CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

The Ultra Nanoindentation Tester: new generation of thermal drift free indentation

Introduction

Recent development of advanced materials has motivated the development of nanoindentation instruments using extremely low loads and indentation depths. These instruments can apply forces from micronewtons up to several millinewtons and measurement depths down to a few nanometers. To achieve such good resolution, these nanoindentation machines are generally placed in a soundproof enclosure on an antivibration table to eliminate the negative influence of vibrations and noise. Of great importance is the thermal stability of the environment which typically should not exceed 0.5°C/hour. This criterion is crucial for nearly all presently commercially available instruments as they all suffer, to some degree, the problem of thermal drift. The 'thermal drift' term is widely understood as 'the change in the displacement signal when the normal force on the indenter is maintained constant and the material does not exhibit time dependent mechanical properties'. Because of thermal drift the use of many indentation instruments is significantly limited for long term measurements such as creep or grid indentation. In general, two main approaches have been employed to minimize the effect of thermal drift: a) introducing a stabilization period before the measurement (such thermal stabilization can take several hours), b) performing the indentation very quickly, within a few seconds. In both cases the effect of thermal drift is minimized but not removed completely. As the source of thermal drift is not removed entirely, performing long term or creep measurements is still complicated and can give severely misleading results.

The Ultra Nanoindentation Tester features

The Nano Hardness Tester (NHT) introduced in 1997 by CSM Instruments has been shown to greatly reduce the problem of thermal drift by the utilization of passive surface referencing. The outcome of continuous development at CSM Instruments has resulted in the recent release of a new generation of nanoindentation instrument, the Ultra Nanoindentation Tester (UNHT). This instrument, using patented active surface referencing and new advanced materials, takes the CSM Instruments technology further towards a thermal drift free high resolution instrumented indentation. The unique surface referencing principle, along with the use of the most advanced electronics and materials, eliminates almost entirely the problem of thermal drift and frame compliance. The UNHT measurement head can be easily mounted on existing CSM Instruments Compact and Open platforms and the system can be completed with other modules such as Atomic Force Microscope (AFM), Nano Indentation Tester (NHT) or scratch testers (Fig. 1).

The UNHT can be used in either force controlled mode or displacement controlled mode. Dynamic mechanical analysis using Sinus mode for storage and loss moduli calculations, as well as depth profiles, is also available. Both simple indentation and Sinus mode indentation can be performed in constant strain rate mode.

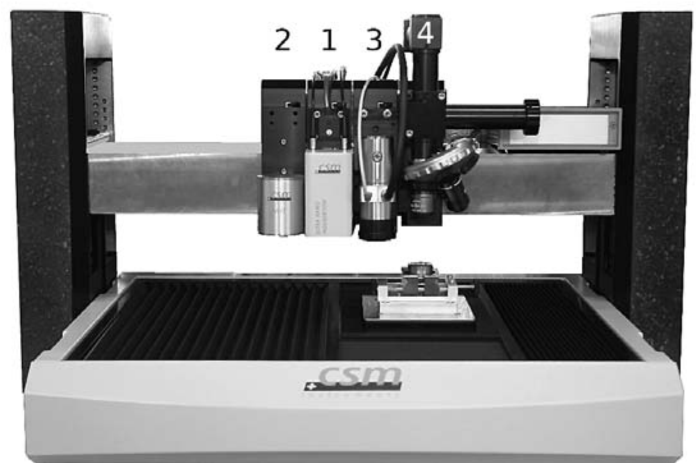

Fig. 1 – The UNHT on CSM Instruments Open Platform modular system: 1 – UNHT head, 2 – NHT head, 3 – AFM, 4 – optical video microscope.

Principle of the UNHT

The Ultra Nanoindenter is based on a novel architecture using two independent vertical axes: one axis dedicated to the indentation itself and one axis used for active surface referencing (see Fig. 2). Each axis has its own actuator, displacement and load sensors. For both axes, the displacement is applied via piezo actuators A1 and A2. The load on the indenter and the reference is obtained from the displacement of the springs S1 and S2, measured with capacitive sensors C1 and C2. The spherical reference (with typical diameter of 3 mm) is located 2.5 mm (9 mm in special case) apart from the indenter. The displacement of the indenter is measured relative to the reference through the differential capacitive sensor C3. The force applied on the reference is maintained on a constant level, ensuring that the reference follows precisely every displacement of the sample surface. Continuous control of normal force on both the indenter and the reference is ensured by precise feedback loops. The crucial components used in the measurement head are made of Zerodur ®, a material with extremely low coefficient of thermal expansion (0.01×10^{-6} K^{-1} over the range 0°C - 100°C). Since the problems of thermal drift and frame compliance have been almost completely eliminated in the UNHT, all measurements contain only raw data without any software or hardware correction of thermal drift or frame compliance.

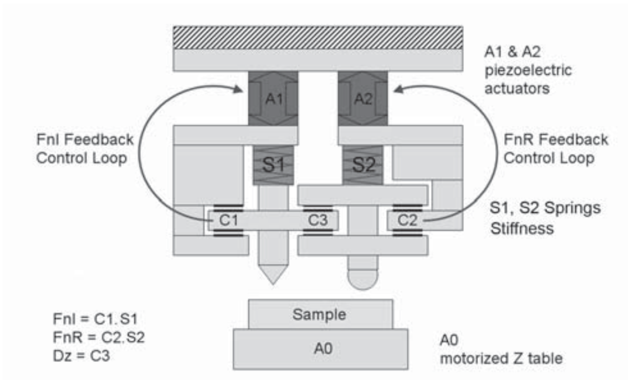

Fig. 2 – Two-axis UNHT principle: A0 represents the displacement of the motorized Z table, A1 and A2 are the piezo actuators used for the application of the active reference force (FnR) and the indenter normal force (FnI). S1 and S2 represent the springs used in the system for measurement of the forces involved. The indentation depth (Dz) of the indenter tip is directly measured via the differential capacitive sensor C3.

Key advantages of the UNHT: Displacement and force stability

The main advantages of the UNHT are its excellent thermal stability during long term measurements, zero frame compliance and force stability during the indenter approach. To demostrate the thermal stability of the instrument, indentation on fused silica – which is known to have time independent mechanical properties – was performed.

Thermal drift

Figure 3 shows two typical displacement versus time curves recorded during a 300 second hold at two different loads. The average value of thermal drift recorded during a large number of tests is below 0.5 nm/min and in many cases as low as 0.2 nm/min.

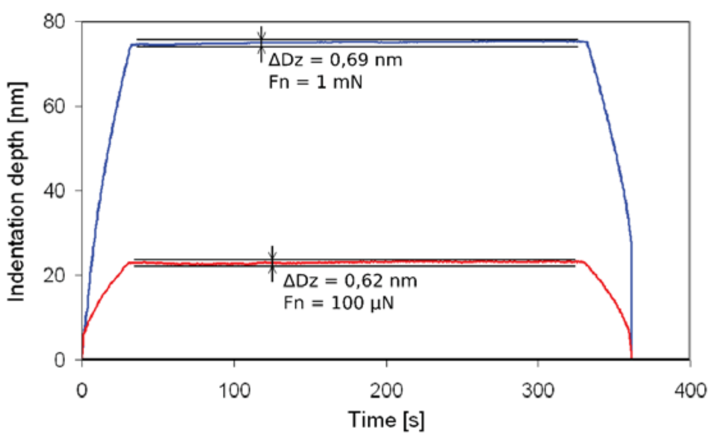

Fig. 3 – Thermal stability of the displacement signal during a 300 s hold at maximum load.

Furthermore, as the symmetrical architecture of the UNHT implies, the system shows very low thermal drift even at elevated temperatures. CSM Instruments provides a special heated stage where sample can be heated up to 450°C. Preliminary tests at 100°C confirmed that the UNHT has a thermal drift on fused silica as low as four nanometers over 120 s.

Stability of the normal force & adhesion measurement

The thermal stability of the UNHT system was also studied by recording the normal force signal during the indenter approach to the surface. The stability of the force signal during this approach is crucial for certain types of measurement where force detection above the materials surface is required (adhesion tests). In our experiments, the approach speed was set intentionally very low and the approach of the indenter to the surface took 5 to 10 minutes. The results show that the drift of the force is less than 1 µN over 8 minutes (Fig. 4a). The same type of measurement was then used for detection of the surface of an oil film smeared on steel. The contact with the surface of the film was detected by a drop in the force signal of ~2 µN and the corresponding thickness of the film was estimated to be approximately 180 nm (Fig. 4b).

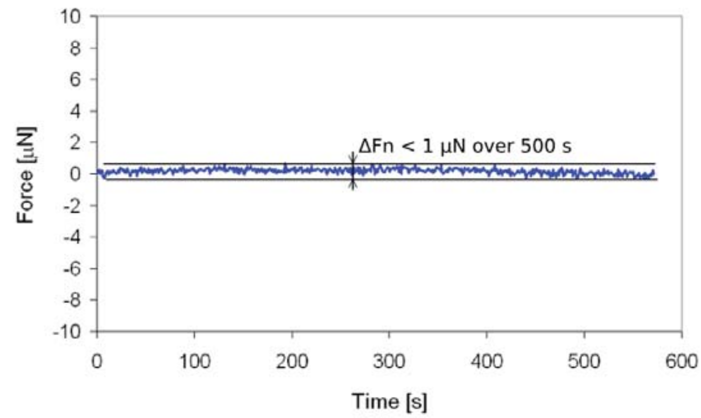

Fig. 4a – Thermal stability of the normal force signal during indenter approach (the indenter approach speed was intentionally set very low).

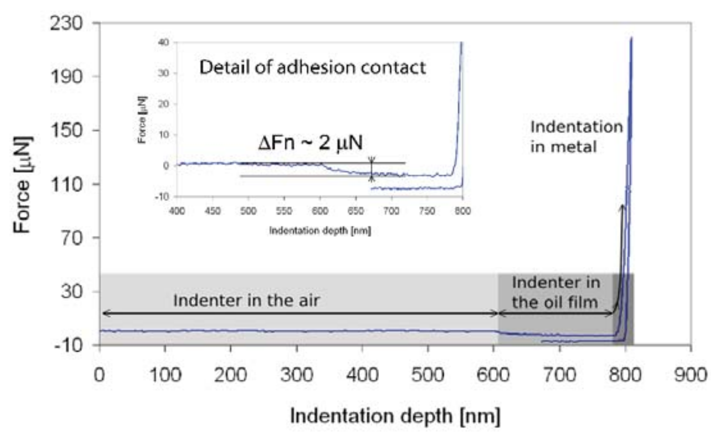

Fig. 4b – Excellent stability of the normal force during indenter approach used for measurements of adhesion and oil film thickness.

Frame compliance

One very important aspect of the active surface referencing is elimination of frame compliance. All of other commercially available instruments must correct the frame compliance that introduces an error component in the displacement signal due to frame deformation. The frame compliance must be measured and the displacement signal corrected. Due to the use of active surface referencing, the effective frame is reduced to the indenter and reference assembly in contrast to the whole frame in other instruments. Our attempts to calculate the frame compliance using the method recommended by ISO 14577 resulted in the conclusion that the compliance value is so small that it is practically impossible to distinguish it from the scatter of the data around the zero value. The indentation data from the UNHT can therefore be used without any frame compliance correction.

Selected applications of the UNHT

The experiments performed with the UNHT were aimed to show its thermal stability and versatile use for various methods on a wide range of materials including hard materials, soft materials, extremely soft materials and multiphase materials. Most of the tested samples exhibited flat, mirror-like surfaces and the influence of surface roughness was therefore minimized. All samples were fixed in a way to prevent their lateral movement and the instrument was placed in acoustic enclosure in an air-controlled laboratory (temperature stability of 23°C ± 0.5°C).

Fused silica: polished fused silica disk was used as it is a reference material in nanoindentation. Mechanical properties of fused silica are known to be time-independent at room temperature and the material is therefore suitable for measurements of instrument thermal drift.

Polymers: two bulk samples of PMMA (Polymethyl Methacrylate) and PC (Polychloride) were used for demonstration of creep measurements and the influence of the loading rate.

Thin coatings: indentation on low-K coating of 200 nm thickness deposited on a silicon wafer.

Extremely soft materials were represented by ballistic gel.

Stress-strain measurements were performed using a spherical indenter on stainless steel sample.

Grid indentation: large matrix indentation was performed on a special sinter-clad coating composed of a CrNiMoV matrix containing fused tungsten carbide (FTC) particles.

Indentation parameters

All measurements except those for stress-strain characterization were performed with a modified Berkovich diamond indenter with total included angle of 142.3°. All indentations were performed according to ISO/ASTM standards. Force controlled mode was used and calculations of elastic modulus and hardness were based on a modified Oliver and Pharr approach. The indenter area function was calibrated using a standard procedure by series of indentations at increasing loads on fused silica.

Fused silica and low-K coating: low load indentations

Two materials were selected for low load indentations: fused silica and 200 nm thick low-K coating. The results are shown in Fig. 5: extremely low load was required for the low-K coating as not to indent above approximately 10 % of the thickness of the coating.

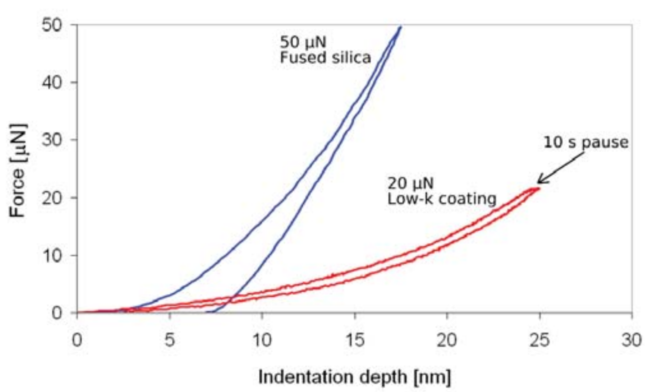

Fig. 5 – An example of two load-displacement graphs for very low loads and indentation depths. Creep was measured during 10 s hold on the low-K coating.

The measured Young's modulus of fused silica at 50 µN load and 16 nm depth was 71.2 GPa which corresponds to the manufacturers specifications (72.0 GPa ± 2.0 GPa). At such low depths care must be taken when evaluating the measurements as indentation size effect or tip rounding may affect the results. Precise determination of the indenter projected area function is therefore essential for all experiments at low indentation depths. Note creep during the 10 s hold period on the polymeric low-K coating: due to quasi zero thermal drift of the UNHT this displacement change represents the true creep of the material.

Creep studies on polymers

Measurements of creep were performed on PMMA and PC polymers. The loading time was set to 30 s, maximum load to 1 mN, hold time to 120 s and unloading time to 30 s. The ΔDz creep value was calculated as the difference in indentation depth between the end of the pause and the beginning of the pause. As the loading rate was identical for both materials, the creep level could be compared: the PMMA showed more than twice as much displacement change compared to the PC (Fig. 6).

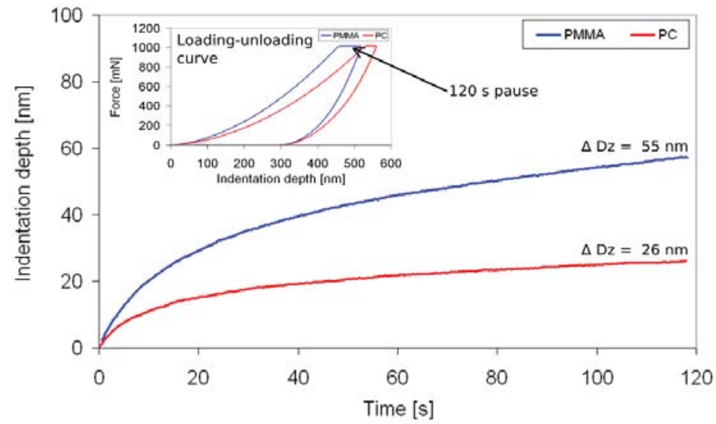

Fig. 6 – Creep measurements on PMMA and PC polymers.

This type of measurement revealed the viscous properties of both tested materials. All creep experiments were performed without any thermal stabilization or thermal drift correction. The UNHT is therefore an excellent instrument for more advanced studies of materials exhibiting time dependent properties or very soft materials.

Extremely soft materials: gels

Until recently, instrumented indentation has never been considered as a suitable tool for the testing of extremely soft materials. The main reason was the necessity of applying very low loads with the capability of measurement of large displacements. Further, very good thermal stability is required in these experiments as most of these materials have significant time dependent properties.

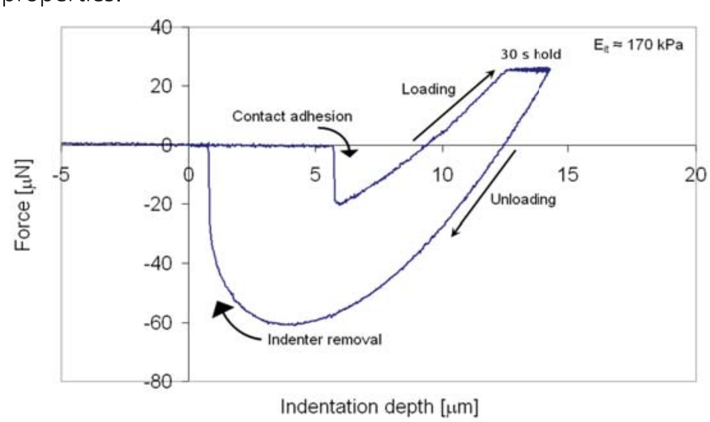

Fig. 7 – Load-displacement graph recorded on ballistic gel. Note high indentation depth when using relatively low loads and adhesion phenomena during indenter approach and removal.

The UNHT indentation experiment performed on gels showed that measurement of such materials is not only possible but also gives excellent results. This is due to the active surface referencing bound with low load and high displacement range capability of the UNHT system. For most of our experiments on gels loads in the range of only several tens of micronewtons were used while the displacement was in the range of 10 µm to 20 µm. A typical example of such a measurement is shown in Fig. 7: for a maximum load of 30 µN the maximum indentation depth was 14 µm and the elastic modulus was ~170 kPa. These values are more than six orders of magnitude lower than those of metals or ceramics. The adhesion phenomena during indenter approach and removal could also be observed due to the stability of the normal force.

Stress-strain characteristics

Based on Tabor's model, indentation can be employed for characterization of the stress-strain behaviour of materials. Indenting with a spherical indenter yields increasing representative stress and strain at increasing indentation loads. This relationship is then used in representative stress versus representative strain plots that can reveal similar information about the elastic-plastic behaviour of materials as tensile stress-strain curves. To obtain the whole stress-strain plot by indentation both very low loads and low depths and high loads and rather high depths are required in order to characterize the material over the whole elastic and plastic range. The UNHT with its high load and displacement resolution is a perfect tool for precise analysis of the stress-strain behaviour of various types of materials. Loads from 20 µN up to 100 mN can be used and the indentation depth can vary from a few nanometers up to fifty micrometers. An example of a stress versus strain plot obtained on stainless steel using a 20 µm radius spherical indenter is shown in Fig. 8.

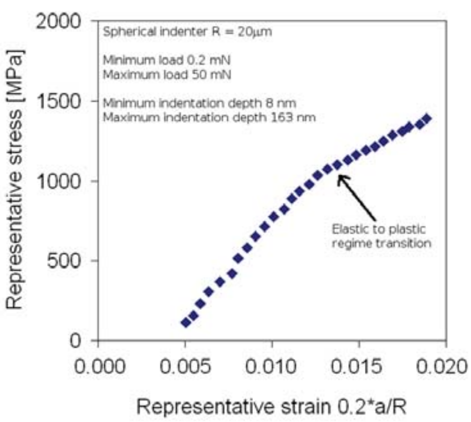

Fig. 8 – Stress-strain curve as calculated from Continuous Multi Cycle indentations with a spherical indenter on stainless steel. The offset of the plot on the X-axis is due to imperfections of the indenter.

Grid indentation: statistical analysis of large sets of indentation data

The excellent thermal stability of the UNHT makes this instrument ideal for characterizing composite or multiphase materials by 'grid indentation'. This method consists of performing a large matrix of identical indentations whose results are then treated statistically. The number of indentations can be from several hundreds up to thousands of indentations. The grid indentation method is particularly useful on multiphase materials where single phases are difficult to locate or their dimensions are too uneven. The grid indentation method was successfully applied on a CrNiMoV metal matrix with FTC particles. A total of 625 indentations in a rectangular pattern (25 x 25 indentations spaced by 5 µm) were made. Statistical evaluation of the results (Fig. 9) shows that at least two phases are present: the metallic matrix and hard carbide particles. Further, an 'envelope' around the FTC particle represents the third phase. The hardness of the three phases was respectively 11681 MPa for the matrix, 26478 MPa for the FTC particle and 22043 MPa for the particle 'envelope'. Apart from properties of individual phases the grid indentation method also allows calculation of the volume fraction of each phase.

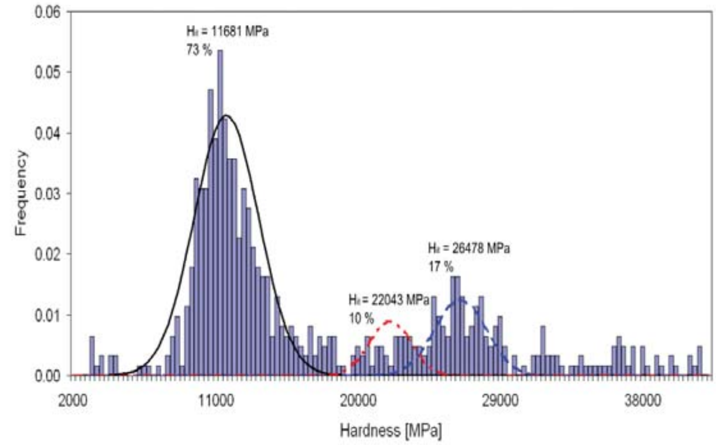

Fig. 9 – Statistical histogram analysis of the results of grid indentation showing combination of three phases.

Conclusions

The presented results show that the new Ultra Nanoindentation Tester is an excellent tool for demanding applications of instrumented indentation. This Application Bulletin highlights the main advantages of the unique UNHT concept: extremely low thermal drift and high load and depth resolution. Superior performance of the UNHT has been achieved thanks to the active top referencing and use of materials with very low coefficient of thermal expansion. The features of the UNHT make it ideal for new methods such as creep or stress-strain measurements and experiments on advanced materials such as gels or very thin films.

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This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

Editor Jiri NOHAVA, PhD.

Should you require further information, please contact:

CSM Instruments
Rue de la Gare 4
CH-2034 Peseux
Switzerland

Tel: + 41 32 557 5600
Fax: +41 32 557 5610
info@csm-instruments.com
www.csm-instruments.com

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