# Calcined Neuburg Siliceous Earth

# in thermoplastics:

# Polyphenylene sulfide (PPS)


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# 1 Introduction

As a raw material, polyphenylene sulfide (PPS) predominantly finds applications for mechanically, thermally and chemically highly exposed molded articles in the areas of the automotive, machine and electrical industries.

Along with high stiffness and strength, the excellent high temperature resistance and the outstanding chemical resistance against solvents, acids and alkali solutions are important properties.

In order to achieve balanced properties, glass beads are frequently used as fillers.

Compounds loaded alone with mineral fillers, until now, have been hardly available.

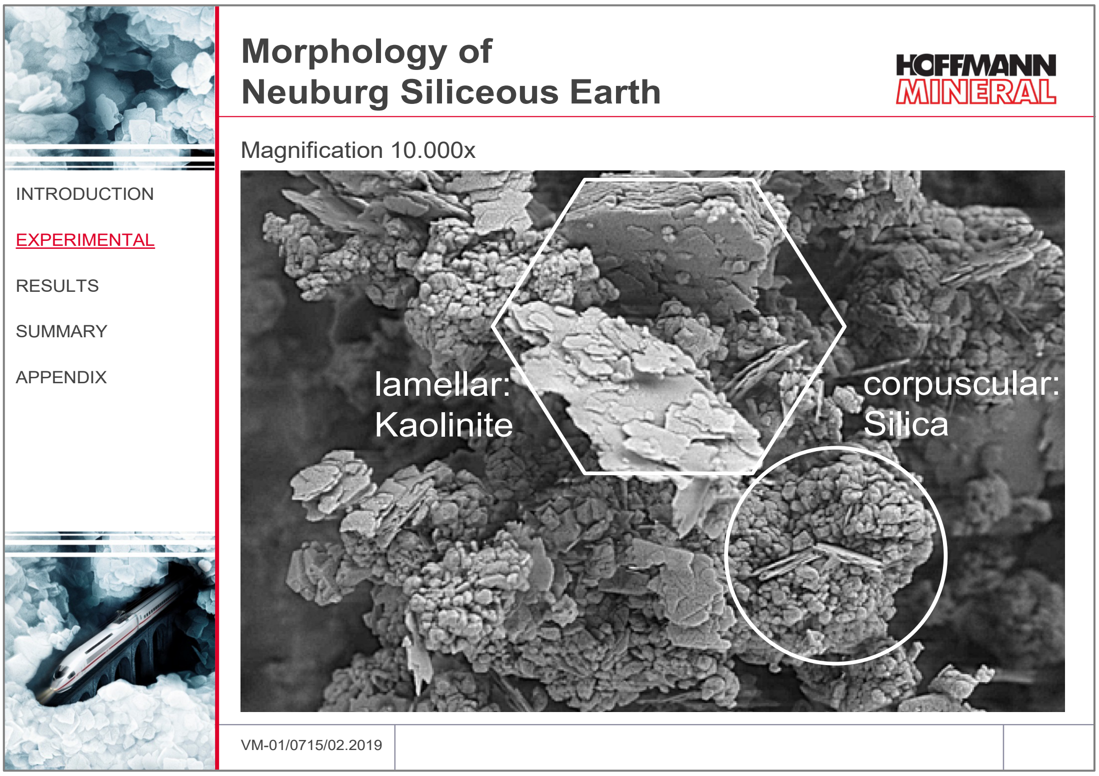
The present study has the objective to present Calcined Neuburg Siliceous Earth as a functional filler for PPS.

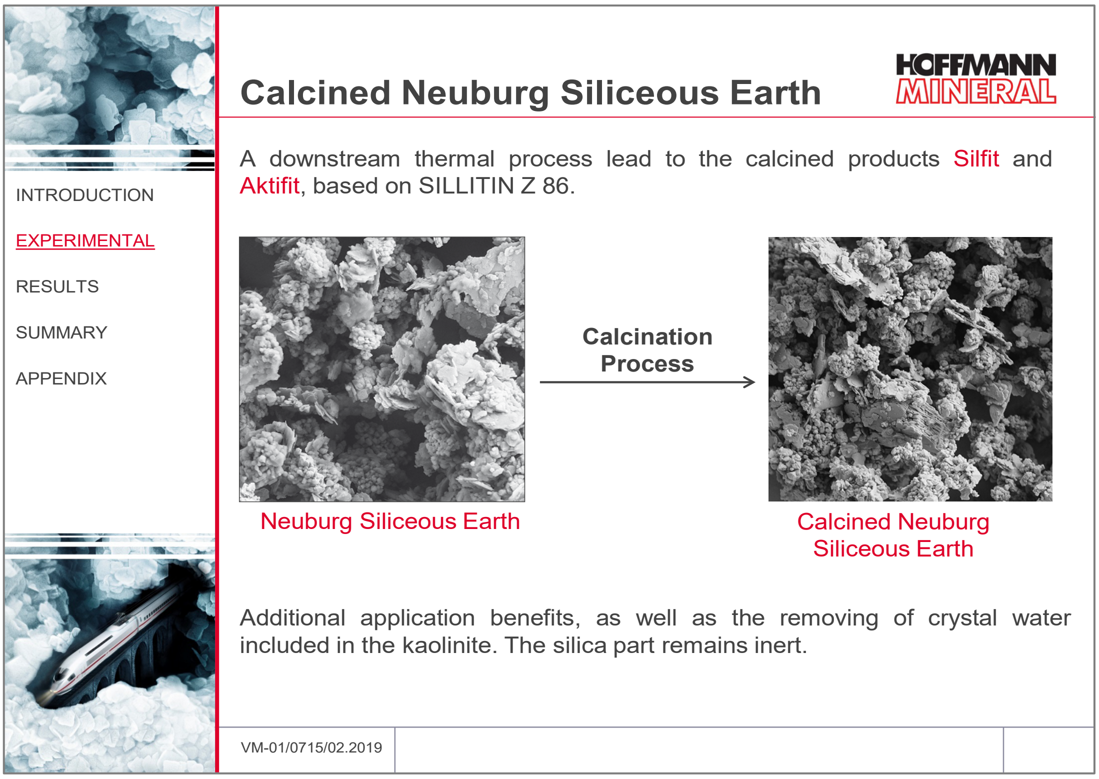
The report includes a comparison of the property profiles of Calcined Neuburg Siliceous Earth with glass beads with respect to flow properties, compound color and mechanical properties.

## 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.

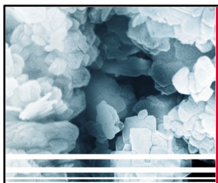



During calcination, Neuburg Siliceous Earth is subjected to a heat treatment. The components and the thermal process lead to a product that offers special performance benefits as a functional filler.

## 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 | Functionali-zation |
|---|---|---|
| Glass beads | $d_{50}$: 15-30 µm, $d_{90}$: 30-80 µm | Yes |
| Silfit Z 91 | Calcined Neuburg Siliceous Earth $d_{50}$: 2 µm, $d_{97}$: 10 µm | None |
| Aktifit AM | Basis: Silfit Z 91 | Amino |
| Aktifit PF 115 | Basis: Silfit Z 91 | Amino |

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Silfit Z 91, Aktifit AM and Aktifit PF 115 were chosen to represent the calcined Neuburg Siliceous Earth grades.

Silfit Z 91 is a Neuburg Siliceous Earth grade which was subjected to a thermal treatment. Aktifit AM is an activated Silfit Z 91 whose surface was modified by treatment with an amino functional group.

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

Comparisons were made with a grade of surface treated glass beads suitable for PPS.

## 2.3 Compounding and molding

The evaluations were made with a stabilized polyphenylene sulfide (PPS). The compounds were composed of 60 weight percent PPS and 40 weight percent 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 PPS 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.

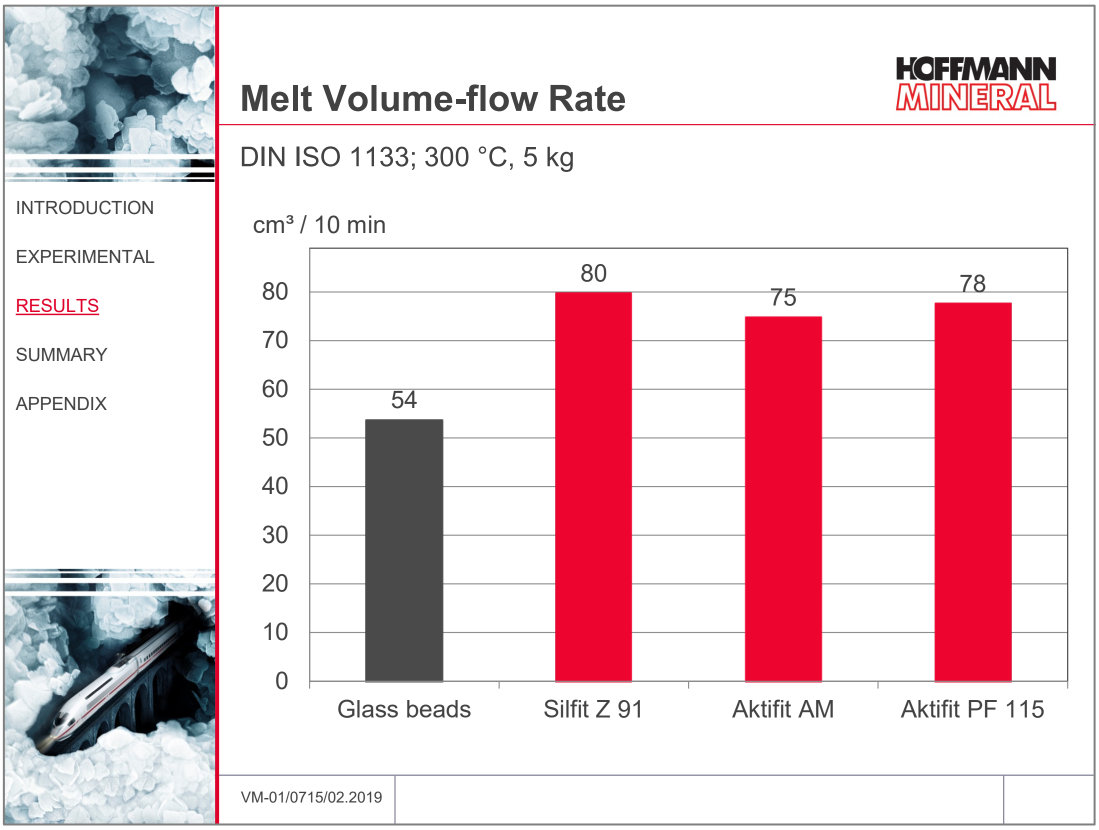
The preparation of the test specimens was made on a screw injection molding unit from Krauss Maffei, using a specimen tool according to ISO 294 with exchangeable inserts for the individual test specimens.
Prior to processing, the pellets were pre-dried for at least 16 hours in a vacuum furnace at 80 °C (residual moisture < 0.04 %).
The PPS granules were injected with a melt temperature of 315 °C at a mold temperature of 150 °C.

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

# 3 Results

## 3.1 Melt volume-flow rate

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

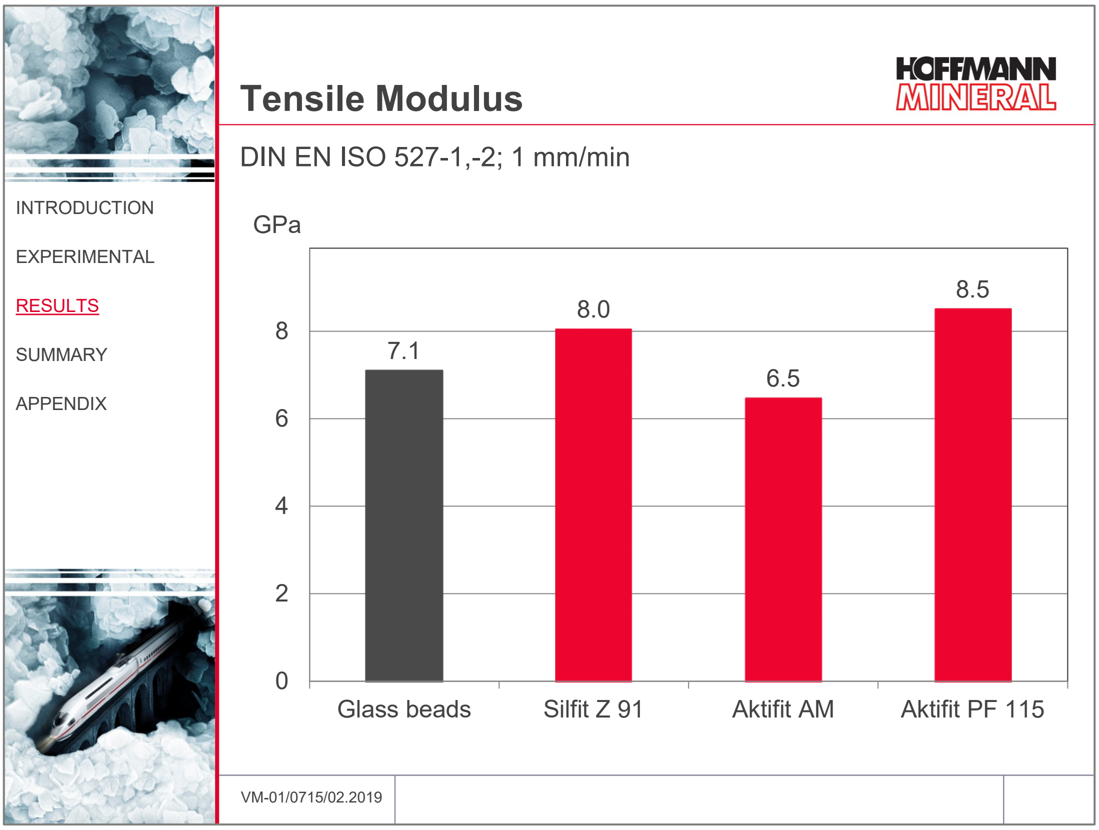



The compounds with Calcined Neuburg Siliceous Earth grades show markedly better flow properties than the compound with the glass beads.

## 3.2 Tensile test

### Tensile modulus

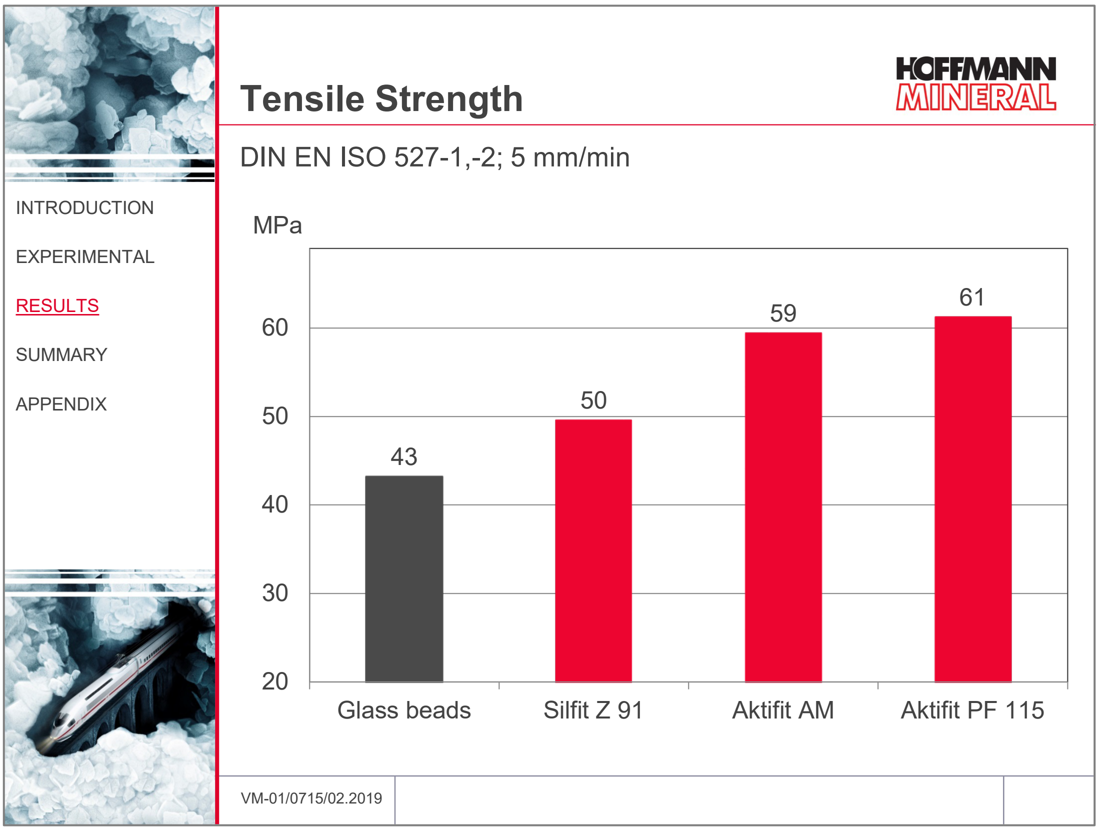
In place of the stiffness of the material, the tensile modulus was determined in a tensile test at an extension rate of 1 mm/min.



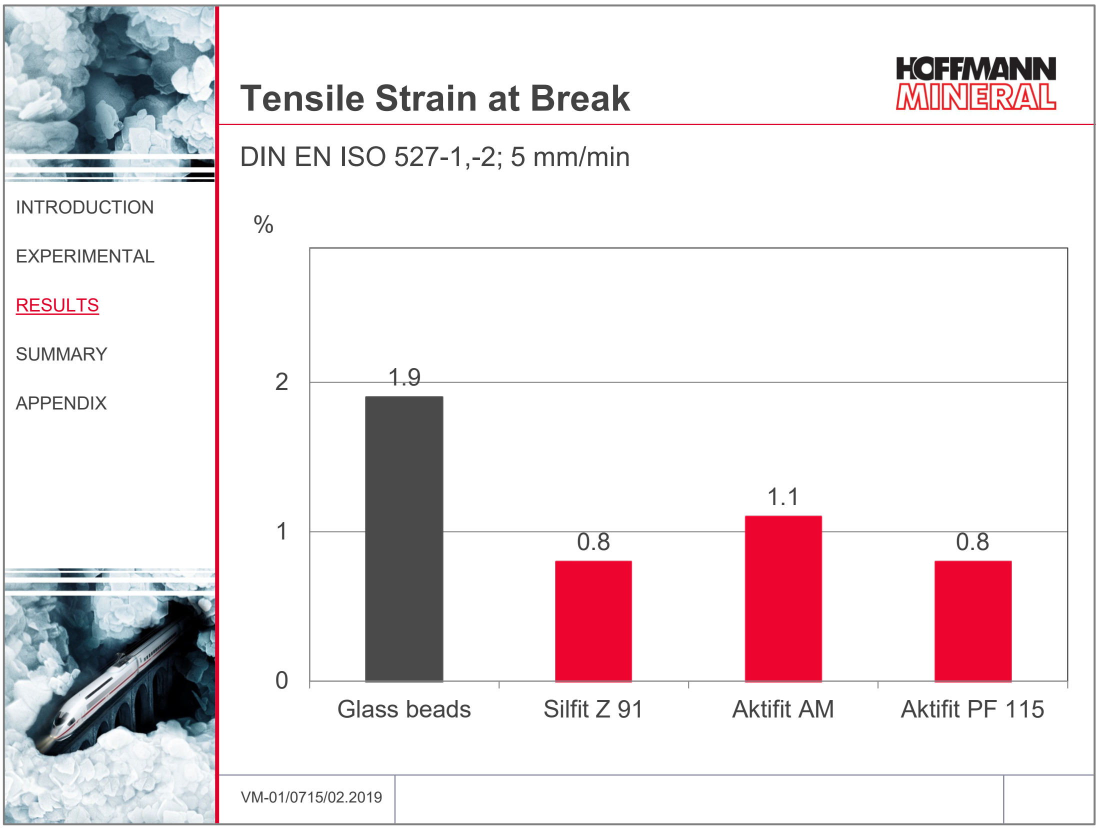

Aktifit AM gives rise to a somewhat lower stiffness. By contrast, Silfit Z 91 and in particular Aktifit PF 115 lead to a stiffness up to 20 % higher than with the glass beads.

## 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.




Already the surface unmodified Silfit Z 91 gives rise to a higher strength than the glass beads. The surface treatment with amino functional group allows to further increase the strength and thus outperforms the level with the glass beads by about 40 %.

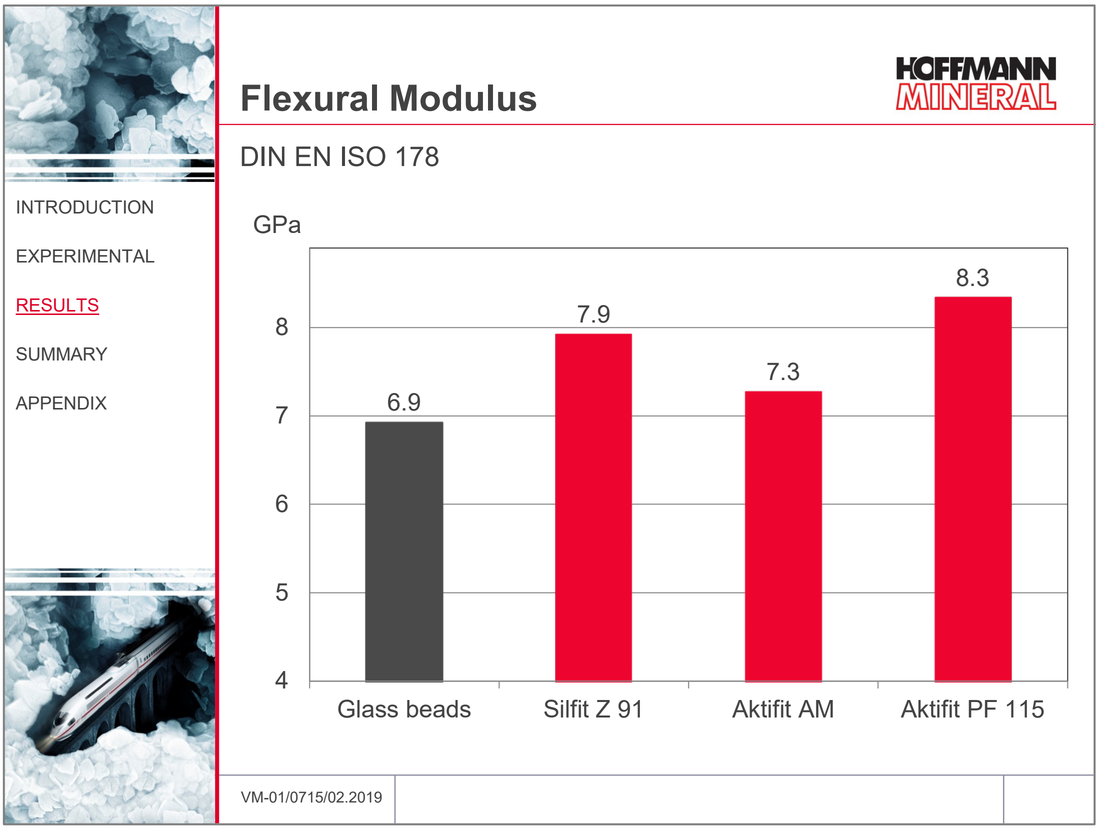



Elongation at break with the glass beads is around 2 %, with the other fillers at about 1 %.

## 3.3 Flexural test

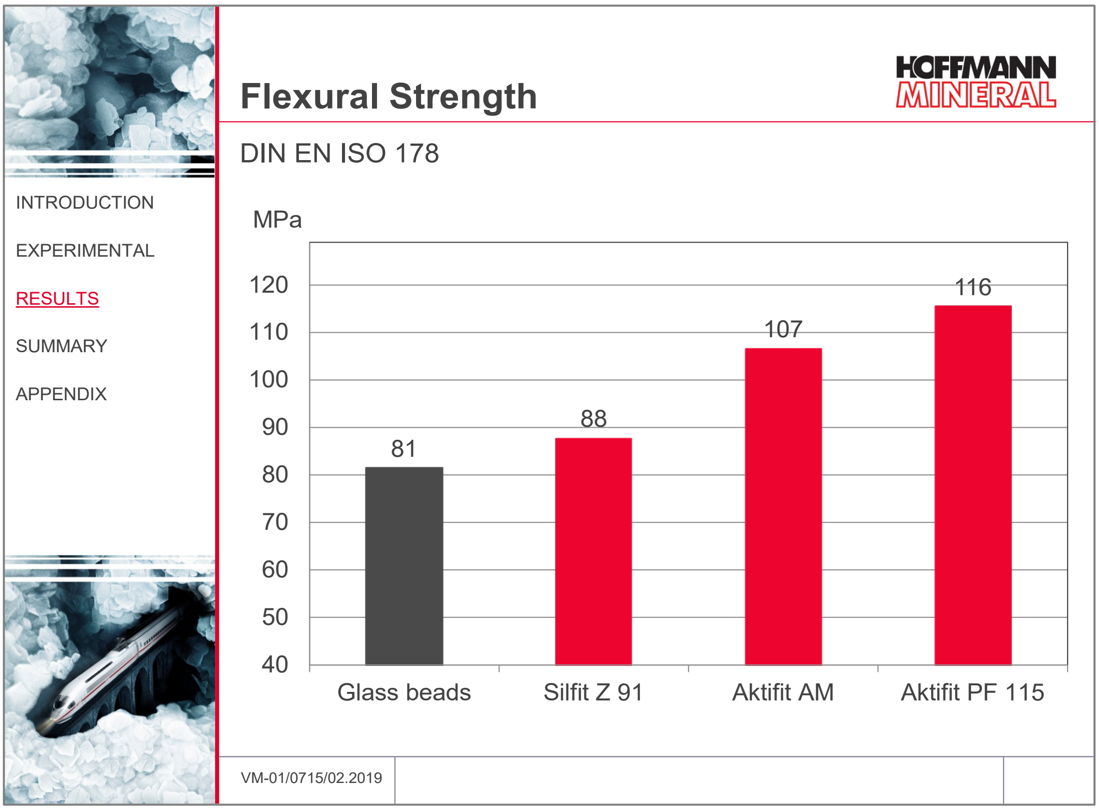
The 3-point bending test was run in accordance with DIN EN ISO 178.
Basically the bending test leads to similar results with respect to strength and elongation as the tensile test.

### Flexural modulus

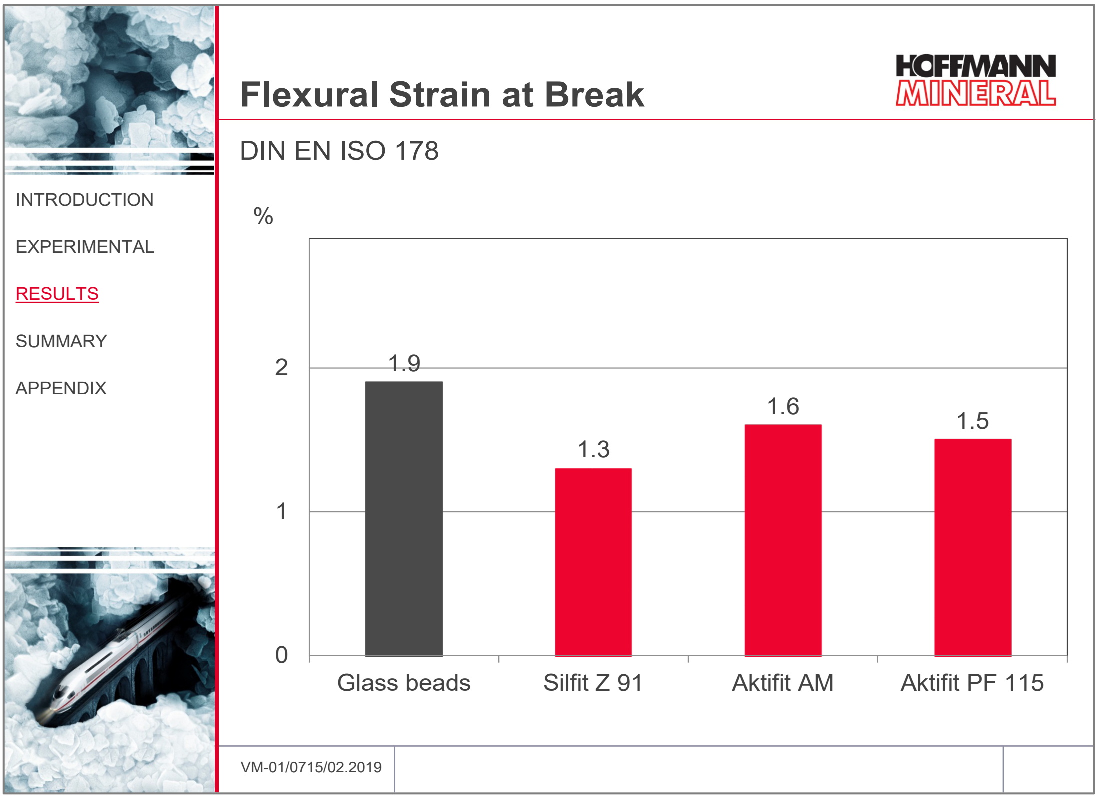



Silfit Z 91 and in particular Aktifit PF 115 lead to a stiffness up to 20 % higher in comparison with the glass beads. Different from the tensile modulus, also Aktifit AM gives rise to a slightly higher flexural modulus.

## Flexural strength and flexural strain at break



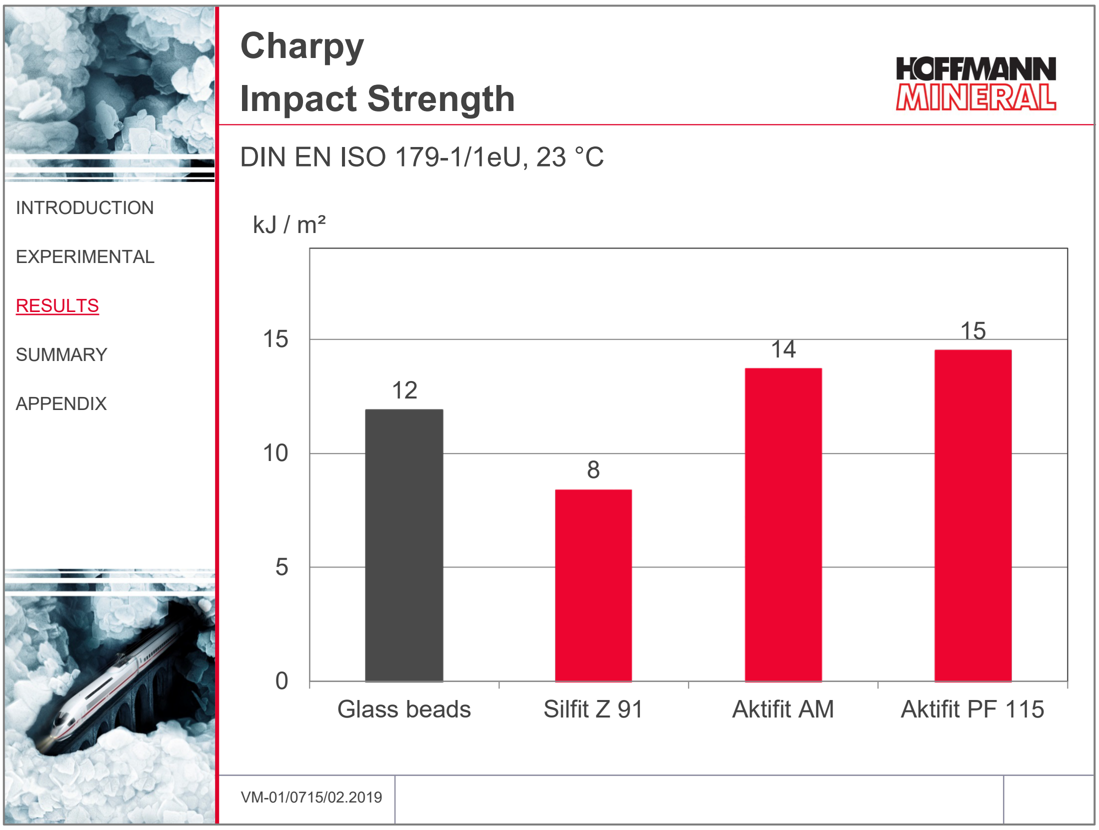

The flexural strength generally comes out at a higher level than the tensile results. Already the surface unmodified Silfit Z 91 leads to a higher flexural strength than the glass beads. The surface modification with amino functional group helps to further increase the strength which then outperforms the glass beads by up to 43 %.




Here again the elongation at break with the glass beads of about 2 % comes out on top. The difference vs. the Neuburg Siliceous Earth grades, however, is generally smaller than in the tensile tests. The amino functional group modified grades Aktifit AM and Aktifit PF 115 only give rise to a reduction by 16 to 21 %, which in view of the strength increase by 40 % looks like a good compromise.

## 3.4 Impact strength Charpy

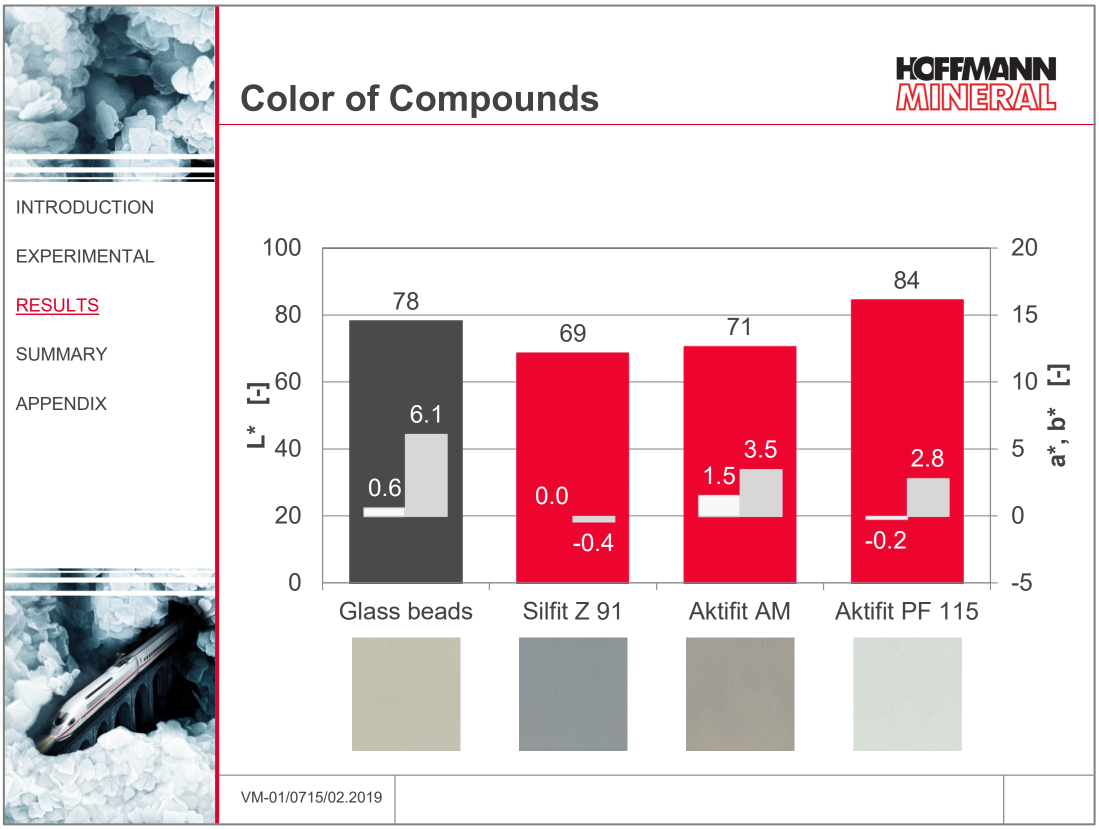
According to the Charpy method, the sample is supported unclamped at both ends and hit in the middle with a pendulum hammer. The test was run on unnotched standard samples 80 x 10 x 4 mm with the impact hitting the narrow side, i.e. the pendulum hits the 4 mm side of the sample.




The two amino functional modified products Aktisil AM and Aktifit PF 115 offer a slightly higher impact strength compared with the glass beads and Silfit Z 91.

## 3.5 Color of compounds

The graph shows the CIE-Lab color values of the compounds.
The small pictures below the graph show parts of the sample sheets, photographed under the same light conditions.

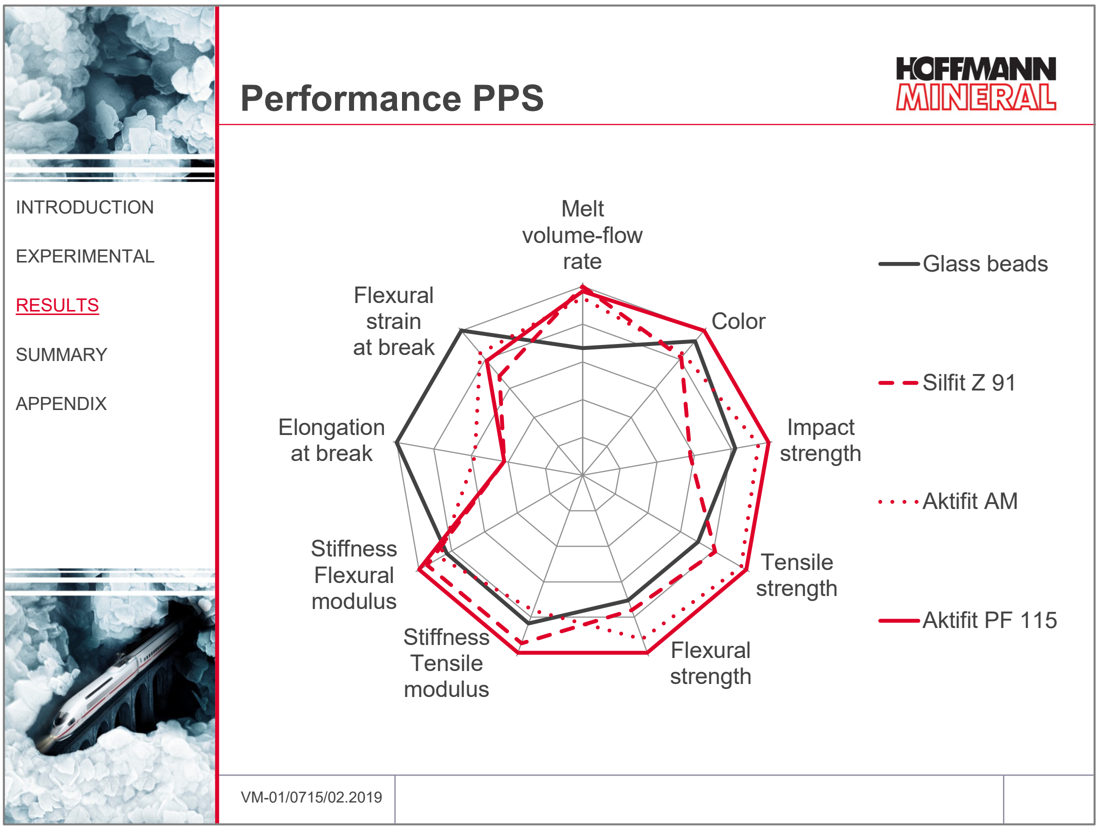



The compound with the glass beads exhibits a fairly strong yellowish tint.

It is true that Silft Z 91 und Aktifit AM give rise to a somewhat darker colored compound, however with markedly lower yellowish tint. In the case of Silfit Z 91, this almost leads to a neutral gray color.

The by far brightest-colored compound along with a low tint is obtained by Aktifit PF 115, which therefore offers more possibilities to adjust colors than the glass beads.

## 3.6 Overview: performance PPS




The glass beads give rise to the highest elongation at break and a fairly bright compound color with yellowish tint.

By contrast, Aktifit AM and Aktifit PF 115 lead to a well-balanced property profile with the best flow properties and a combination of good stiffness, tensile strength and impact strength.

Compared with Aktifit AM, Aktifit PF 115 offers higher stiffness and flexural strength and especially a very bright and neutral compound color.

# 4 Summary

**Aktifit AM** shows in PPS vs. surface treated glass beads:

- Significant higher melt flow rate
- Markedly higher strength at reduced strain at break
- Higher impact strength
- Lower yellowish tint of the compound

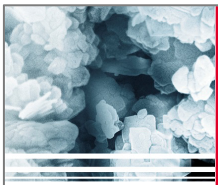
**Aktifit PF 115** offers additionally:

- Higher stiffness
- Higher flexural strength
- Brighter, almost white color of the compound

In comparison with the glass beads, the amino functional treated Calcined Neuburg Siliceous Earth grades show advantages with respect to processing properties, strength and impact resistance. Noteworthy also is the markedly more neutral color of the compounds, in particular with Aktifit PF 115.

Application areas for calcined Neuburg Siliceous Earth in PPS should be found where low warpage in combination with high attractive surface quality are just as important as good processability, high strength and impact resistance along with a neutral color of the compounds.

# 5 Appendix: Summary table



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## Table of Results

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| | | Glass beads | Silfit Z 91 | Aktifit AM | Aktifit PF 115 |
|---|---|---|---|---|---|
| Melt Volume-flow Rate | cm³/10 min | 54 | 80 | 75 | 78 |
| Tensile Modulus | GPa | 7.1 | 8.0 | 6.5 | 8.5 |
| Tensile Strength | MPa | 43 | 50 | 59 | 61 |
| Tensile Strain at Break | % | 1.9 | 0.8 | 1.1 | 0.8 |
| Flexural Modulus | GPa | 6.9 | 7.9 | 7.3 | 8.3 |
| Flexural Strength | MPa | 81 | 88 | 107 | 116 |
| Flexural Strain at Break | % | 1.9 | 1.3 | 1.3 | 1.5 |
| Impact Strength 23 °C Charpy, 1eU | kJ/m² | 12 C | 8 C | 14 C | 15 C |
| Notched Imp. Strength 23 °C Charpy, 1eA | kJ/m² | 1.4 C | 1.0 C | 1.0 C | 1.0 C |
| Color Value CIELab L* | - | 78.1 | 68.6 | 70.5 | 84.4 |
| a* | - | 0.6 | 0.0 | 1.5 | -0.2 |
| b* | - | 6.1 | -0.4 | 3.5 | 2.8 |

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