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### Combination of normal and lateral force-displacement measurements as a new technique for the mechanical characterization of surfaces and coatings

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#### Abstract

Nanoindentation with Vickers or Berkovich tips has become an established technique for the measurement of hardness and Young's modulus. Also the use of spherical indenters has attracted more and more attention for the determination of the Young's modulus and yield strength. However, these techniques are still far away from the conditions in a real applications where usually normal forces are combined with lateral forces in a tribological system. Therefore, often scratch and wear tests are used additionally to characterize the mechanical behavior. However, conventional techniques, which apply lateral forces, have the disadvantage that they often do not deliver characteristic and comparable material parameters which are independent of the measurement conditions and which can serve as input parameters for the modeling with analytical or finite element methods. This shall be overcome by a new technique, which will allow the measurement of lateral force-displacement curves with the same accuracy like conventional nanoindentation can do in normal direction. The realization of two independent measurements with the same tip at the same sample position will allow the calculation of more unknown material parameters than before. The use of spherical indenters with forces at which elastic or beginning plastic deformation takes place makes it easier to combine the measurements with analytical stress calculations and to derive critical material parameters. As a first step towards the abovementioned characterization method, a new nanomechanical tester with a high-resolution measurement of force and displacement in normal and lateral direction was developed and tested.

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#### 1. Introduction

Since the beginning of the 1990s, nanoindentation has become a powerful tool for the mechanical characterization of surfaces and thin films. The existing instruments have a high force and displacement resolution (see for example [1-3]) and reliable and reproducible results could be obtained using the well known method of Oliver and Pharr [4]. Based on this method, the nanoindentation technique was standardized in ISO 14577 [5] in order to obtain comparable results independent of the measuring instrument. However, in up-to-date nanoindentation, only normal forces are considered whereas in practical applications also lateral forces occur due to sliding and sticking friction. Additionally, some material parameters like Poisson's ratio, the critical tensile stress for crack formation or the yield strength of thin films cannot be measured by nanoindentation in normal direction alone. Some nanoindentation devices have an additional scratch option [6]. However, the lateral force cannot be applied without a lateral displacement because the bending of the indenter shaft is measured. In general, scratch tests do not help to close the gap because the available force and displacement resolution is not good enough. First attempts to increase the resolution in wear tests have been made [7], but the "nanotribometer" has a very limited operation range. The result of a common scratch test is not a real material property but depends on the measurement conditions such as radius and material of the indenter or scratch speed and on the thickness of the coating. Therefore, such results are not acceptable as input parameters for mathematical modeling.

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In this article, we report on the experimental realization of an Universal Nanomechanical Tester (UNAT) consisting of two measuring heads, one for the normal direction as used in common nanoindentation devices and another for high resolution measurements of force and displacement in lateral direction. Both are coupled by a special sample stage with high stiffness in normal direction and low stiffness in lateral direction. Some possible applications of the new instrument will be pointed out in this article.

#### 2. Experimental details

In principle, the combination of both normal and lateral measuring heads as stand-alone devices is possible by putting together a common nanoindentation device and a lateral force unit (LFU) as demonstrated in [8]. The disadvantage of this set-up is the independent operation of the two devices which have to be synchronized manually. Further, the lateral stiffness of the indenter shaft is mostly not as high as desirable because those devices are made for normal forces only. Therefore, a new Universal Nanomechanical Tester (UNAT) was developed (ASMEC GmbH, Germany), the software of which allows the synchronized operation of normal and lateral load-displacement cycles in a very flexible manner. A sketch of the set-up is shown in Fig. 1. The displacement is measured by an LVDT. A second LVDT measures the displacement of a leaf spring and therefore the force (patent pending). The force measurement is completely independent of the force application,

which is done by a piezoelectric element. This enables a highly stabilized force application over time for creep experiments and other investigations. The device is equipped with a high-resolution optic system (and optional with an AFM) for the imaging of indentations and scratches.

## 3. Range of application of lateral forces in nanoindentation

In this section, a brief presentation of possible applications of combined normal and lateral forces will be given. However, by far not all possibilities can be pointed out.

#### 3.1. Determination of the yield strength of thin coatings

The yield strength as a measure for the onset of plastic deformation is a very important property of protecting thin films and substrates [9]. Since the mechanical properties of thin films can significantly differ from those of the according bulk materials, they have to be determined independently. The measurement of yield strength of thin and especially of hard and brittle coatings is very difficult and cannot be done by conventional methods like tensile tests. A method, which uses cyclic indentation experiments (load–partial unload) in normal direction with spherical indenters was introduced in [10,11]. In such experiments, the critical (normal) force for the onset of plastic deformation can be determined. This force depends on the indenter geometry. However, with the known indenter



Fig. 1. Set-up of the UNAT.

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