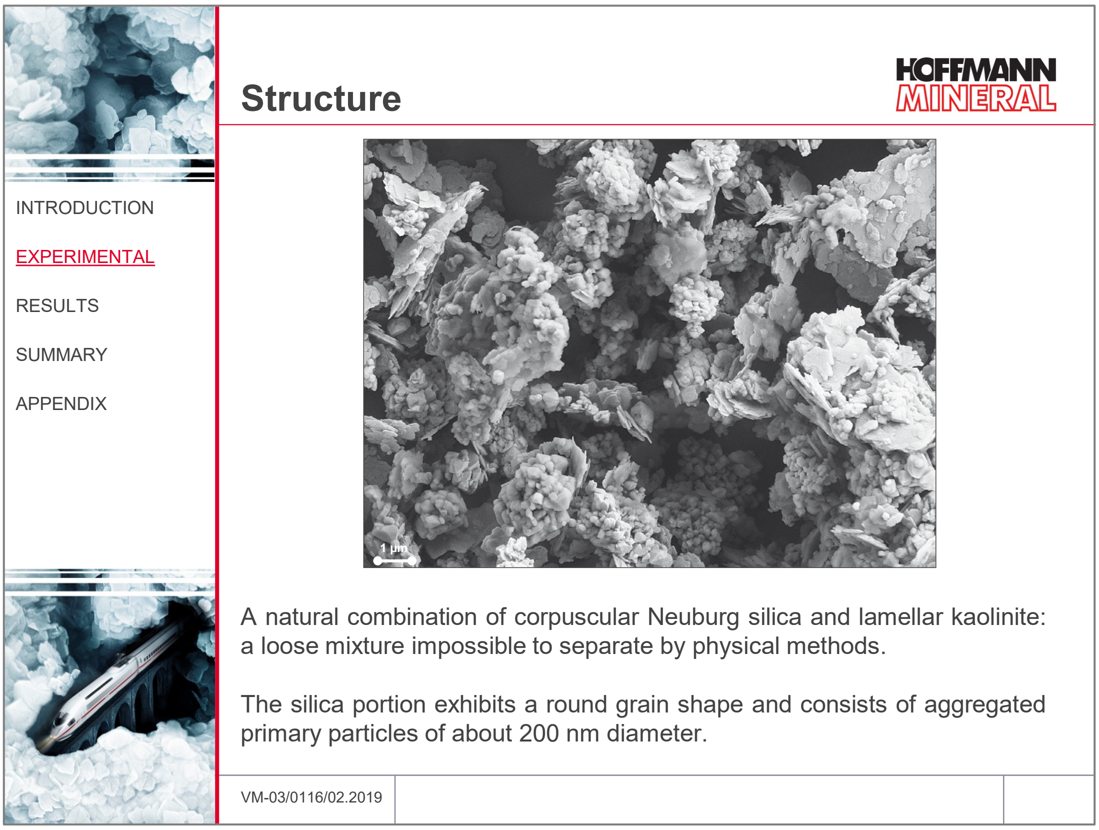
The present study has the objective of presenting calcined Neuburg Siliceous Earth as a functional filler for polyketone.

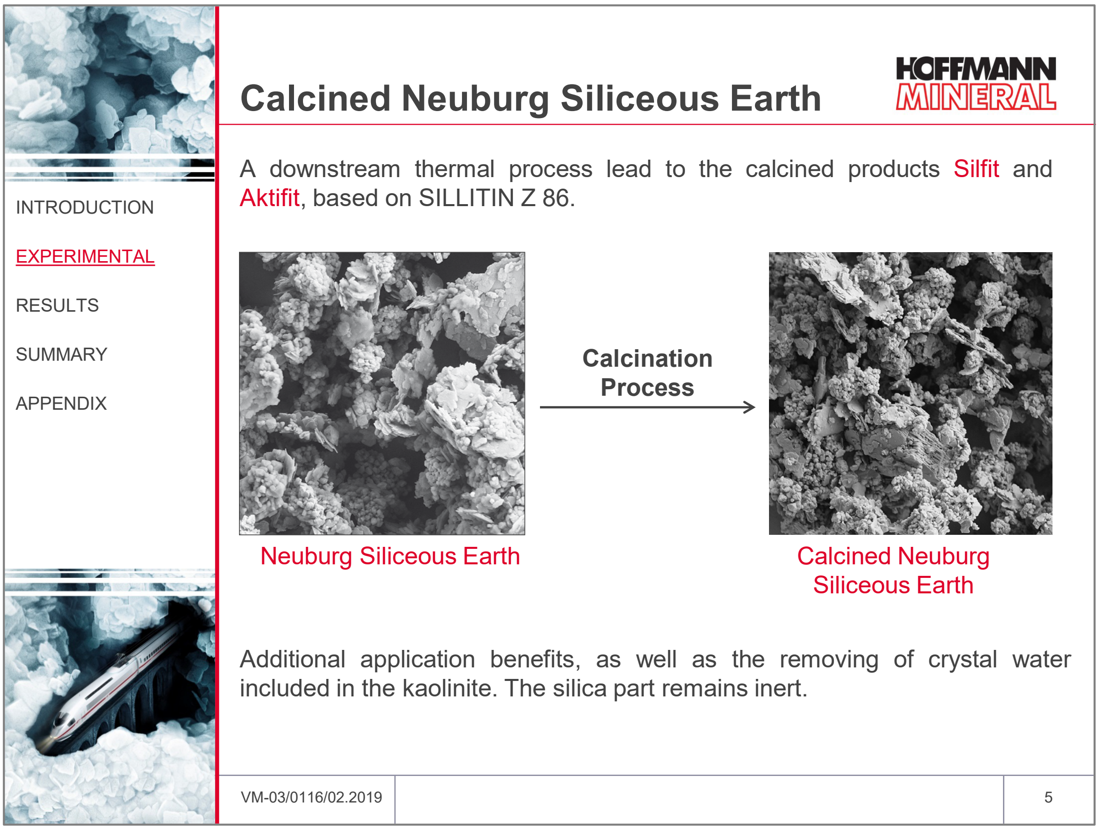
The report will discuss the performance of compounds with calcined Neuburg Siliceous Earth grades in comparison with unfilled polyketone with respect to flow behavior, processing characteristics, compound color and mechanical properties.

2 Experimental

2.1 Neuburg Siliceous Earth

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

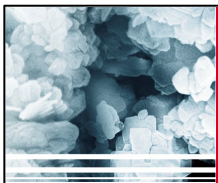




During calcination, Neuburg Siliceous Earth is subjected to a heat treatment. The components and the thermal process lead to a product that offers special performance benefits as a functional filler.

2.2 Fillers and their characteristics

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



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Fillers and Characteristics

HOFFMANN MINERAL

Filler	Description	Functionalization
Aktifit AM	Calcined Neuburg Siliceous Earth d_{50}: 2 µm, d_{97}: 10 µm	Amino
Aktifit PF 115	Calcined Neuburg Siliceous Earth d_{50}: 2 µm, d_{97}: 10 µm	Amino

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Aktifit AM is an activated Calcined Neuburg Siliceous Earth grade, whose surface has been modified by treatment with an amino functional group.

For Aktifit PF 115 also an amino functional group was used for the surface modification.

2.3 Compounding and molding

The starting point of the study was the polyketone (PK) M330A, a grade with medium flow behavior of the Hyosung company (in South Korea). The mineral fillers were loaded at 30 weight percent.

The compounding was made in a Werner & Pfleiderer twin-screw extruder ZSK 30 (screw diameter 30 mm).
In the compounding operation, the polyketone 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 60 °C (residual moisture <0.04 %).

The polyketone granules were injected with a melt temperature of 245 °C at a mold temperature of 80 °C.

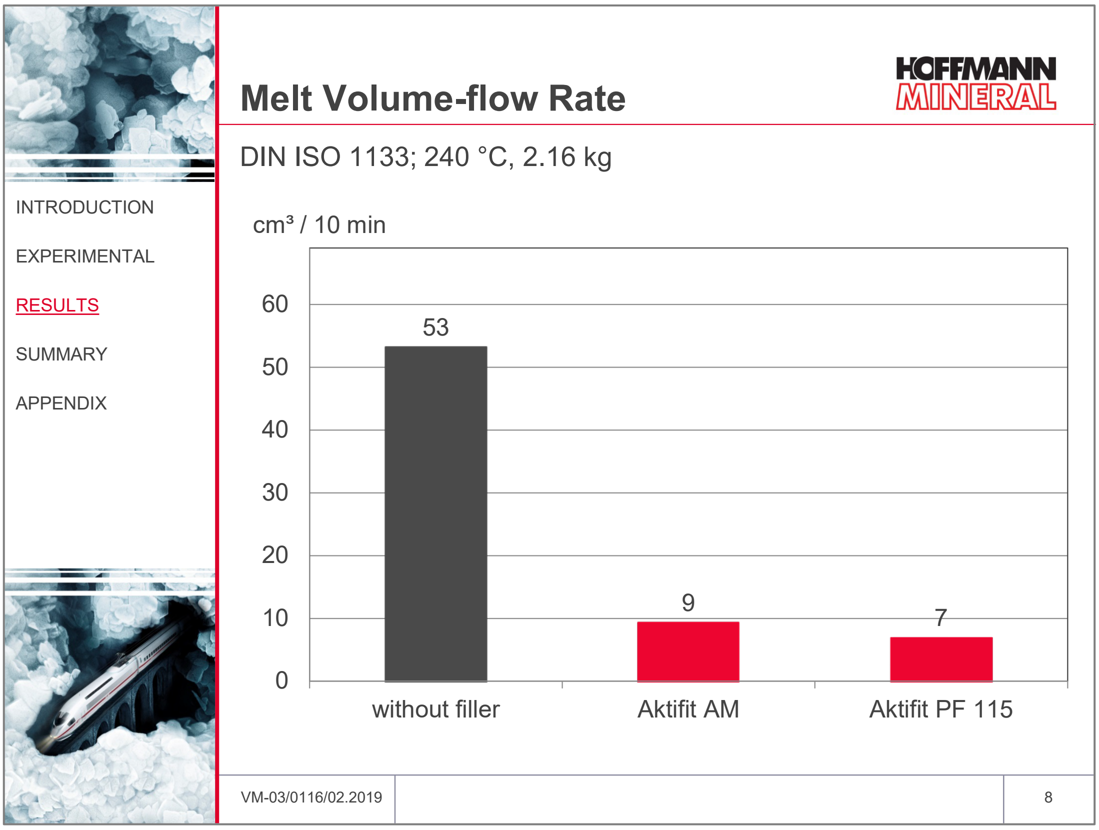
Compounding, injection molding and subsequent tests were carried out at A. Schulman in Kerpen, Germany.

3 Results

3.1 Flowability

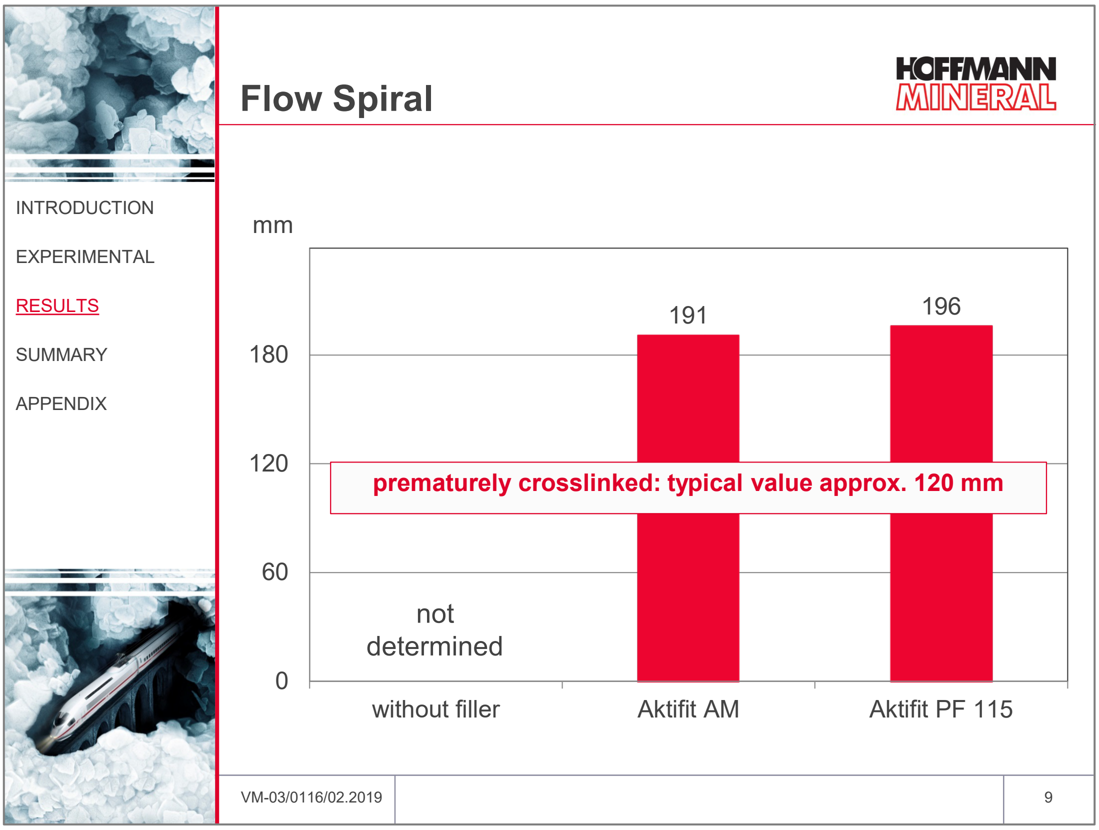
Melt volume-flow rate

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



The filler-loaded compounds exhibit a markedly lower volume-flow rate compared with the unfilled polymer. However, they can be extruded without giving rise to premature crosslinking problems.

Flow spiral

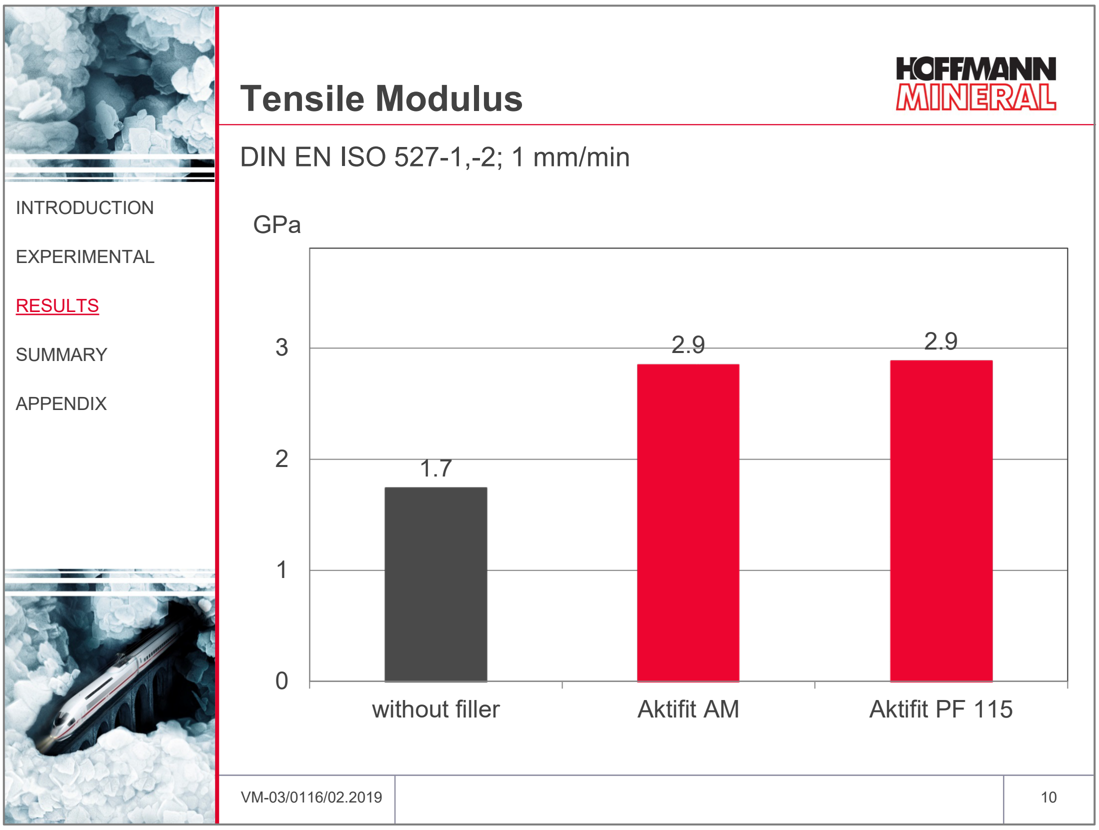


The flow path obtained gives an indication of whether or not a compound already starts to crosslink during extrusion. Typical results for prematurely crosslinking compounds with the mold in use are >120 mm. The flow distances with the two Neuburg Siliceous Earth grades of around 190 mm underline that the compounds can be extruded satisfactorily and without premature crosslinking.

3.2 Tensile test

Tensile modulus

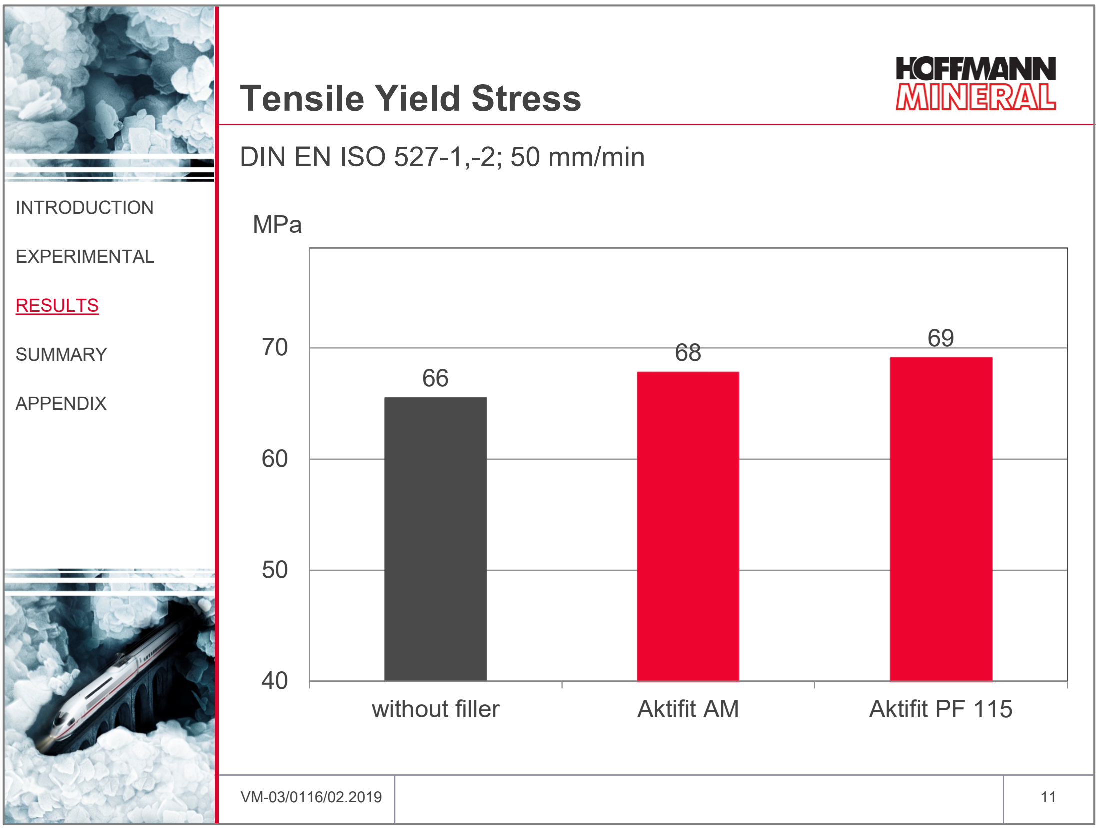
In place of the stiffness of the material, the tensile modulus was determined in a tensile test at an extension rate of 1 mm/min.



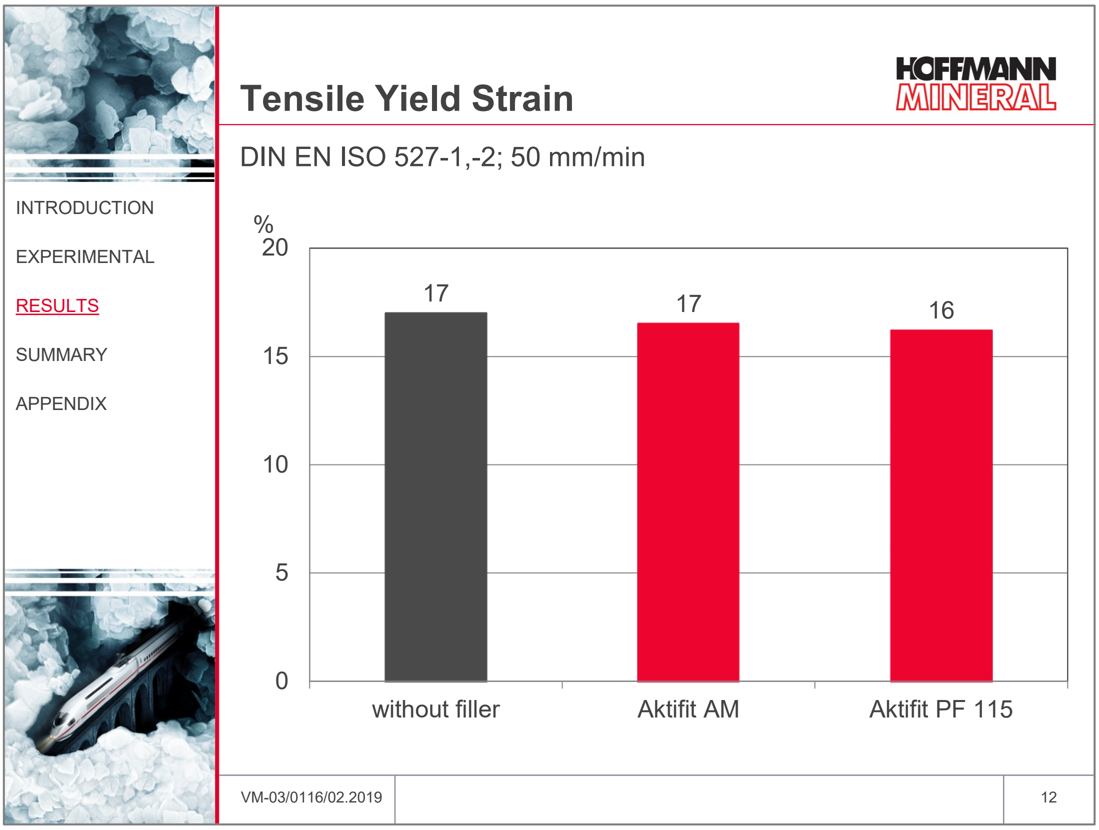
The compounds loaded with the Siliceous Earth grades, according to expectations, exhibit a stiffness higher by 70 % in comparison with the unfilled polymer.

Yield stress and yield strain

The test was run with specimens type 1A at an extension rate of 50 mm/min up to break.



The addition of fillers gives rise to a minor increase of the strength level.

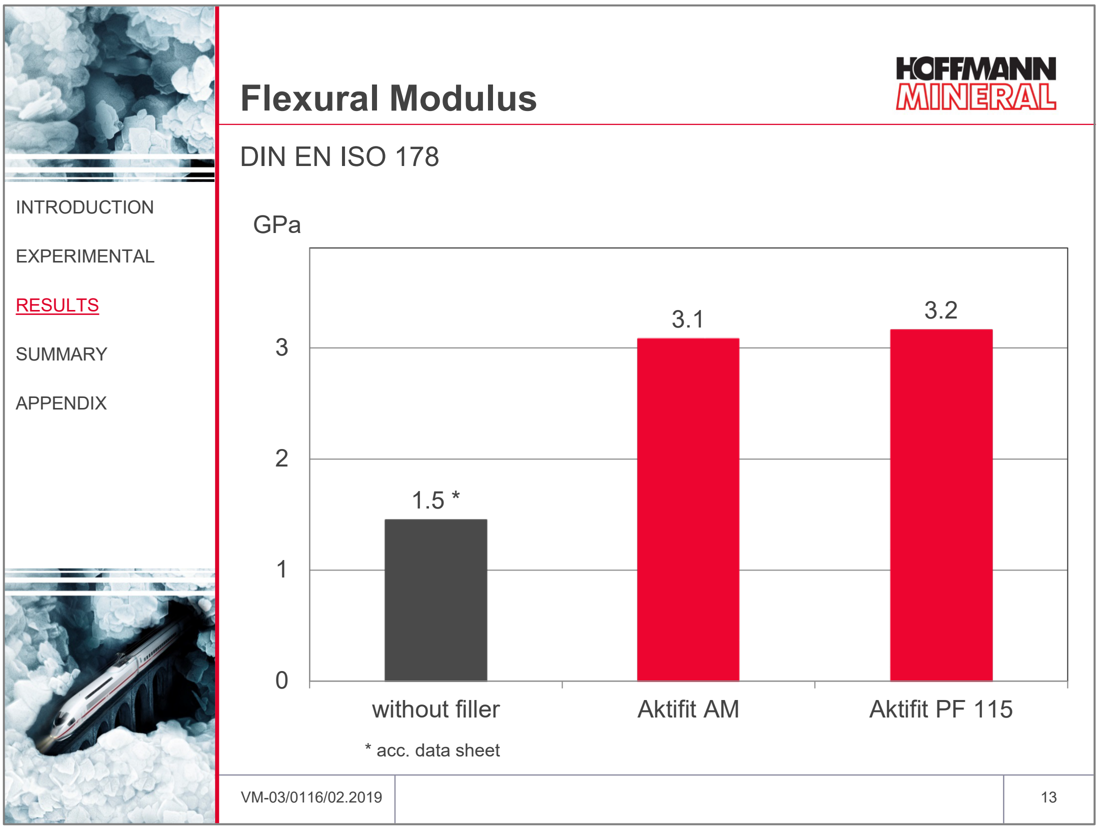


With respect to yield strain, there arise no negative effects of the mineral fillers, as the two compounds loaded with Neuburg Siliceous Earth grades show the same yield strain as the unfilled polymer.

3.3 Flexural test

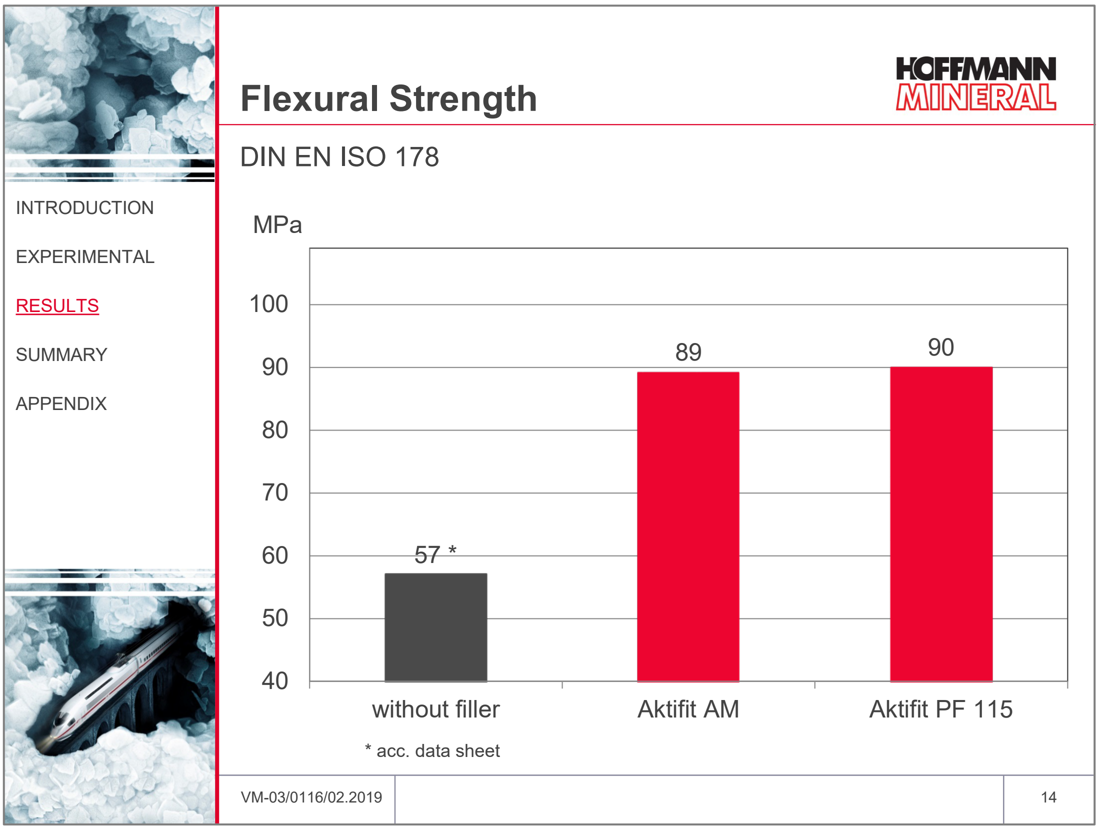
The 3-point bending test was carried out in accordance with DIN EN ISO 178.
Basically the bending test leads to similar results as the tensile test.

Flexural modulus



The unfilled polymer has with 1.5 GPa a significantly lower stiffness than the two filled compounds. Between the two Neuburg Siliceous Earth fillers, no remarkable difference can be noted. The flexural stiffness comes off slightly higher than the stiffness of 2.9 GPa obtained in the tensile test, and thus the resulting increase tends to be somewhat above the 70 % increase of the tensile modulus.

Flexural strength



Also the flexural strength is significantly higher for the filled compounds than the unfilled compound, without having a noticeable difference between the two Neuburg Siliceous Earth fillers.

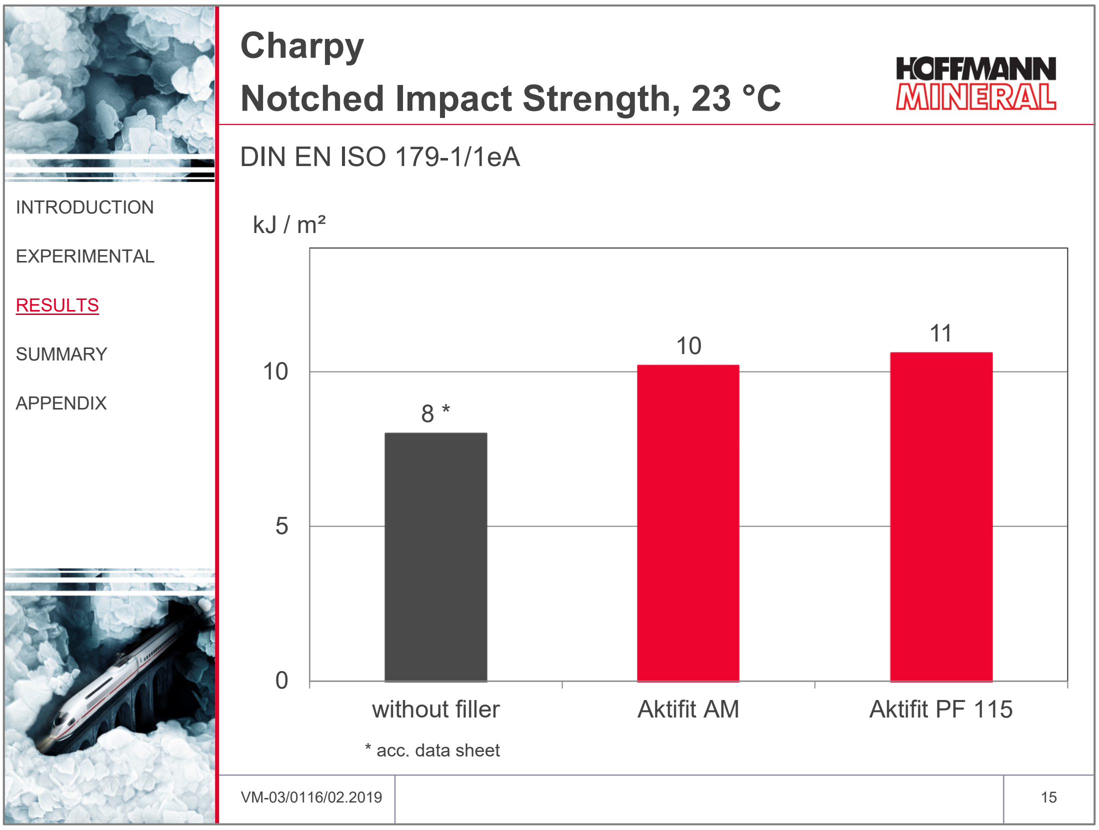
The flexural strength, however, comes out markedly higher than the yield stress measured in the tensile test, and is improved with the two Neuburg Siliceous Earth grades by more than 50 % compared to the unfilled compound.

3.4 Impact strength Charpy

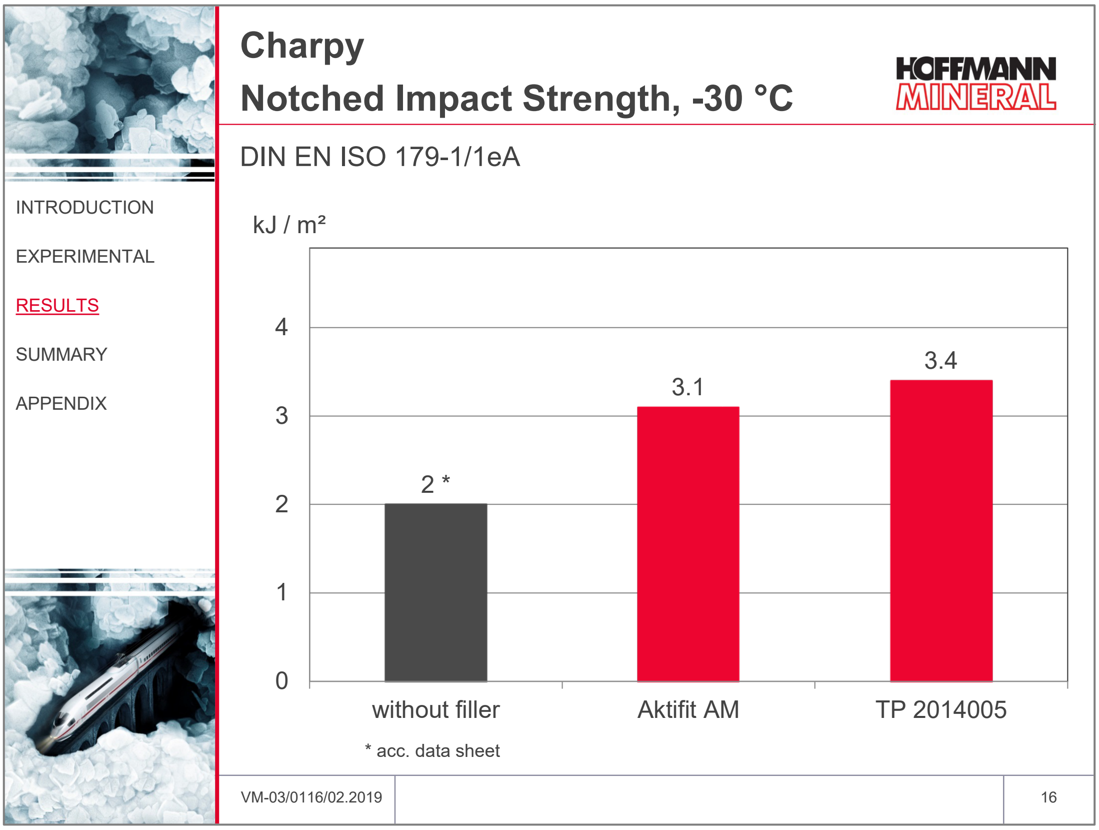
According to the Charpy method, the sample is supported unclamped at both ends and hit in the middle with a pendulum hammer.

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).



The notched impact strength will be increased by the addition of fillers from 8 kJ/m² to 10 kJ/m².



Even at low temperatures, there is an increase in the notched impact strength by the filler. Aktifit PF 115 tends to slightly higher values.

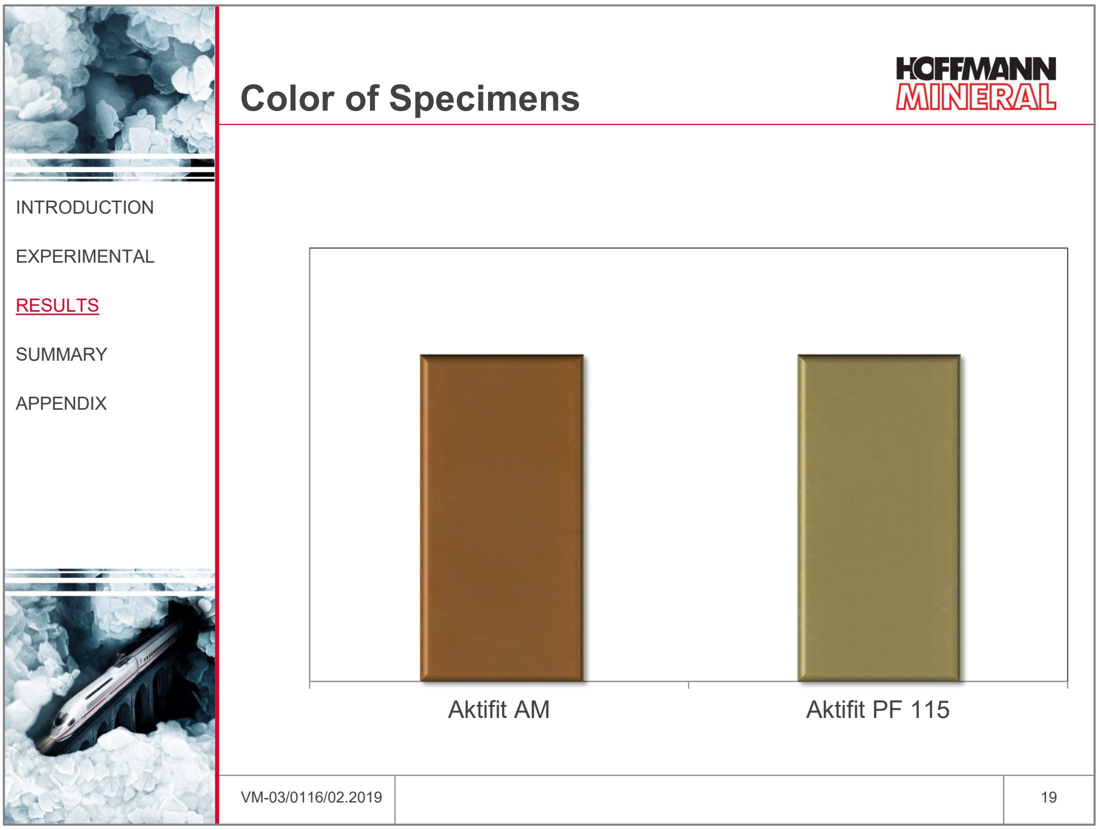
Unnotched impact strength

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.
The test was carried out with the generally used 4 J pendulum, which allows differentiating impact strength results up to 100 kJ/m².

During the tests at ambient temperature, the samples did not break at all, and surprisingly the same was true even at low temperature (-30 °C). A differentiation between the unfilled polymer and the filler loaded compounds is not given.

3.5 Color of compounds

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



The color with Aktifit PF 115 is distinctly brighter in comparison with Aktifit AM. There was no image available of the unfilled polymer.

4 Summary

Aktifit AM and **Aktifit PF 115** are attractive mineral fillers for polyketone, because they do not cause premature crosslinking during processing.

Compared to the unfilled polymer they show the following properties:

- Lower melt flow rate
- Higher stiffness
- Slightly higher tensile yield stress at comparable tensile yield strain
- Markedly higher flexural strength
- Similar or slightly higher notched impact strength
- Good unnotched impact strength: no break with 4 J pendulum, even at low temperature
- Brighter, more color-neutral compounds with Aktifit PF 115

Among the Neuburg Siliceous Earth grades, Aktifit AM and Aktifit PF 115 are the best choice for polyketone with respect to melt stability, deformation behavior and impact strength at ambient and low temperatures. Stiffness and tensile strength come out higher than with the unfilled polymer.

From the experience with other polymers, further improvements can be expected with respect to scratch and wear resistance, chemical resistance and barrier properties.

5 Appendix: Summary table

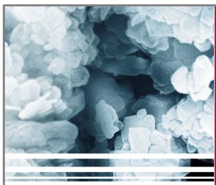


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Table of Results

			M330A unfilled	Aktifit AM	Aktifit PF 115
Melt Volume-flow Rate		cm³/10 min	53.2	9.3	6.9
Tensile Modulus		GPa	1.74	2.85	2.88
Tensile Yield Stress		MPa	65.5	67.8	69.1
Tensile Yield Strain		%	17.0	16.5	16.2
Nominal Strain at Break		%	224	21	16
Flexural Modulus		GPa	1.45 *	3.08	3.16
Flexural Strength		MPa	57 *	89.2	90.0
Impact Strength	23 °C	kJ/m²	No break	No break	No break
Charpy, 1eU	-30 °C	kJ/m²	-	No break	No break
Notched Impact Strength	23 °C	kJ/m²	8 *	10.2	10.6
Charpy, 1eA	-30 °C	kJ/m²	2 *	3.1	3.4

* acc. data sheet

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