# NEW METHOD TO DETERMINE THE INFLUENCE OF MICRO/NANO-PARTICLES ON SURFACE & FUNCTIONAL PROPERTIES

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**ABSTRACT**

Common material properties (e.g. hardness, tensile modulus and strength, poisson´s ratio) are well known. On the other hand, properties of thin layers and micro volumina can differ significant from those data, in particular for micro/nano-particle systems. AFM and FFM methods work with excellent lateral, topographic and force resolution but often do not provide testing-situations close to practical applications, e.g. high pressure flat contact within micro wear and micro friction investigations.

Optimization of friction/wear-properties of multi micro-area contact especially of relevance on curved and topographically structured surfaces need access to measurement technique, that covers the gap between micro and nano scale. The results presented here are done with the UST (Universal Surface Tester) to determine the influence of incorporation of micro/nano-particles and their distribution on micro mechanical and functional properties of polymer materials close to the surface.

## 1. COMPOSITE MATERIALS

During the last decade there was an increase in the usage of composite materials even for consumer products. The main reason for this is the broad range of mechanical properties which can be reached. Tailor-made properties are possible for nearly each application.

In a lot of cases carbon is used as the appropriate filler. Carbon black is used for rubber whereas carbon fibres are used for optimizing mechanical properties like the young's modulus, tear strength and wear resistance. Even the dynamical properties of composite materials are better.

## 2. NANO- AND MICROCOMPOSITE MATERIALS

Another increase in properties is possible by usage of micro- or nanoparticles. For example carbon shows a whole family of unusual $sp^2$-hybridized modifications like fullerenes [1] and carbon nano tubes [2]. Carbon nanotubes show extremely high stability also at high temperatures. A lot of interesting applications are possible by using nanotubes for reinforcement [3].

Table 1 shows the properties of typical fibres in comparison to carbon nanotubes [4].

"**Table 1.** Typical Properties of fibres."

| Property | carbon fibre | carbon nanotube |
|---|---|---|
| Young's Modulus (GPa) | 230 | 1000 |
| Tear strength (GPa) | 4 | 30 |
| Length/Diameter ratio | <100 | >1000 |

## 3. SURFACE PROPERTIES

As mentioned micro- and nano particles are used for reinforcement of the material. In a lot of cases the bulk properties like modulus are not so important. Surface properties or functionalities like wear or scratch resistance are of more importance in some applications.

Besides the technical advantage there is an important reason for producer to concentrate on surface properties. The appearance of a surface let a customer judge the value of the product. Therefore it is necessary to improve the surface resistance over time. Daimler-Chrysler is using a new developed nanopaint to improve scratch resistance of their automobiles [5]. The new 40 µm thick lacquer finish includes ceramic particles with a diameter of 20 nm. Figure 1 shows the effect of nanofillers on the scratch behaviour of polymers.

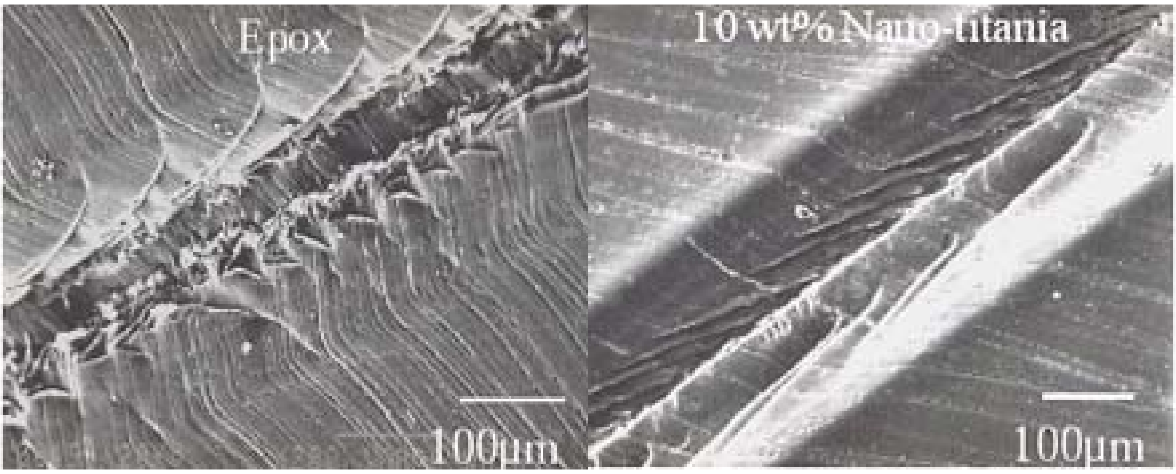


**Fig. 1.** Scratch on an epoxy with and without nanofillers [6].

The use of lacquer or special coatings improve the surface properties like elasticity, hardness, friction without using a big amount of material. Environmental resistance and chemo mechanical resistance are also improved.

## 4. MICRO STRUCTURE ANALYSIS (MISTAN®)

All measurements are done with an Universal Surface Tester (UST®) which is a new and patented measuring and testing device for continuously determining the deformation behaviour of a material in a region near its surface. The UST yields measured values of : permanent, plastic, elastic, viscoelastic and total deformation, micro hardness, hardness structure, abrasion and scratch resistance, creeping and relaxation, roughness, haptic and softness, cutting and piercing resistance, thickness and structure of layers and of microfriction. All this values are obtained with a high precision with only one single instrument.

Whereas existing procedures, e.g. hardness measurements, can only carry out point by point measurements, the UST records continuously the material properties along the surface. If required, the roughness parameters are determined from the surface profiles according to the DIN or ISO standards depending on the used tip geometry and the applied forces. Since the results include formation of every single deformation, it is possible to investigate specific deformation inhomogenities. Transitions between layers can also be easily recorded.

The instrument is capable of applying free programmable loads with specific tip on a surface while recording the Z-value continuously with a resolution of 60 nm, the load range is defined by two different modules either 0-100 nm or 2-600 nm. The load and test cycles can be applied to a static specimen or on one that is moving during the test procedure. Because of this continuous data can be recorded from the surface and the functional behaviour can be studied.

First it scans along a defined path X on the material's surface and determines continuously the vertical deflection Z and also the surface profile. Then the same path is once again scanned with the same tip which is loaded additionally with a defined load. The resulting surface profile G represents the local total deformation. At last the surface is scanned once again with the same load as in step 1. The elastic part of the total deformation E is now recovered. Thus the surface profile is established only by the remaining deformation. The difference between the measured profiles results from the determined surface deformations – total deformation G, elastic deformation E and plastic deformation P (figure 2).

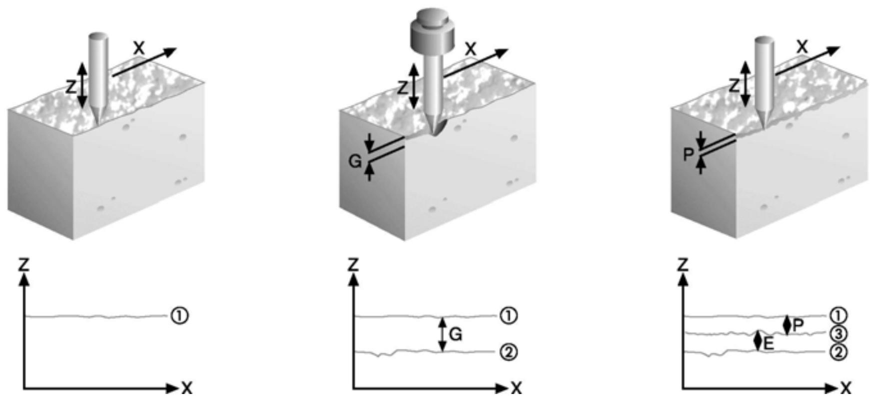


**Fig. 2.** The total deformation G and its elastic and plastic parts E and P could be calculated from the profiles ① and ③ measured with minimum load (0.7 mN) and ② with a load range between 1 and 100 mN.

It is possible to do this investigation even on curved or structured surfaces which are common in many applications. Figure 3 shows a standard measurement on a wave-like profile.

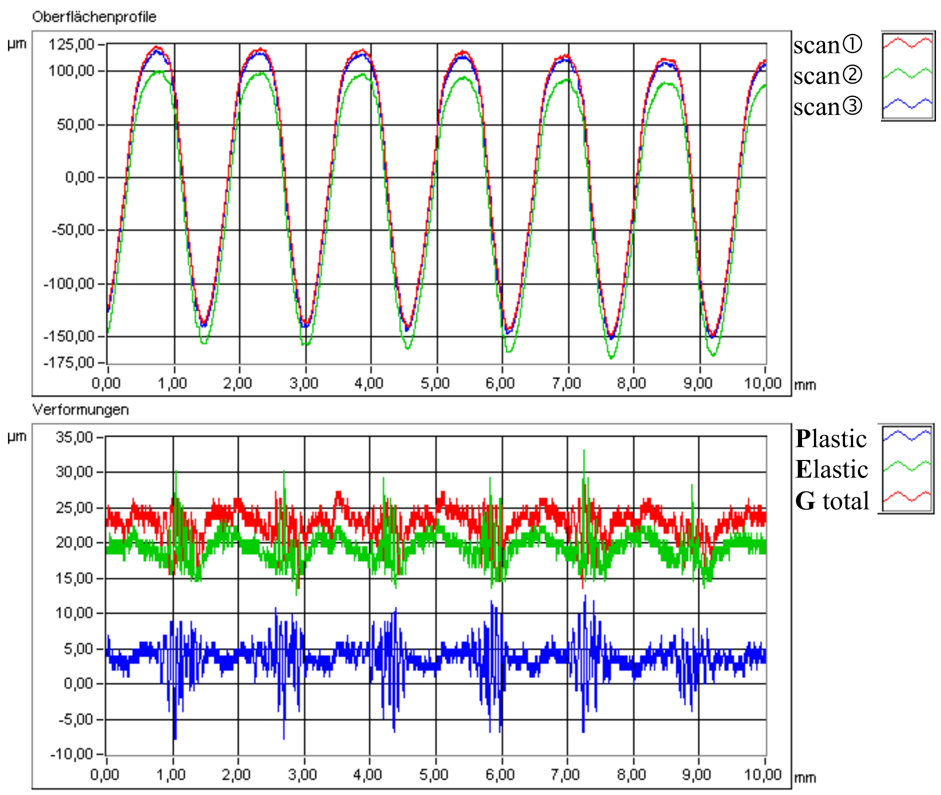


**Fig. 3.** UST standard measurement (profiles in the upper part and deformation in the lower part) on a curved surface

3-dimensional plots are also possible. An example is shown on figure 4:

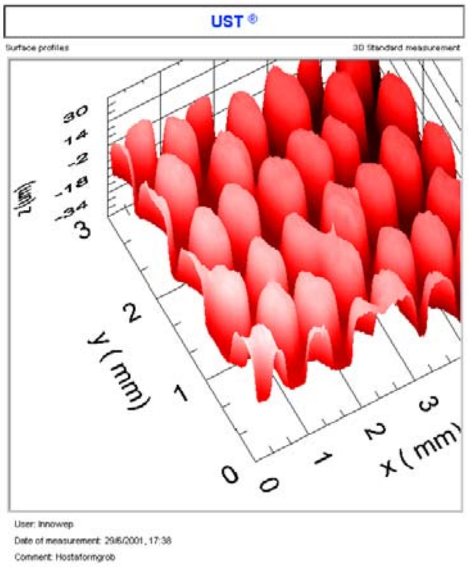


**Fig. 4.** Profile measurement of a micro structured POM sample

## 5. WEAR AND SCRATCH RESISTANCE

With the Universal Surface Tester it is possible to determine the scratch resistance [7] of a surface or a coating. In this case the load during the second profile scan is increased step by step. A scratch can be seen by an sudden increase of the permanent deformation followed by higher fluctuations of the deformation measurement due to material in front of the tip. Another measurement procedure is to repeat the second scan under load for several times. A scan by scan increase of the permanent deformation is the result. The wear resistance can be determined under those circumstances. The next figure (5) shows a scratch measurement on a coated glass surface. Starting with 10 mN the load is increased every 500 µm by 10 mN. With 50 mN a sudden increase in deformation occurs. The coating is damaged and the diamond top touches the original glass surface.

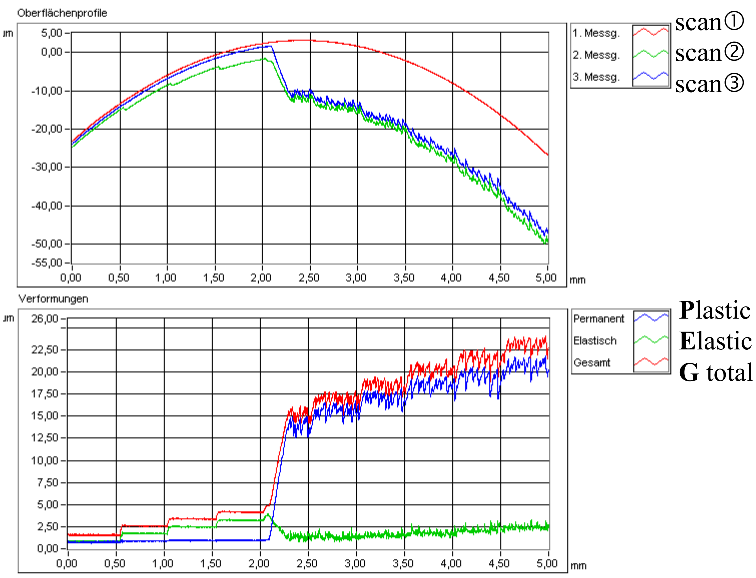


**Fig. 5.** Scratch test of a coated glass surface

## 6. MICROTRIBOLOGY

During the standard measurement the load on the surface is controlled. With a new module it is now possible to measure also the tangential forces during the movement of the tip over the surface. With this additional information of the frictional force it is possible to calculate the coefficient of friction $\mu_r$. Figure 6 shows the experimental setup for micro friction.

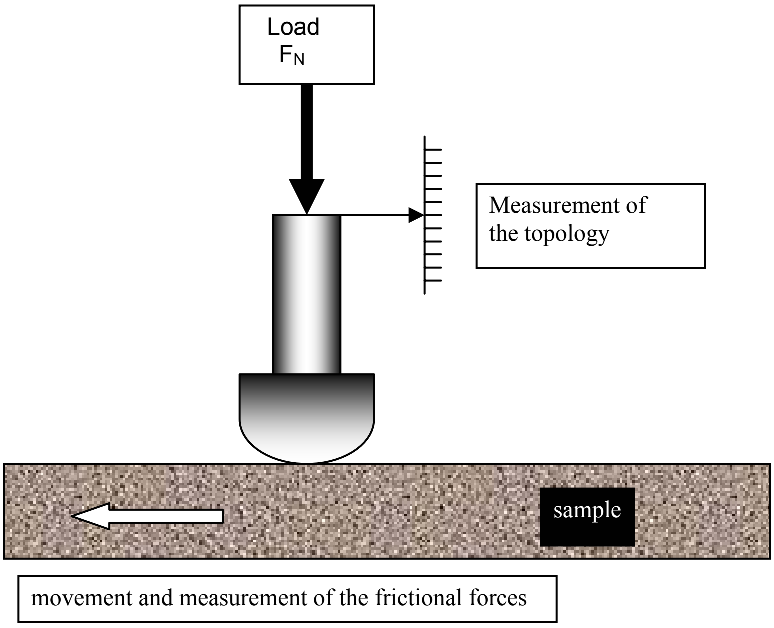


**Fig. 6.** Experimental setup

The results are nearly independent of the surface structure [8]. Figure 7 shows two results from the same material (POM) with different surface topology measured with a 0.8mm diameter steel tip and a load of 100 mN. One is nearly flat and the other structured. In both cases the frictional forces are approximately 35 mN leading to a frictional coefficient of 0.35.

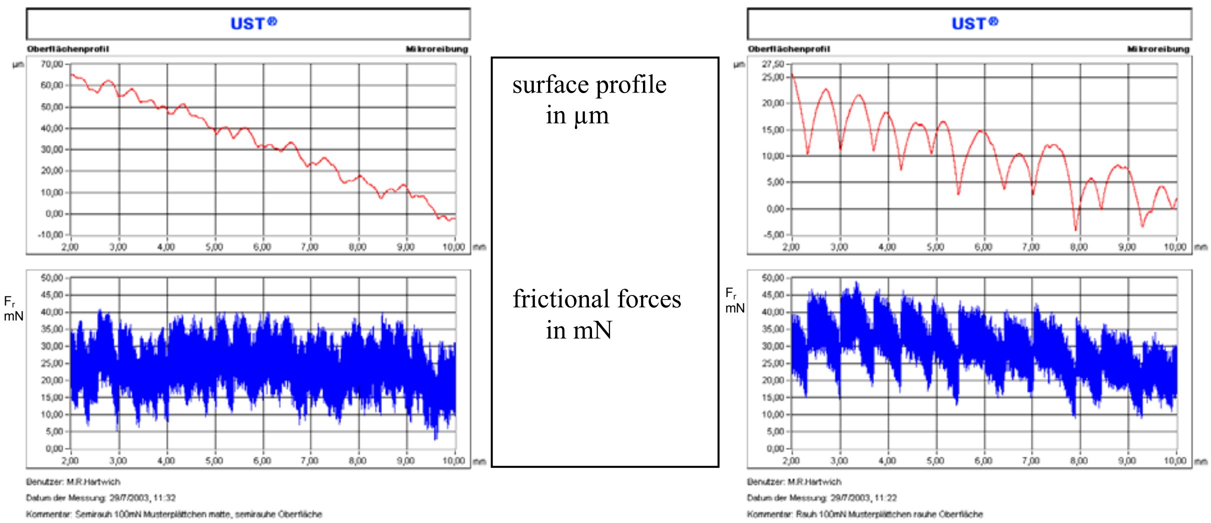


**Fig. 7.** Friction measurement of different structured POM surfaces

## 7. INFLUENCE OF NANOFILLERS

Nanomaterials can also be used to reduce the friction of a polymeric surface. An increasing content of the nanoscaped filler lead to an increase of the surface roughness. Therefore the effective surface contact is reduced. The following example shows an increase in total deformation from 1.94 µm to 2.22 µm for a PET film sample by increasing the filler content. This reduces the frictional forces (figure 8) from 23.66 mN to 15.87 mN without using slipping agents.

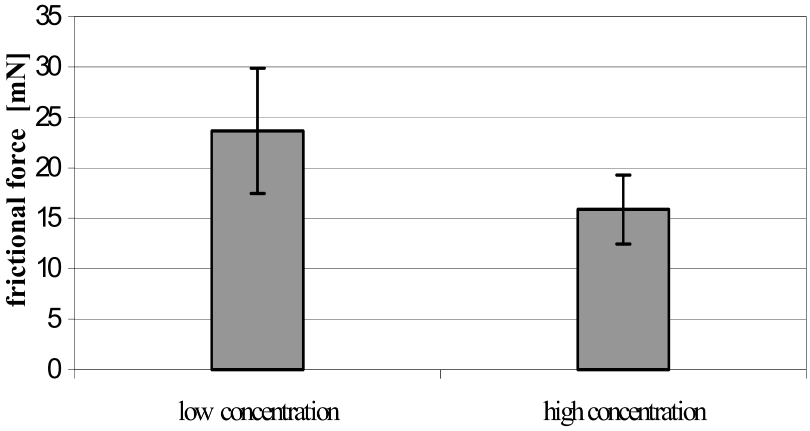


**Fig. 8.** Frictional forces of nanofilled polymers

## 8. CONCLUSIONS

With the described method it is possible to measure surface properties like elasticity, plastical deformation under load, recovery and functionalities like wear and scratch resistance or friction. All these information can be determined also on structured surfaces. By using appropriate tip material and geometry it is possible to simulated real application conditions. Especially in the field of nanocomposites this opens a wide field of testing conditions.


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