Speech at the Meeting of the Community of Tribology at 09/26/07 Göttingen

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“Impacts of different Nano powders (NanoVit®) within epoxy resin concerning the attrition”

Abstract
The influence of different mixtures of nano powders (graphite, graphite/SiO2, graphite /SiO2/Al2O3) on the wear performance of epoxy resin is shown in this paper. The filler content was varied.

1. Introduction

Polymers are more often used for products which have to bear up against tribologic impacts like bearing shells, gear wheels, layers/coats to lower the friction. Therefore epoxy resins (ER) are marked through a high thermal form and material durability as well as abrasiveness and abruption. ER is predominant against thermoplastic use especially at high temperatures and corrosive circumstances [1].

However against metals and ceramic it is inferior concerning the load and thermal steadiness. Polymers were mixed up with other internecine lubricants and reinforcing agents to conquer these disadvantages and to expand the coverage.

- The load and thermal abilities improve through an add-on of hard particles or fibres. Especially by increasing the amplification stage as well as the thermal conductivity which leads to a reduction of the temperature within the friction contact.
- Self-lubricating matters like PTFE or graphite reduce the friction coefficient. That means it lowers the shear stress on the surface as well as the attrition.

Since the last decade Nano matters were used more often to amplificade tribologic artificial compounds. The advantages against classic micro matters are e.g. producing extreme thin layers and micro mechanic components. Then it will not affect a brittleness and lower the tensile strength like it is at microscopic fillings. [2,3]

Many publications describe the positive impact of nano matters (TiO_2, Al_2O_3, SiO_2, Si_3N_4 and others) on tribologic abilities of ER [e.g.4-8]. The combination of nano matter and micro scale lubricants and amplification matters has a positive impact, too, like it was shown in new studies. [E.g. 8-14]

This speech is about the systematic wear protection of epoxy resins by filling it with graphite, nano-SiO_2 and nano-Al_2O_3 as well as other combinations and the variation of the complete volume of the filler medium.

2 Experimental Details

2.1 Producing Compounds

Epoxy compound with mixtures of different add-ons were produced in different nano-concentrations. The used resin is called Bisphenol-A-ER-resin (EEW171). The hardener was Albidur HE 600 (AEW 174) both of the company nano-resins AG, Geesthacht. The nano filler medium was NanoVit®, a mixture of plasma used graphite, a modified nano scale silicon connection (SiO_2), as well as aluminum oxide (Al_2O_3), which is provided by MSH. NanoVit® reduces under pressure and temperature the attrition and friction and forms a firmly adhering, elastic, self-regulating layer on the friction surfaces as an add-on within the oil. [15]

In order to understand the influence of the single components on the tribologic characteristics of epoxy resin, the ER was mixed with graphite, graphite and SiO_2 as well as a NanoVit-Mixture of SiO_2 and Al_2O_3. The additives were mixed with epoxy resin by 2 % Masterbatch (2 weights-% total additive part due to the resin component without hardener) and were diluted on the different concentrations. Mixtures of 0.05 wt % to 0.2 wt% nano particles were produced to a compound and hardened due to the manufacturer information. The optimal nano concentration was determined with these compounds (min. attrition rate).

The nano powder was mixed with the Speedmixer™DAC 150FVZ of the Hauschild Engineering Company (Fig.1a) into the basic resin for producing the masterbatch. It works by the principal of a dual asymmetric centrifuge. On a rotary disc under a specific angle is a rotatable arm which takes the cup with the sample material. While the disc is rotating with 3500 min-1, the cup rotates in the opposite direction. Iit was mixed 3 minutes by the highest rpm of 3500 min -1.

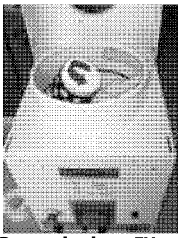

Fig.1 Speedmixer™ DAC 150 FVZ Hauschild Company

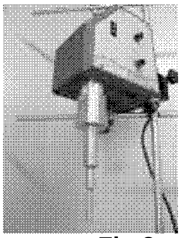

Fig.2 Ultrasound-Processor UP 400S (400W) Dr. Hielscher Company

After the mixture it was made an ultrasound by the dispersant to get an improving dispersant of the particles. Therefore the Ultrasound processor UP 400S (400W performance) with a 7mm titanium Sonotrode of the Dr. Hielscher company (Fig.2) was used. Inmittently it was done an interval of full amplitude for one minute due to the heating of the resin during the ultrasound. The Ultrasound lasted for about 10 minutes.

The air mixed into the resin mixture, due to process-relation, has to be eliminated of the resin. Therefore the mixture was evacuated for about 10 minutes. These master batches were diluted with the basic resin from 0.05 to 0.2 % and were mixed again by ultrasound (Speedmixer). Afterwards the hardener was filled in. This one was filled into under vaccoum by a magnetic stirrer.

These preparated mixtures of the cups were put into the produced silicon forms. The following hardening was done in the oven due to data of draft 1:
Draft 1: Cycle of Hardening for the Bisphenol-A-ER-resin + Albidur HE 600

Time (h)	1	1	1	4
Temperature [°C]	90	120	140	160

Experimental Practice:
The attrition experiments were done within the ball-shaped prism tribometer “tribodata” by Dr. Tillwich Company Werner Stehr due to ISO7148-2. The samples were exposed by a steady sliding movement at the tribometer. The principle of the function as well as the arrangement of prism and ball is seen in Fig.3.

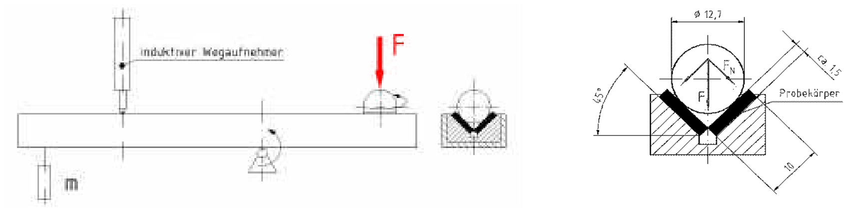

Fig.3 Function of the ball-shaped prism tribometer, Prism open angle 90° as well as arrangements of ball and prism.

The samples were stuck into the prism which is fixed at the end of a compensator. The prism is pushed against ½ inch ball through a weight which steady rotates around the vertical axis. A weight of 30 N that means a normal strength of 21.2 N per sample was used for the accomplished tests. The rpm was 1 Hz, the sliding speed at the friction contact was at app. 28.2 mm/s. Test time was 60 hours (means 6000 m sliding way). The p*v value is not constant it is getting lower due to the spherical contact geometry. The aperture of the attrition samples was 2-5mm i.e. contact pressure of 1-7 MPa, after the test. The p*v values had a maximum of 0.2 MPa*m/s at the end of the test.
The inductive position encoder recorded the data of the compensator. The attrition volume (V) was calculated by the tilt movement. The attrition volume was functionalized as the sliding way. At the stationary status it results a line, where a function was fixed to in the formula of V=V0 + w *L. The increase of this course was divided by normal strength FN to get the specific attrition rate ks:

$$k_s = \frac{w}{21{,}2\ N} \quad \left[\frac{mm^3}{Nm}\right]$$

All tests were accomplished by a normal climate (23°C; 50 % humidity). Every combination was tested three times, average of the results. Two tribometer had six compensators each, that means 12 samples could be done at the same time. The attrition surfaces were always the untreated casting surfaces.
The samples with the different nano filler mediums were tested against X5CrNi18-10. Roughness and abrasiveness of the opposite material is listed in draft 2

Draft 2: Measured Roughness and abrasiveness of the balls

Material	Roughness Rz	Abrasiveness HV
X5CrNi18-10	0.31	375

The tensile strength was done in the tensile strength test by EN ISO 527-2 with test piece 1BA (Length of the parallel part =30 mm, width 5mm, size 2 mm) by a tensile strength console of the Instron Wolpert company. For the force measurement a load cell was used with a nominal load of 25 kN. The direct strain measurement was done at the cross section of the sample. Next to the machine way the inductive position encoder was used to measure the length change of the samples and it was calculated the strain. The measured length of the position encoder was 25mm. The test piece was strained across the main axis at a constant speed of 1 mm/min until it broke.
All measured values are means of the test of 5-6 test pieces per test material.

Impact bending tests by Charpy (EN ISO 179) at non-notched test pieces were done to appraise the durability. A pendulum was used for the test with a worth of 0.981 Nm. The test pieces were in the size of 80mm x 6mm x 5mm. The states impact resistance displays the means of the test report of 5 test pieces per sample material.

3. Results and Discussions

3.1 ER resin with graphite

Fig.4 shows the attrition volume-friction way diagram of compounds with a different graphite concentration. Displayed are the average curve progression, which result from the single values out of three tests. At the beginning of the test all compounds had a balanced increase of attrition, absolute resin at the lowest, it will increase during the rest of the test and after 4000 m sliding way it has its stationary point with a very high attrition rate. The test piece with the lowest graphite content of 0.05 % performs the same way. At the beginning it has nearly the same low attrition rate. The following increase is less than the absolute resin. The sample with 0.1 % and 0.15 % have a linear increase from the beginning, while the attrition rate of these samples with the highest content of 0.2 % decreases during the process and achieves the stationary point with the lowest value of all samples with graphite.

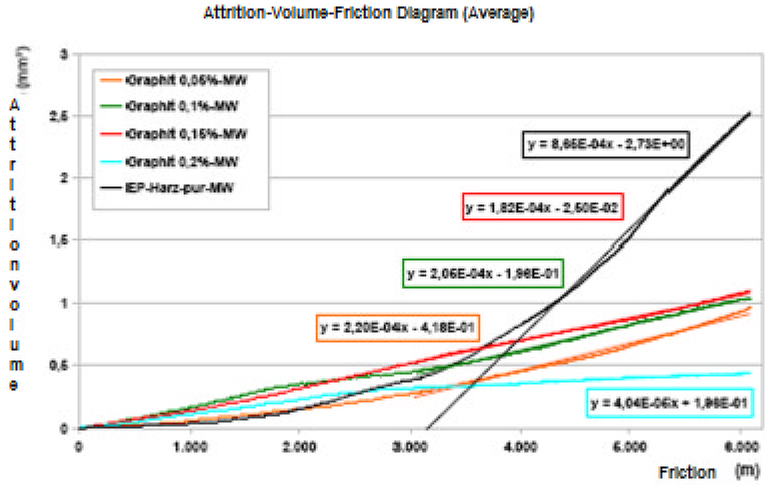

Fig. 4 Attrition volume friction way of ER resin with different graphite concentrations vs. X5CrNi18-10

The specific attrition rates (ks) of ER graphite sample, as well as the mechanic data are summarized in draft 3. The attrition rates are average values of the stationary point.

Draft 3: Attrition rate as well as mechanic data of ER resin with different graphite concentration

ER resin with graphite	k_s $[10^{-6}\ mm^3/Nm]$	a_{cu} $[mm^3/Nm]$	Tensile strength (MPa)	Elongation (%)	E-Modul (MPa)
0.05 %	1,037	12	47.18	2.48	2,770
0.10 %	967	8	54.69	3.39	2,780
0.15 %	858	8	46.76	2.39	2,926
0.20 %	190	9	53.71	2.52	3,019
Absolute ER resin	4,078	22	71.39	3.68	2,655

3.2 ER resin with graphite and SiO_2

The attrition-volume-friction-way-diagram of the samples filled with graphite and SiO_2 compared with the sampled without graphite and SiO_2 are shown in draft 5.

The filled samples are moving linear from the beginning, an increase of the attrition rate cannot be seen as it was at absolute resin. The attrition rate at stationary state is extremely low. The only exception is the compound with 0.05 %. The attrition is higher than the other samples at the beginning of the test and the curve progression is lightly progressive. But the specific attrition rate at the stationary state is below the absolute resin.

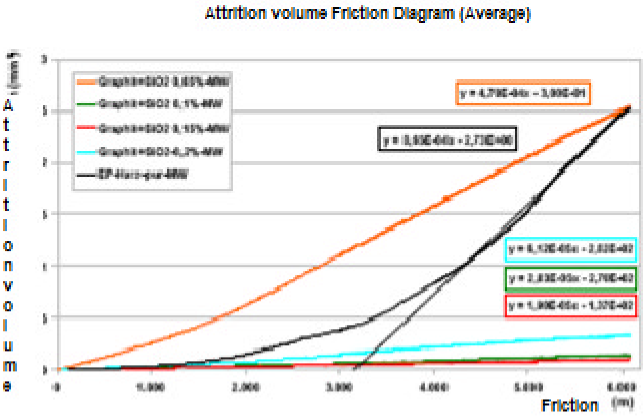

Fig.5 Attrition Volume of ER- resin with different concentrations of the mixture of graphite and SiO_2

Draft 4 compares the specific attrition rates with mechanic statistical value
Draft 4: Attrition rate and mechanic statistical value of different concentrations of filled compounds – graphite and SiO_2

ER-resin with graphite/SiO2	k [10^{-6} mm³/Nm]	a [mm³/Nm]	Tensile strength (MPa)	Elongation (%)	E-Modul (MPa)
0.05 %	2,216	11	43.89	2.08	2,756
0.10 %	133	12	71.44	4.36	2,614
0.15 %	89	11	74.54	5.75	2,433
0.20 %	288	10	64.47	3.26	2,795
ER-absolute resin	4,078	22	71.39	3.68	2,655

3.3 ER-resin with graphite, SiO_2 and Al_2O_3 (NanoVit®)

The average curve progressions of ER-resins with different concentrations of graphite/SiO_2/Al_2O_3 are shown in Fig.6.

Furthermore the curve of the filled resins moving linear and are increasing lightly from the beginning. Exception is the compound with 0.05% of NanoVit®. The attrition is relative high at the beginning. The curve progression is declining and after app. 1000 m it reached a steadiness with a low increase. After a short time all compound have a low attrition than the absolute resin.

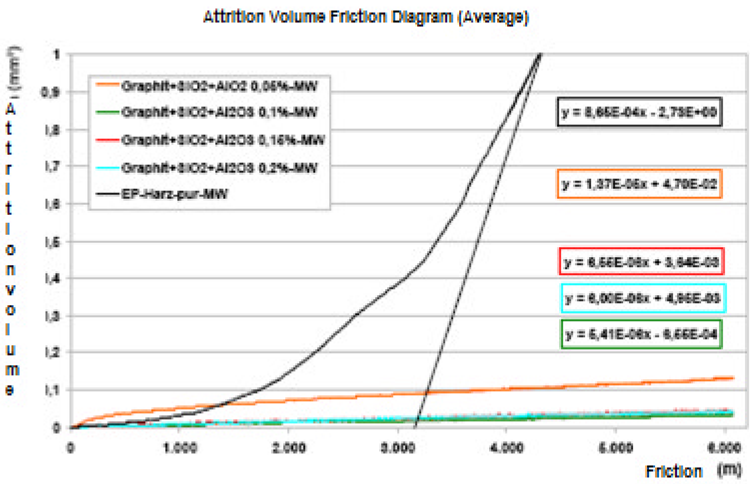

Fig.6: Attrition volume of ER-resin with different concentrations of graphite/SiO_2/Al_2O_3

Draft 5 shows a combination of the specific attrition rate and the mechanic statistical value of the filled samples

Draft 5: Attrition volume and mechanic statistical value of ER-resin with different concentrations of graphite/SiO_2/Al_2O_3

ER-resin with graphite/SiO2	k_s [10^{-6} mm^3/Nm]	a_{cu} [mm^3/Nm]	Tensile strength (MPa)	Elongation (%)	E-Modul (MPa)
0.05 %	65	11	51.84	2.69	2,267
0.10 %	26	13	73.61	5.58	2,136
0.15 %	31	12	75.56	5.69	2,330
0.20 %	28	13	57.78	2.82	3,002
ER-absolute resin	4,078	22	71.39	3.68	2,655

3.4 Comparison of the different materials

The following diagrams show the dependence of the abilities due to matter concentration and their ingredients. Fig.7 shows the attrition rates of the Compounds with the different matter concentration concerning the dependence on the complete matter concentration.

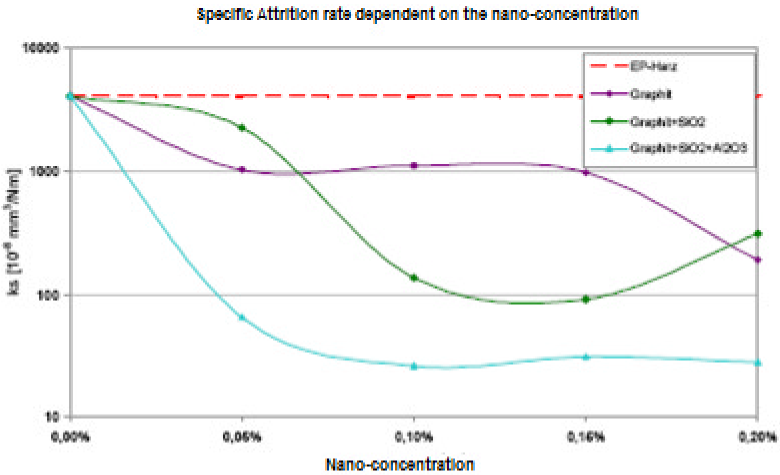

Fig.7: Impacts of the matter concentration on the different attrition rates

At first you can see that all filled samples have a lower attrition rate than the absolute resin. The matter concentration of the compounds which are filled with the NanoVit®-Mixture of graphite, SiO_2 and Al_2O_3 are two sizes smaller and lower than all tested materials.

The filled graphite causes an improvement of the wear protection with an increase of the matter concentration, while the matter mixtures of SiO_2 and Al_2O_3 have a decrease of their attrition rate at the increase of the matter concentration. It reaches a minimum at 0.1 % as well as at 0.15 % and is increasing lightly rather than stays constant low at the tested area. This run through of an attrition minimum is normal for boosted nano particle plastics. [E.g. 7-11]

Fig. 8 shows the background story at the bar diagram. There are drawn the error bars, too, which have a limit at the highest and at the lowest point of the measured data. It is shown that all compounds with the lowest attrition rate also have the lowest diffusion.

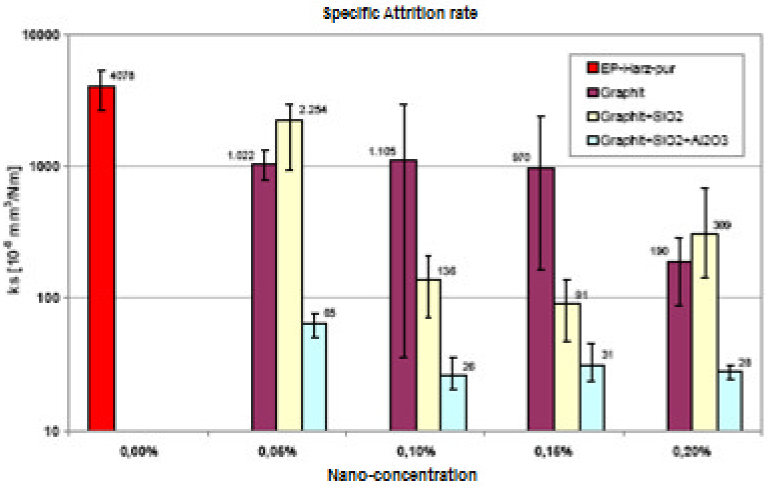

Fig. 8 Comparison of the specific attrition rates

The impact strength of all tested materials can be seen in Fig. 9

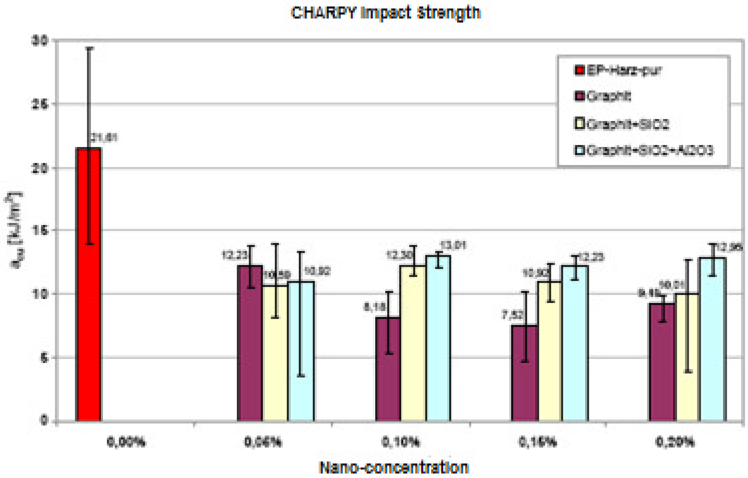

Fig.9 Comparison of CHARPY Impact Strength

The highest Impact strength with the lowest diffusion is at the filled resins with the mixture of all three matters by a filling ratio of 0.1% to 0.2 %.(shown is the highest and lowest value of the measured data). These samples had the highest wear protection, too. The tenacity of every filled compound is lower than the absolute resin. The differences between the filled samples among each other are relative low than to the unfilled resin, that's because the resin has a higher diffusion. A negative impact on the tenacity has the graphite, which can be seen at the elongation of Fig.11.

At Fig. 10 the tensile strengths are compared among each other. While the samples with the mixture of graphite and SiO_2 rather graphite, SiO_2 and Al_2O_3 at a matter concentration of 0.1 % and 0.15 % have a higher tensile strength than the absolute resin, it will be reduced at other concentrations. Graphite does have the biggest impact.

An identic picture is shown at the comparison of the elongation at Fig.11. The compound with more components of concentrations of 0.1% and 0.15% beat the values of the absolute resin, while a lessening is seen at other concentrations as well as the graphite.

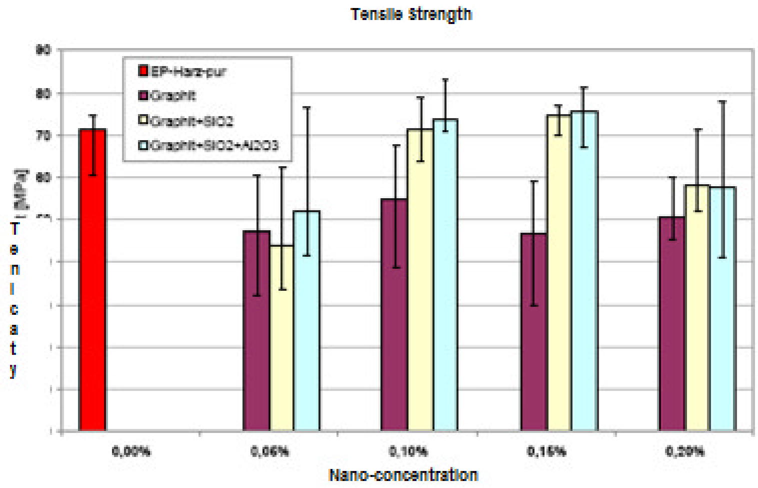

Fig. 10: Comparison of tensile strength

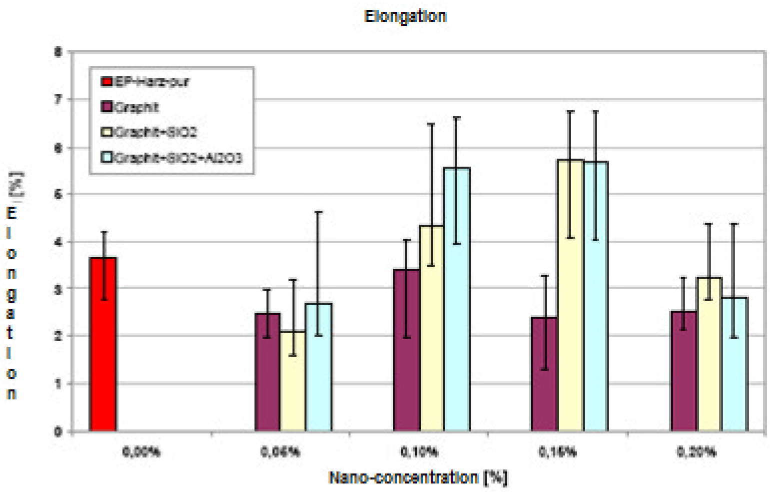

Fig. 11: Comparison of Elongation

The E-Module is increasing due to the graphite (Fig. 12), while the other compounds do not affect the E-Module rather than lead to a decrease. The decrease can be seen at the compounds with Al_2O_3. Exceptions are compounds with a matter concentration of 0.2 %. These compounds are lightly higher than the absolute resin within the E-Module, but its E-Module lies at the diffusion area of all filled resins.

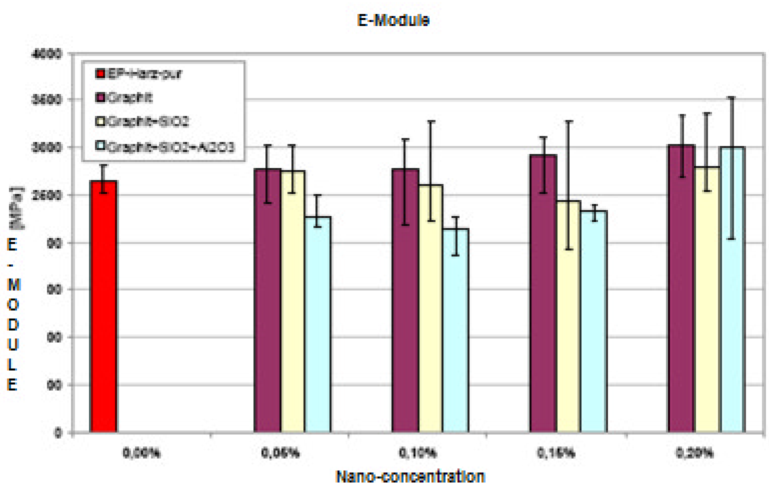

Fig.12: Comparison of E-Modules

4. Summary and Forecast

The attrition of epoxy resin can be reduced due to the concentration of the matter of plasma processed graphite, Nano-SiO_2 and Nano-Al_2O_3 (**NanoVit®**). (Up to two sizes)

The greatest impact shows the compound of all matters. It is shown a low minimum of the attrition rate at 0.1 %. At the compound out of graphite and SiO_2 it was seen a minimum at 0.15 %, but with high attrition rates. The attrition rate of the graphite – ER – Compound is decreasing due to the increase of the matter concentration.

The attrition consistent compounds do not have a special running-in characteristic and there will not be an increase of the attrition rate compared to the filled resins. Due to the matter concentration the tenacity becomes negative. The compounds with the highest wear protection compared to the other compounds do have the highest tensile strength, the highest diffusion and elongation. While the tensile strength is lightly higher than the absolute resin, the elongation is clearly higher. Tensile strength and elongation of other compounds are lower compared to the values of the clean resin.

The filled compounds of the E-Module have nearly the same level as the absolute resin, but the graphite causes a lightly increase, while the matter concentration of Al_2O_3 at the lower concentrations also causes a decrease.

The attrition of the epoxy-compounds has improved by filling them with Al_2O_3. It would be necessary to do more tests about the resin with aluminum oxide and mixtures of Al_2O_3 and graphite as well as combinations of Al_2O_3 and SiO_2 to understand more about the influence of the aluminum oxide.

If you want to improve the compounds you have to change the matter concentration. Another investigation could be the effect of graphite with nano matters with a higher concentration and if it causes a lower attrition.

Not least possess the measurements of the matters and the potential to get better triboligical characteristics at polymers matrix it also is useful for sliding lacquer.

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