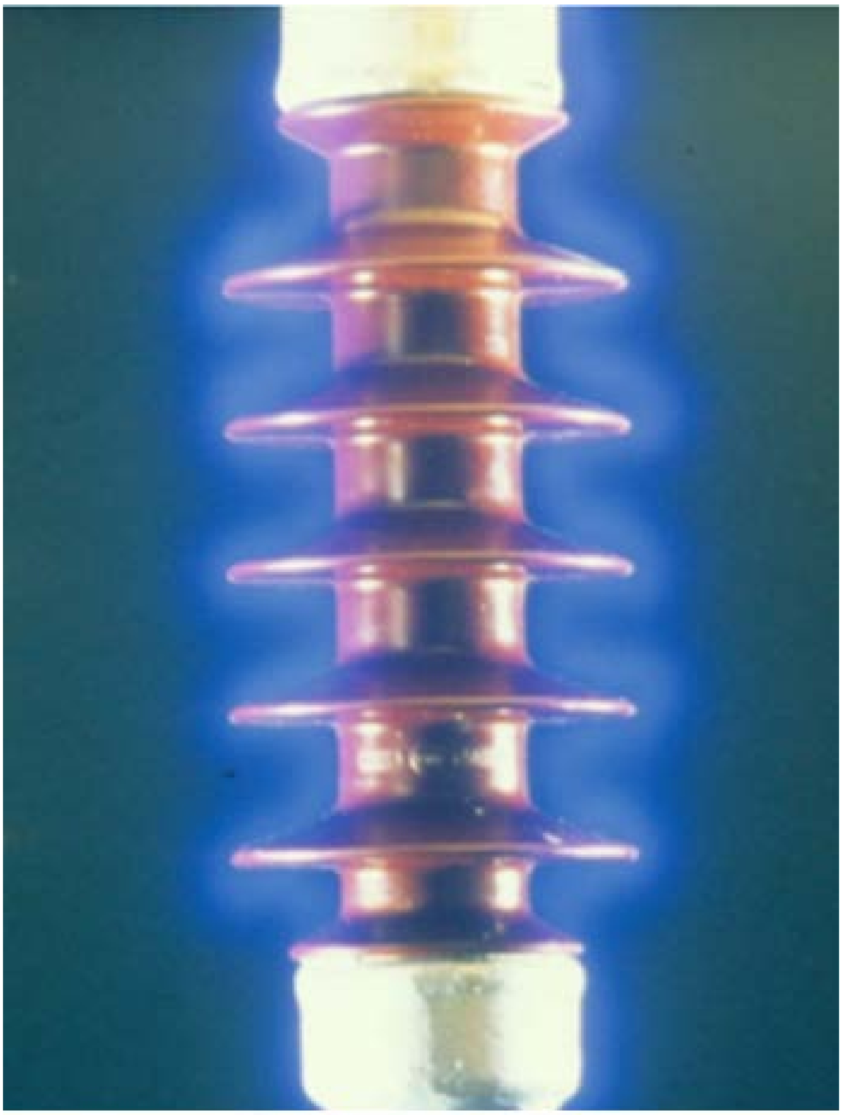

Technical Information

High Performance Fillers in Epoxy Resin for Use in Medium High-Voltage Technology

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Introduction

Ever greater requirements are being made of moulded parts with respect to the usage temperatures, electrical powers and electrical strengths, which is why improved moulding material properties are required. Functional fillers can make a considerable contribution towards the optimization of casting resin systems.

The selection of suitable fillers is of outstanding importance in the manufacture of epoxy compounds for use in medium high-voltage technology. As the requirements differ greatly, it is not a case of there being just “one” optimal filler.

The optimal integration of a filler into the resin system is achieved by silanization. Due to this the components can then perform their service without any difficulty over a period of many years, even outdoors. In addition the use of silanization leads to finished parts that are optically sophisticated as well as being mechanically and electrically more durable.

Our new product developments show very special grain size distributions. Therefore they enable lower processing viscosities by maintaining the same filling degree. On the other side a higher filling degree can influence the crack sensitivity positively.

The requisite mechanical, thermal and electrical characteristics of casting resin masses are largely determined by the functional filler that is selected. The basic physical and electrical properties of the epoxy resin compounds, manufactured with the following high-performance fillers on the basis of various mineral raw materials, have therefore been investigated in detail:

Overview of the fillers used

mineral	silica flour				fused silica	wollastonite	alumino silicate
product	MILLISIL® W 12	SILBOND® W 12 EST	MILLISIL® 126	SILBOND® 126 EST	SILBOND® FW 12 EST	TREMIN® 283-100 EST	SILATHERM® 1360-012 EST
Chemical formula	SiO_2	SiO_2	SiO_2	SiO_2	SiO_2	$CaSiO_3$	Al_2O_3 SiO_2
pH-value	7	7	7	7	7	10	6
conductivity [µS/cm]	10	10	10	10	10	300	60
medium grain size d_{50} [µm]	16	16	22	22	14	12	17
density [g/cm³]	2.65	2.65	2.65	2.65	2.20	2.85	3.60
BET [m²/g]	0,90	0.90	0.70	0.70	1	2	1

The use of mineral fillers in epoxy casting resins allows:

- thermal control during the casting process
- to obtain good mechanical properties
- low thermal expansion of the mixture
- low electrical loss (tan δ)
- cost-effective formulation

MILLISIL® W 12 - SILBOND® W 12 EST (silanized)

The high performance fillers **SILBOND®** silanized silica powder and **MILLISIL®** silica powder have proven their worth in epoxy resin systems for several decades. One reason that they are used is on account of their excellent mechanical properties and the other is that they make an important economic contribution.

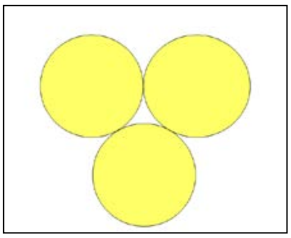
On account of the excellent physical and chemical integration of surface-modified silica powder (**SILBOND® W 12 EST**) into the epoxy polymer system this has long since been the standard for weather-resistant outdoor applications. **SILBOND®** silica powder is nowadays also increasingly being used for indoor cast resin parts.

High-performance fillers on a silica basis are characterized by the following properties:

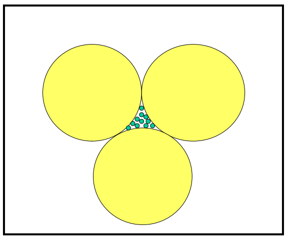
- Angular particles
- High degree of hardness of 7 (Mohs)
- High weathering and chemical resistance
- Low coefficient of thermal expansion: $14*10^{-6}K^{-1}$ (at a temperature of 20 – 300°C)
- Relatively good thermal conductivity: 9 W/m*K
- Good electrical insulating properties (low tan delta)
- High mechanical strengths
- Good degrees of filling and excellent processability
- increased filling degree of 3 – 5 % with constant viscosity
- reduction of shrinkage and thermal expansion due to lower resin part
- the mechanical and electrical properties remain unchanged

SILBOND® 126 EST is an advancement of the established surface treated silica flours **SILBOND® W 12 EST** and **SILBOND® W 6 EST**. In addition **SILBOND® 126 EST** features an increased filling degree due to the optimized grain size distribution with a constant viscosity.

pattern: SILBOND® W 12 EST



pattern: SILBOND® 126 EST



Fused silica SILBOND® FW 12 EST (silanized)

The outstanding property of fused silica is its extremely low thermal coefficient of expansion. In the case of epoxy casting compounds it is imperative for high-quality electrical engineering applications.

- Hardness of 6.5 (Mohs)
- Chemically inert
- Extremely low coefficient of thermal expansion 0.5 $*10^{-6}K^{-1}$

TREMIN® 283-100 EST, silanized wollastonite flour

TREMIN wollastonite is particularly suitable for crack-sensitive applications and is characterized by the following features:

- good electrical properties
- low thermal expansion
- very good reinforcing properties

SILATHERM® 1360-012 EST, silanized alumino silicate

SILATHERM® based on a naturally occurring alumino silicate is mainly used to improve the thermal conductivity of thermosets and offers the following advantages:

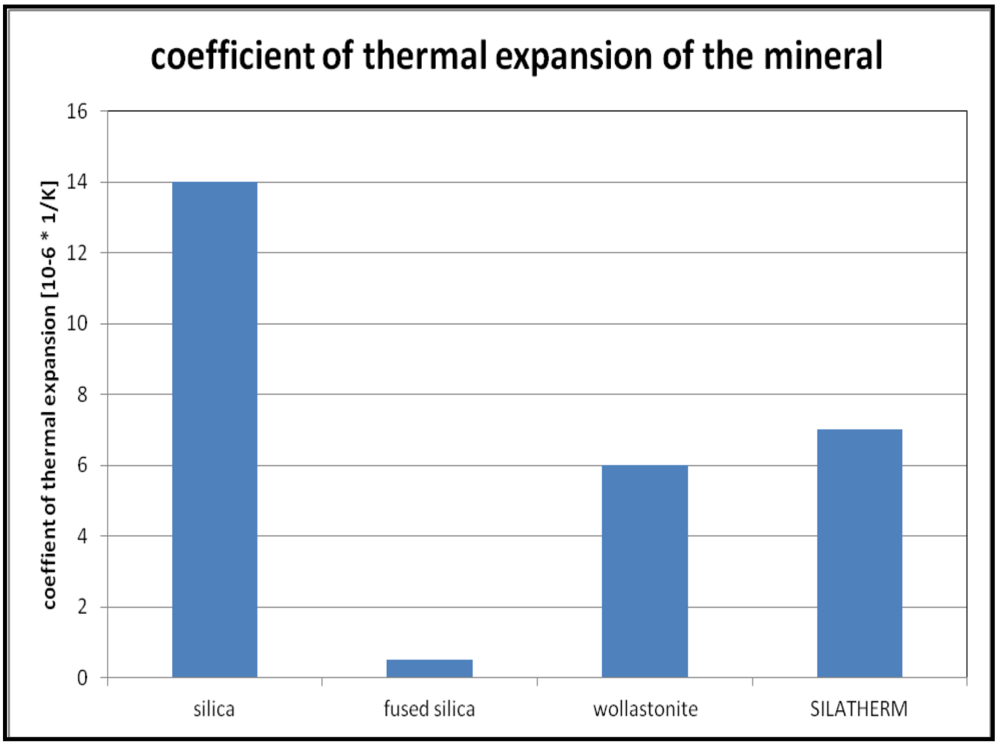
- heat-resistant
- high thermal conductivity (up to 2W/mK in optimized compounds)
- chemically inert

Thermal expansion of minerals

In electrical engineering the epoxy casting compounds and the metallic material are exposed to an intensely alternating thermal load. The dimensional changes to the various materials that are due to temperature must be as equal as possible so that no damage is incurred by the component. Epoxy resin is characterized by a coefficient of thermal expansion of $65*10^{-6}K^{-1}$, while metal has a thermal elongation of between (12 and 24)$*10^{-6}K^{-1}$.

The differing dimensional changes between the casting compound and metal that are due to temperature can be minimized by using selected high-performance filters with a low coefficient of thermal expansion in epoxy casting compounds. It is these that make crack-sensitive applications (large metallic cast parts or complex geometries) possible in the first place.

The following diagram illustrates the thermal elongation of various mineral raw materials:



Advantages of surface-treated fillers

The interfaces of the polymer matrix and the filler system influence the final properties of the system vitally. If the polymer and the filler do not harmonise, the whole system is weakened considerably. The surface treatment of mineral fillers with different silanes or silane-based compounds enables optimized effects at the interfaces. An adapted silanization improves the efficiency of the whole system significantly.

Investigation results at a glance:

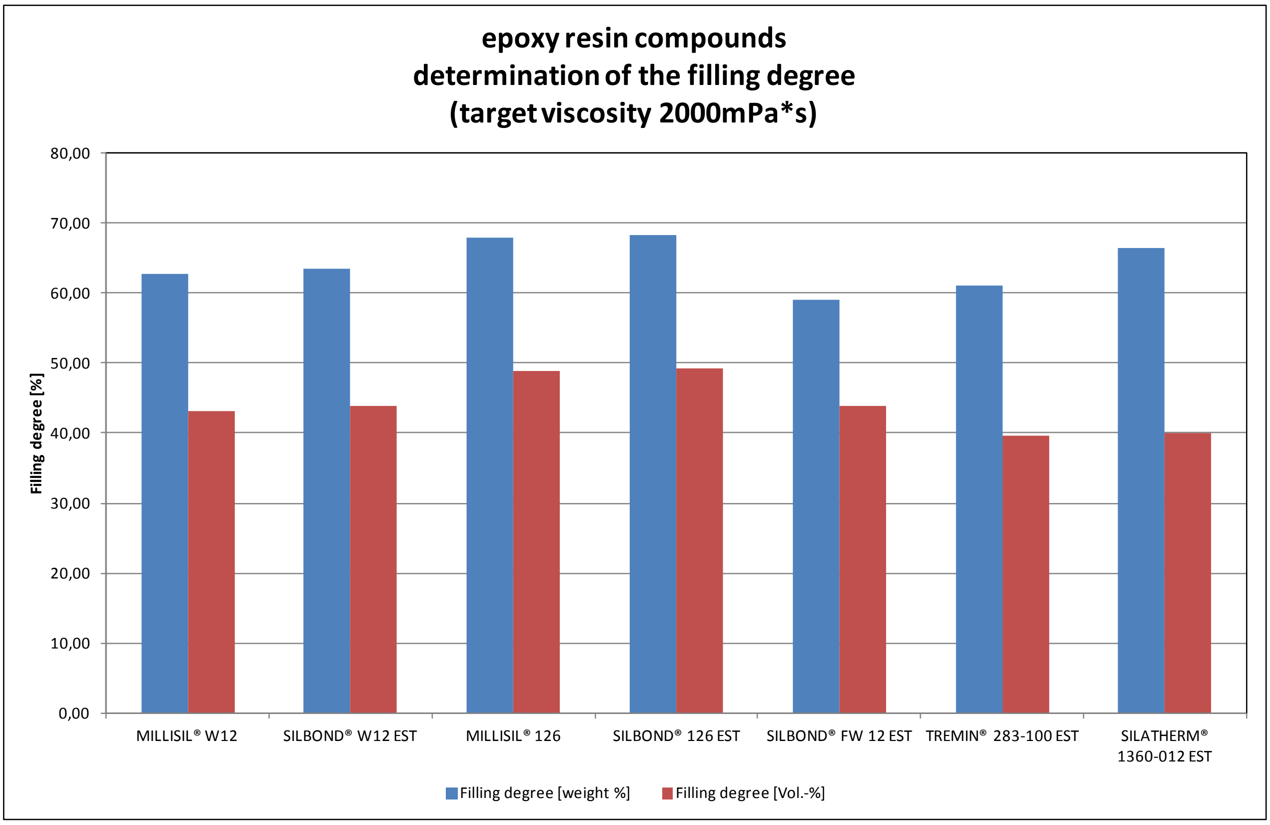
Electrical tests	MILLISIL® W 12	SILBOND® W 12 EST	MILLISIL® 126	SILBOND® 126 EST	SILBOND® FW 12 EST	TREMIN® 283-100 EST	SILATHERM® 1360-012 EST
before water immersion of 100 days							
tan δ	0.022	0.024	0.026	0.020	0.004	0.012	0.012
Permittivity ε_r	4.04	4.08	4.15	4.40	3.64	4.65	5.01
after water immersion of 100 days							
tan δ	0.434	0.059	0.430	0.051	0.019	0.528	0.305
Permittivity ε_r	11.66	4.57	10.99	4.57	3.92	21.32	14.00

Mechanical tests	MILLISIL® W 12	SILBOND® W 12 EST	MILLISIL® 126	SILBOND® 126 EST	SILBOND® FW 12 EST	TREMIN® 283-100 EST	SILATHERM® 1360-012 EST
before water immersion of 100 days							
Modulus of elasticity [MPa]	11,000	11,400	12,800	13,200	10,600	12,400	12,030
Stress of failure [MPa]	82	133	117	143	148	137	111
Breaking elongation [%]	0.78	1.30	0.98	1.24	1.58	1.27	1.09
Impact resistance (Charpy) [kJ/m²]	6.1	11.8	8.3	9.8	11.7	14.4	7.3
after water immersion of 100 days							
Modulus of elasticity [MPa]	10,800	11,500	12,100	13,600	10,900	10,500	11,600
Stress of failure [MPa]	54	119	49	118	135	78	84
Breaking elongation [%]	0.64	1.15	0.48	0.95	1.39	0.85	0.85
Impact resistance (Charpy) [kJ/m²]	4.1	9.7	2.5	8.3	10.9	6.7	5.9

Degrees of filling in processing / resin requirement

The processing behaviour of casting resins is of significant importance in practice. Excessively high viscosities lead to a considerable influencing of the production of parts. In the test that was performed it was important that the filler-resin mixtures all exhibited a similar level of viscosity. First of all the resin requirement for production of the specimens with different fillers was determined. It turned out that the volume proportion of the fillers is very similar with the same viscosities. Only **SILBOND® 126 EST** enables a higher degree of filling by volume.

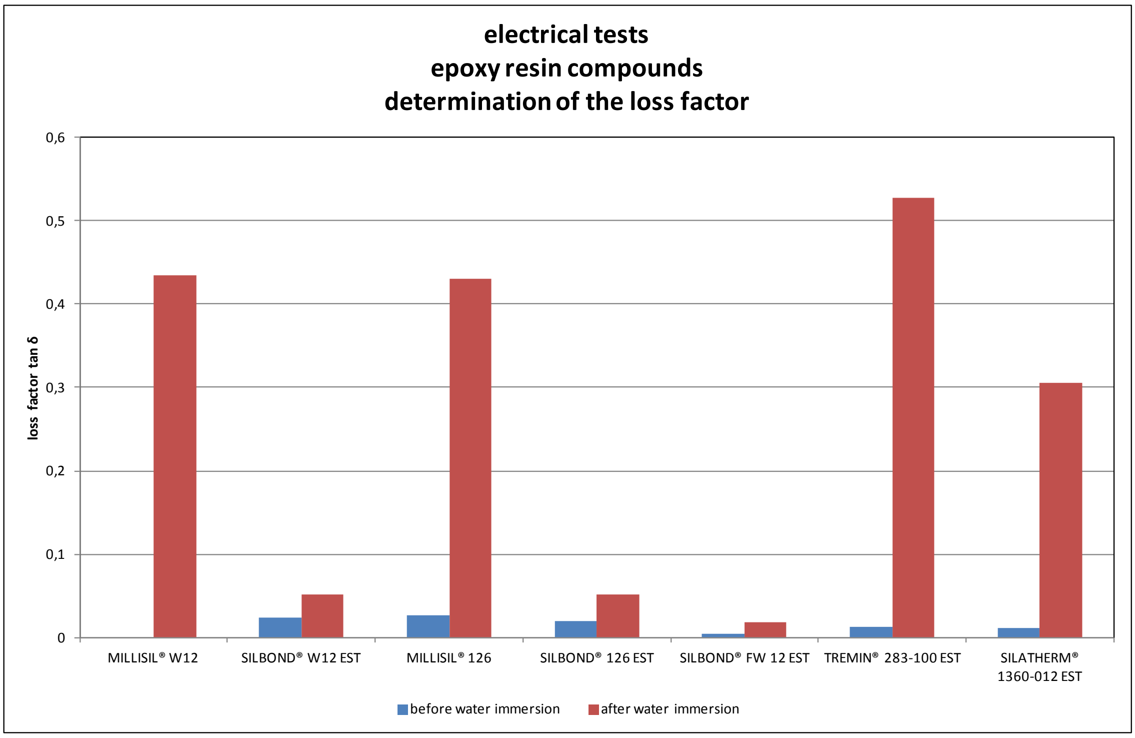
As the densities of the minerals differ and some of the tested fillers are optimized regarding the viscosity this means that there is an intensely differing proportion by weight of the fillers.



Electrical tests:

Determination of the loss factor tan δ before and after water immersion

The test specimens produced with **SILBOND® FW 12 EST** are characterized by very low electrical loss factors prior to water immersion. These are slightly higher with the other fillers used. The results after water immersion showed the excellent quality of **SILBOND® FW 12 EST**, **SILBOND® W 12 EST** and **SILBOND® 126 EST**. In the case of these products the loss factor increases only slightly, while the loss factors in the case of non-coated **MILLISIL® W 12, TREMIN® 283-100 EST** and **SILATHERM® 1360-012 EST** increase very clearly.



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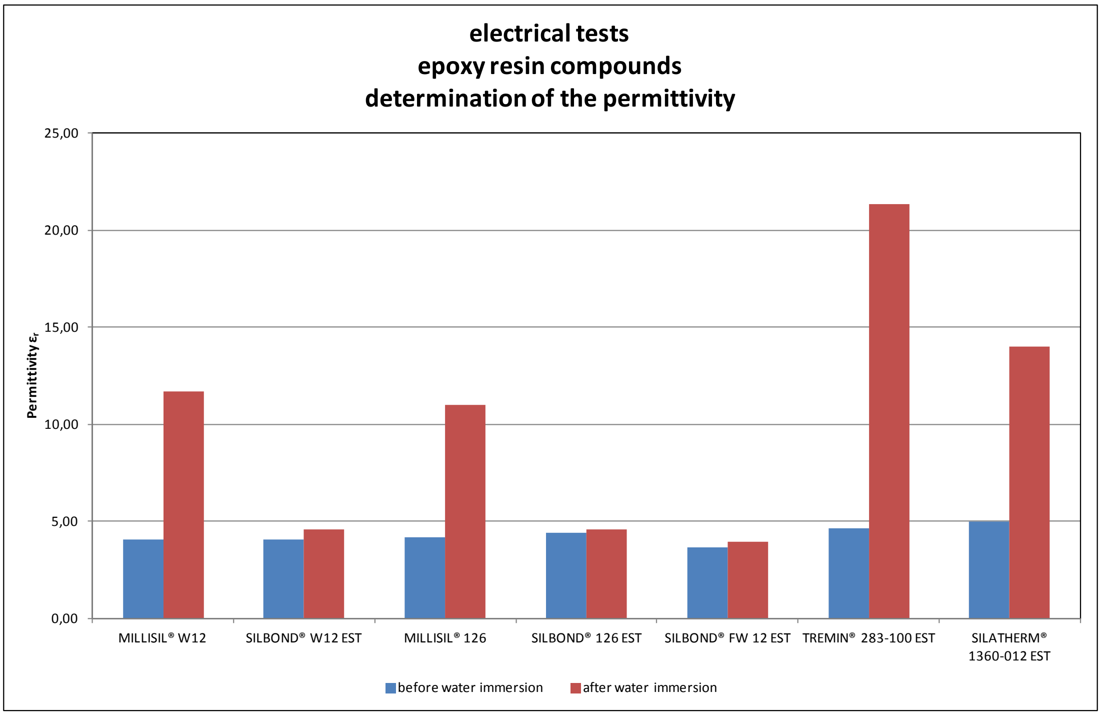
Determination of the permittivity number ε_r before and after water immersion

The permittivity number ε_r is determined above all in order to be able to estimate changes, due to hydration in the test specimens.

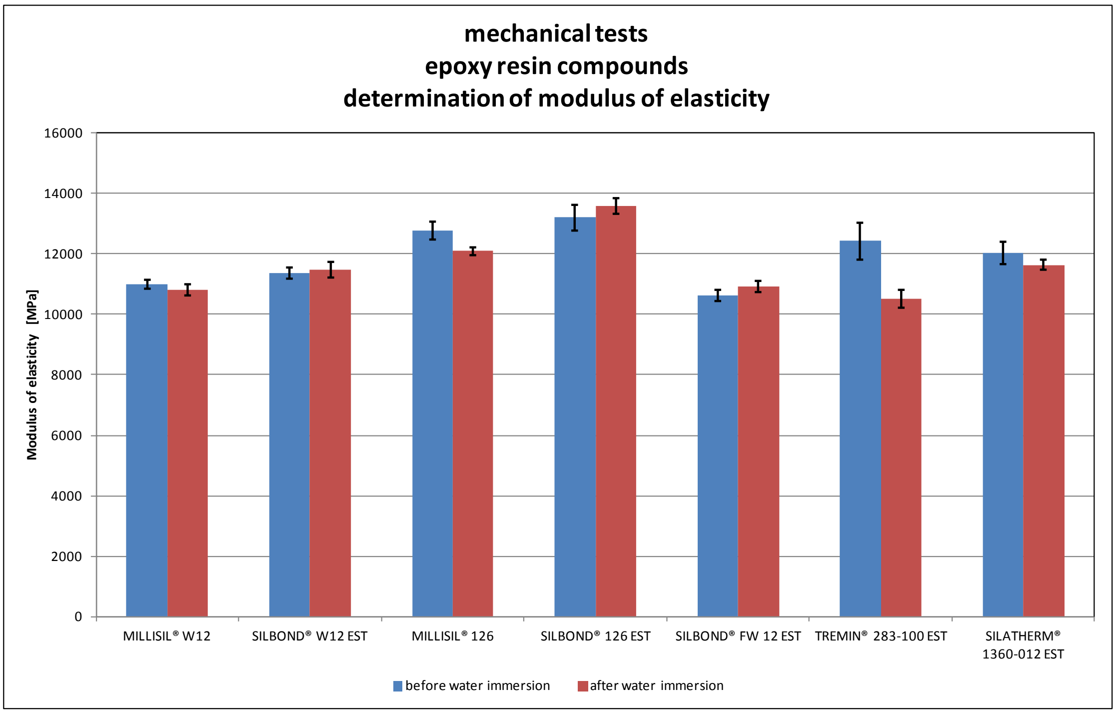
SILBOND® FW 12 EST, **SILBOND® W 12 EST** and **SILBOND® W 126 EST** show pronounced strengths in this case. The permittivity measured before and after water immersion is unchanged. In contrast to this the non-coated silica powders the wollastonite and the new product **SILATHERM ®1360-012 EST** are stable to a limited degree only. The results indicate a significant degree of hydration.



Mechanical tests before and after water immersion:

Modulus of elasticity, stress at failure and breaking elongation from the 3-point bending test

The respective modulus of elasticity of the mixtures investigated is on a comparable level of approx. 10,000 – 11,000 MPa. Only test specimens with **SILBOND® 126 EST** are somewhat more rigid (approximately 13,500 MPa), and keep this level even after water immersion. This result is due to the high filling degree in the formulation. The water immersion has no mentionable influence on the modulus of elasticity of the silica-based fillers.

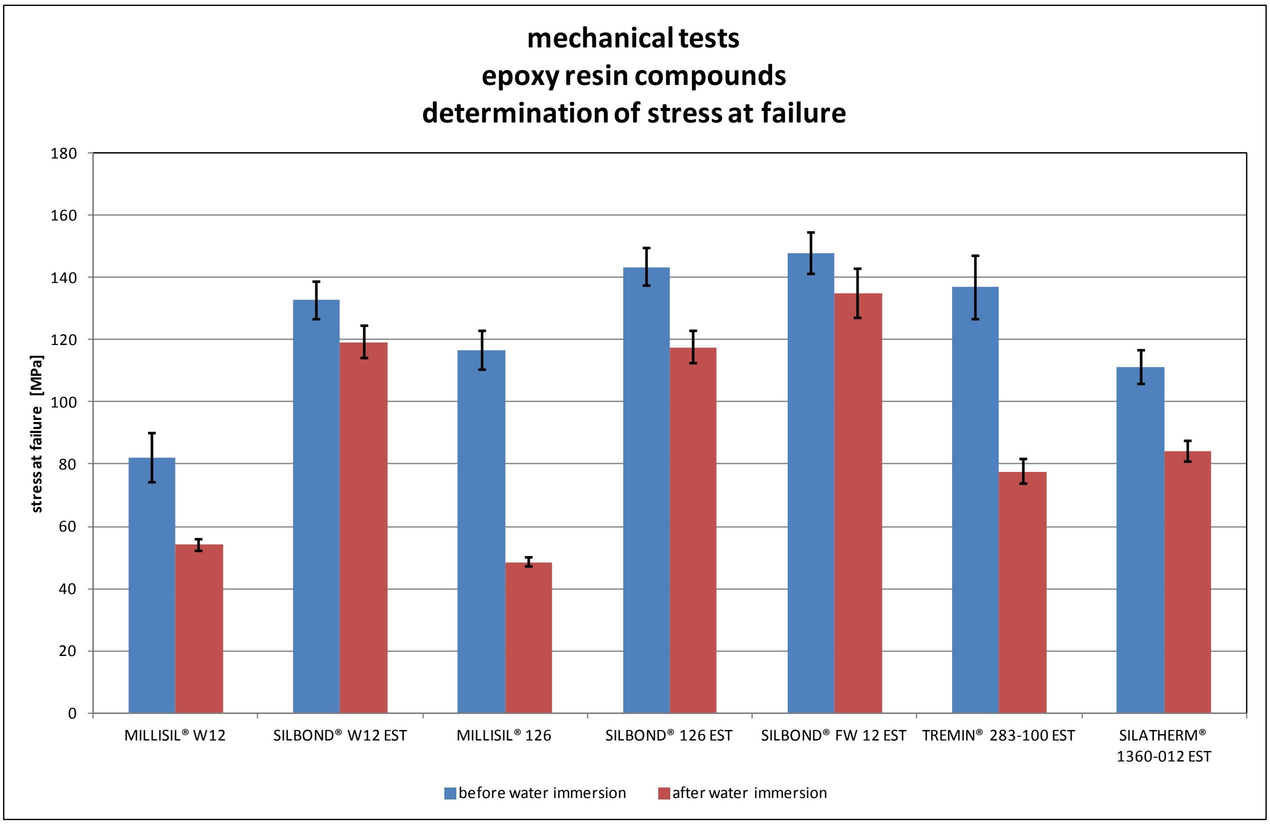


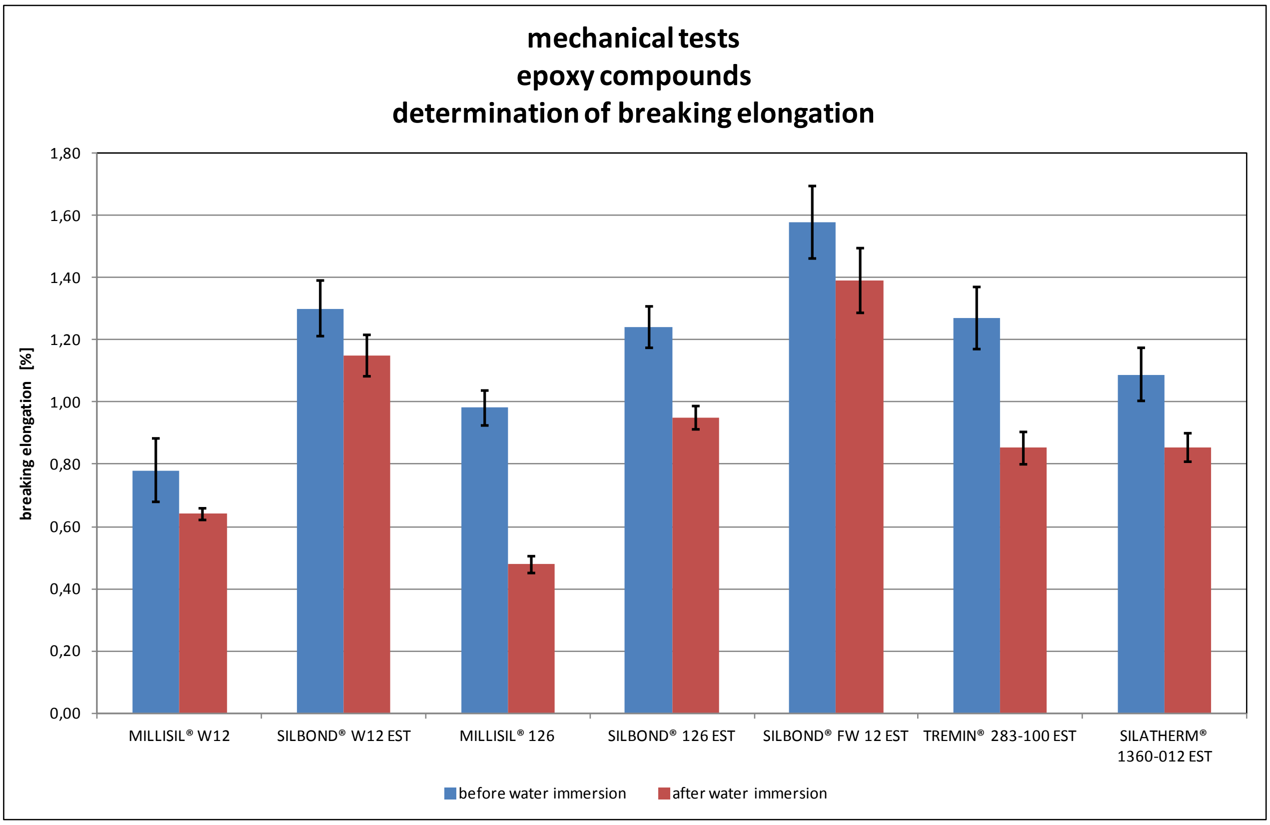
Both the stress at failure values and the breaking elongation show impressively that the silanization of our fillers results in clearly higher strengths of the moulding materials. The comparison between the non-coated **MILLISIL® W 12** and **SILBOND® W 12 EST** shows that the breaking resistance is significantly increased by silanization. Even more serious are the differences after immersing the specimens in water; while the compounds manufactured with silanized fillers survive this test without any major losses in strength, the compounds produced with non-silanized products lose approx. 1/3 of their original strength.

After water immersion only half of the strength level can be achieved with **MILLISIL® W 12** as can be realized with **SILBOND® W 12 EST**.

The cause of the better values with the silanized fillers is that the mixtures survive a much greater degree of elongation. The best mechanical properties are achieved by using grain size optimized **SILBOND® 126 EST** and silanized fused silica.

Stress at failure and breaking elongation from the 3-point bending test

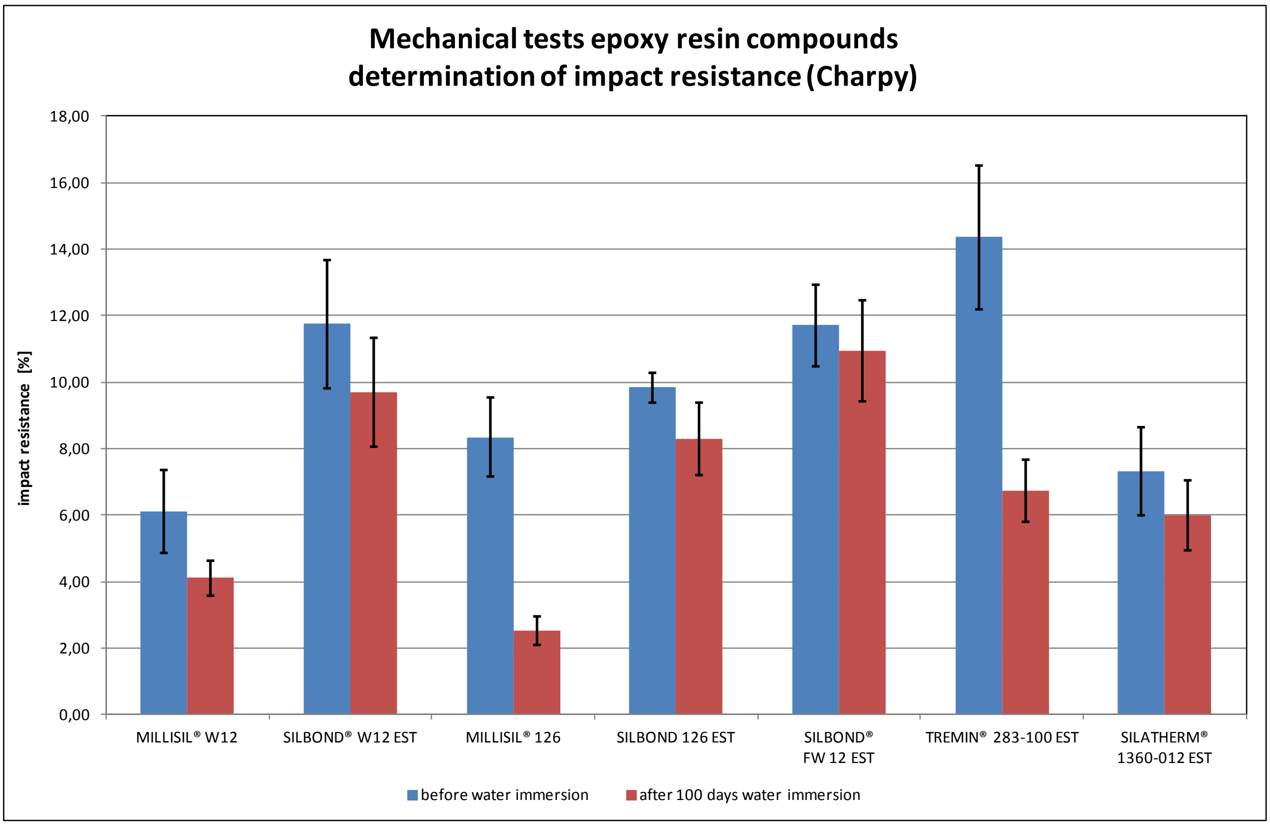




Charpy impact resistance

The impact resistance test underlines the results of the bending test.

The silanized fillers are all characterized by a better degree of rigidity than those that are not surface treated. Compounds filled with fused silica and silica powder are at a very good level. Their impact resistance is much higher than of the compounds filled with untreated silica powder. The values of **TREMIN® 283-100 EST** after water immersion decrease sharply.



Test performance

Test setup:

An Araldite casting resin system was used as the epoxy resin:

Resin:	Araldite CY 184:	100 parts by weight
Hardener:	Aradur HY 1235:	90 parts by weight
Accelerator:	DY 062:	0.6 parts by weight

The filler proportion was determined in preliminary tests in such a way that the casting resin formulations were able to be processed at 60°C with a viscosity of 2000 mPa s.

MILLISIL® W12:	320 parts by weight
SILBOND® W12 EST:	330 parts by weight
MILLISIL® 126:	405 parts by weight
SILBOND® 126 EST:	408 parts by weight
SILBOND® FW 12 EST:	274 parts by weight
TREMIN® 283-100 EST:	298 parts by weight
SILATHERM® 1380-300 EST:	331 parts by weight

The individual components (resin, hardener, accelerator and filler) were weighed separately in the weight ratios described and preheated in an oven at 60°C. The recipe components were then homogenized and subsequently degassed in a vacuum mixer. The finished casting resin was cast into metal moulds preheated to 100°C. Curing took place for 2 hours at 100°C and for another 16 hours at 140°C.

Specimen geometries:

Plate specimens with the dimensions 200 x 200 x 2 mm and 200 x 200 x 4 mm were made. The 2 mm thick plates were used unchanged for measurement of the electrical strength. The small specimen sticks (80 x 10 x 4 mm) for the purpose of measuring the mechanical properties were sawn out of the 4 mm thick plates.

Water immersion:
For the purpose of measuring the moisture resistance of the manufactured compounds the manufactured plate specimens were stored for a period of 100 days in demineralised water at a temperature of 50°C. All the electrical and mechanical tests were performed both before and after water immersion.

Test procedure:
The tests were performed in accordance with DIN VDE 0303-4. The plate specimens with the dimensions 100 x 100 x 2 mm were first of all provided with a layer of graphite for the purpose of better contact, which also compensates for the microscopic unevenness of the material.

Measurement of the mechanical properties:
The mechanical properties of the test specimens were determined in accordance with DIN 178 by means of the 3-point bending test and in accordance with DIN 179 “Determination of the impact resistance in accordance with Charpy”.