DEVELOPMENT OF NANOINDENTATION TOOL FOR TESTS AT DIFFERENT TEMPERATURES AND ITS USE FOR MICROCARACTERISATION OF MEMS

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ABSTRACT

Nanoindentation is a powerful technique to determine various mechanical properties of thin layers, such as for example Young's modulus. MEMS characteristics, like the stiffness of beams and bridges can also be extracted. Until now, we were able to perform this type of characterization at CNES at room temperature. In order to get closer to environmental conditions where MEMS are used, a new nanoindentation tool has been developed to determine material properties at different temperatures ranging from -25°C to 100°C. After a brief overview of the main analysis used in nanoindentation, the effects of temperature are summarized. Then, the developed thermal nanoindenter tool is presented. Since the estimation of the accuracy of results is necessary to improve our skills about nanoindentation experiments, a sensitivity analysis has been performed, allowing in addition to understand the impact of temperature on the quality of the results. The thermal nanoindenter tool is used to carry out measurements on PMMA and SiO_2 samples. The obtained results are compared to literature/other measurement results and validate successfully the developed tool.

INTRODUCTION

MEMS inherent properties make them very interesting for space applications where they have to work in very harsh environments (temperature changes, radiation….) and a great reliability is necessary. In order to insure this reliability, we have to dispose of precise knowledge concerning the materials properties. Often this knowledge is not easily available because the mechanical properties of thin layers depend strongly on fabrication processes and environmental constraints such as temperature.
Nanoindentation can be used to measure mechanical parameters of thin layers, such as for example Young's Modulus and at CNES we have been carrying out nanoindentation experiments at room temperature for a long time [1]. Moreover they can be used to determine MEMS characteristics, like the stiffness of beams and bridges [2]. Various studies have already been successfully carried out. Unfortunately most nanoindentation tools just can work at room temperature.
We are now developing a nanoindentation tool that carries out experiments at different temperatures and improves so our knowledge of material properties. In a first part of this paper we will present the functional principle of nanoindentation, then we will present the development of the thermal nanoindenter tool developed at CNES, in a third paragraph we will deal with the quality of measurement results and in the fourth part some experiments carried out with the thermal nanoindenter tool will be presented.

NANOINDENTATION TECHNIQUE

Indentation experiments have been used for more than a century to measure mechanical properties (hardness…) of materials. The principle is simple: a tip supposed non-shrinking is pressed against the test materials. An example of diamond tip is shown in Fig. 1. The hardness can then be obtained by measuring the size of the remaining impression versus the applied load.

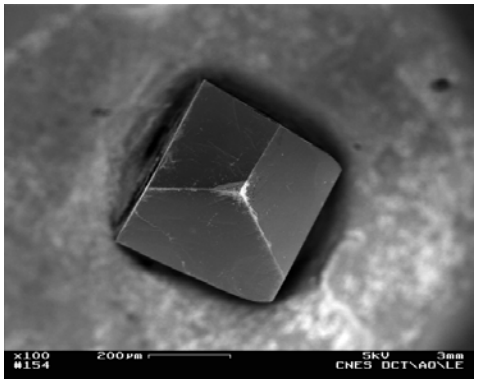

Fig. 1. Berkovich tip used for nanoindentation (SEM)

The nanoindentation technique is based on the same principle, but scales are so small that the direct observation of the print is no longer possible with conventional means. For this reason continuous measurements of the applied load and the tip displacement are used to determine mechanical properties. Current technology allows a load resolution in the range of 0.1 µN and a displacement resolution in the range of 0.1 nm. With the nanoindentation technique, not only hardness is available, but also Young's Modulus and some other mechanical properties, like toughness or yield stresses.

Determining Young's Modulus *E* and the Hardness *H*

In order to determine mechanical parameters, such as the hardness *H* or Young's Modulus *E*, the tip of the nanoindentor penetrates into the material. During this load-unload cycle the load *P* and the displacement *h* are measured.

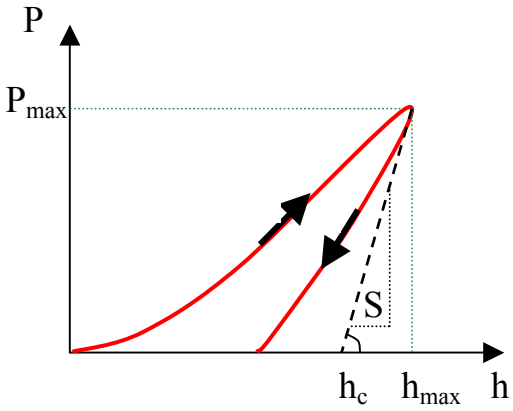

Fig. 2. Load *P* versus displacement *h* curve

Later on the hardness *H* can be calculated with the following definition:

$$H = \frac{P_{\max}}{A_c} \tag{1}$$

where P_{max} is the maximum applied load and A_c the contact area of the tip on the surface of the material. The contact area can be calculated based on the contact depth h_c

Young's Modulus is calculated based on the Sneddon equation :

$$E^* = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A_c}} \tag{2}$$

where E^* is the composite Young's modulus depending on both Young's modulus and Poisson's ratio of test material and of tip material, S is the contact stiffness and β a correction coefficient near to 1 (β=1.034 for a Berkovich tip).

Based on the results obtained for E^*, Young's Modulus of the test material E can be calculated, where "i" is the subscript for the properties of the indenter:

$$\frac{1}{E^*} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{3}$$

Determining the Contact Area A_c and the Contact Stiffness S

The calculation of A_c is based on the measurement of the penetration depth during load h_c. To take into account the tip defects, Oliver and Pharr have proposed the use of a polynomial function of contact depth [3]:

$$A_c = C_0 h_c^{\,2} + C_1 h_c + C_2 h_c^{\,1/2} + C_3 h_c^{\,1/4} + C_4 h_c^{\,1/8} + \ldots \tag{4}$$

where C_i are the tip coefficients. C_0 is a value given by the tip supplier, the other coefficients depend on the tip abrasion and have to be determined regularly. The value of h_c can be determined by the following equation :

$$h_c = h - \varepsilon \frac{P}{S} \tag{5}$$

where h is the displacement of the tip, P is the applied charge and ε a constant ($\varepsilon = 0.75$ for a Berkovich tip).
The contact stiffness can be calculated by differentiation of the unload part of the experimental curve. This analytical expression has been proposed by Oliver and Pharr [3] :

$$S = \left.\frac{dP}{dh}\right|_{h=h_{\max}} = Bm\left(h_{\max} - h_f\right)^{m-1} \tag{6}$$

where h_{max} is the maximal penetration depth, B and m are experimental coefficients determined by interpolation and h_f the depth of the print that remains on the surface after tip removal.

A second technique to determine the contact stiffness S has been developed during the last years: the Continuous Stiffness Measurement (CSM). This technique is an important improvement for measuring the contact stiffness. The indenter is driven during loading by superposing a small oscillating force on the primary load signal and the resulting harmonic response is analyzed. The technique is based on an accurate model for the dynamic response of the indentation system [4-5]. As far as S is measured continuously, one can obtain the hardness and Young's modulus as a continuous function of depth.

Influences of Temperature on Nanoindenter Measurements

In the description of the theory of nanoindentation, the reaction of the materials to an applied load has been considered as (nearly) instantaneous as it is the case for most metals or ceramics at ambient temperature. However, the experiments show that the deformations are time dependent with a high influence of temperature. Indeed, polymers viscoelastic behavior at room temperature is well-known and time-dependent creep is an important phenomenon for metals and ceramics at higher temperatures. If those phenomena appear, in the worst case, the indentation depth can increase even when the indenter is unloaded. Anyway, the slope of the load-unload curve is modified and induces an abnormally high contact stiffness, resulting in a false calculation of Young's modulus or hardness.

Material Properties

Two different material effects can be changed by temperature: creep and viscolelasticity. If there is a risk that creep appears, its influence can be taken into account and corrected in a lot of case by holding the maximal load constant for a while before unloading [6]. This technique allows short-lived creep materials to accommodate to the load. Besides, nanoindentation tests can be performed to evaluate the creep rate.
Viscoelasticity of polymers is often a problem even at room temperature. This phenomenon can be characterized thanks to continuous stiffness measurements. Indeed, the dynamic model takes into account internal friction and damping, and the measurement of the response and its phase angle are analyzed, resulting in the estimation of a storage modulus and a loss modulus [7].

Nanoindentation Measurements

Not only material effects, but also the sensitivity of the nanoindentation tool itself might influence the experimental results through one specific parameter measured during a nanoindentation experiment: the thermal-drift. The measured displacements have to account for small amounts of thermal expansion or contraction in the test material or indentation tool. The drift rate varies in a relatively short time, therefore a new correction must be calculated for each test. Assuming that the only reason for a displacement when the indenter is pressed against the test material at small and constant load is thermal drift, the thermal-drift calibration can be performed as soon as the unload cycle was traversed up to 90 %: the applied load is then clamped for a certain time and the tip displacement is measured. Then all the displacements measured during the test are corrected according to the time at which they were acquired.

3. THERMAL NANOINDENTATION TOOL

In order to carry out nanoindentation tests at different temperatures, a thermal nanoindenter tool has been developed at CNES. The nanoindenter was placed inside a thermal chamber (Thermotron). As temperature variations lead to dilatations of the tool and the test material, the thermal characteristics of the chamber like inertia play an important role in the quality of the results. So, various characterizations were carried out.

The study of the thermal behavior of the chamber has been made thanks to several thermocouples placed inside the chamber. The characterization results showed no stratification and a very good thermal homogeneity inside the chamber and on the sample. They also showed that the wanted temperature was exceeded by more than 1°C during heating. This can become critical for the test of polymers that are near their transition temperature. Moreover, this chamber controls temperature thanks to two serial compressors and a condenser. Mechanical vibrations due to this equipment are definitely incompatible with nanoscale measurements. Hence, one have to work with thermal regulation switched off. In order to verify the stability of temperature during a nanoindentation experiment, the time constant of the system was determined. The system's dynamic is slow with a very high measured time constant (τ = 1700 s). As a consequence, if one takes into account the typical duration of a nanoindentation's test, this time constant assures a temperature's variation within 0.5°C on sample for experiments at 100°C.

The dynamic behavior of the chamber has been improved with different settings. Thus, two different regulation cycles avoiding the transgression of the target temperature and offering different compromises between rapidity and accuracy of temperature adjustment are available. The first one adjusts the temperature with an accuracy of 0.5 °C on the sample, the second one has a more accurate temperature adjustment (0.2 °C) but the regulation time is longer. Currently, four indentations at 100°C can be realized within one hour.

The nanoindentation experiments at high temperatures require specific needs. That is why a particular load cycle has been built, accounting for those new parameters. The new load cycle is based on the analysis method suggested by Oliver and Pharr for experiments at room temperature. In a first step, the target temperature is adjusted inside the thermal chamber. Once the temperature stabilized, the regulation of the chamber is switched off, the temperature is measured and saved and the standard procedure for one load-unload cycle starts. At the end of the cycle the temperature is once again measured and the thermal regulation is switched on for readjusting the temperature.

Currently, we are able to perform nanoindentation experiments between 0 and 100°C. At temperatures under 0°C, some humidity problems have been encountered. With some care, experiments can be carried out between 0 and -25°C and we are working on tool enhancements that will enable us to carry out experiments between –25 °C and –50 °C.

4. SENSITIVITY ANALYSES

The correct use of a nanoindentation tool necessitates a very good understanding of its environment and the different parameters that influence the measurement results. So, another study has been carried out to analyze the quality of the results and to determine the influence of specific input and environmental parameters. The number of parameters that can be considered during such a sensitivity analysis is huge. Hence a choice of parameters that seems particularly important has been made, it will be completed by complementary studies in the future. The results obtained in this sensitivity analysis are later on used to monitor the effects of temperature on the accuracy of measurement results.

In order to carry out the sensitivity analysis, the software algorithm used to calculate Young's Modulus E and the hardness H based on the results of a load-unload cycle has been implemented in Matlab and the sensitivity of the model

to parameter variations has been estimated thanks to numerical simulation. Then, improvements have been built up in order to improve consequently our knowledge on some parameters.

Validation of the numerical Model

In a first step a comparison between simulation and experimental results has been carried out, in order to validate the chosen approach. During this step only dispersions on continuous measurements due to various noise parameters such as for example mechanical vibrations, homogeneity of the test material or surface roughness have been taken into account. In the numerical simulation these dispersions were represented by white noise (normal distribution). For the experimental part a great number of experiments has been carried out. As one can see, the results obtained by simulation are coherent with experimental results obtained for a SiO_2 sample at room temperature. The numerical model is hence validated.

Table 1. Comparison between numerical simulations and experiments

Hardness (en GPa) :

	Simulation	Experiment	Difference
Min	9.1	9.2	1%
Max	11.6	10.7	8.5%
Mean	10.18	9.94	2.4%
Standard Deviation	0.36 (3.5%)	0.3 (3%)	

Young's Modulus (en GPa) :

	Simulation	Experiment	Difference
Min	68.1	69.5	2%
Max	79.4	76.5	3.8%
Mean	73	72.2	1.1%
Standard Deviation	1.6 (2.1%)	1.4 (1.9%)	

Impact of several Parameters

After having validated the numerical model for the sensitivity analysis, the influence of four specific parameters on the accuracy of the values for E and H has been monitored. These four parameters are the frame stiffness correction K_f, Poisson's ratio v, the support spring stiffness K_s and the tip coefficients C_i used in equation (4). The support springs are the suspensions that hold the measurement column. The inaccuracy for each of these parameters has been estimated by empirical means and their influence on the E and H values has been determined as follows:

Frame stiffness K_f

A range of frame stiffness values has been sampled and it has been observed that the sensitivity of the hardness result to this parameter is very low, though for Young's Modulus result errors of 3% can be induced by the imprecision of K_f (accuracy of 25 % for this value).

Poisson's ratio ν

During a nanoindentation experiment Poisson's ratio for a given test material must be adjusted by the user in order to calculate E and H. For example Poisson's ratio for a standardized SiO_2 sample is 0.18, but the accurate knowledge of this parameter generally remains a problem for the user. During our sensitivity analysis a Poisson's ratio of $v = 0.25 \pm 0.05$ has been sampled, this range induces a 6% deviation of Young's Modulus E.

Support spring stiffness K_s

A range of values has been sampled and it has been observed that the sensitivity of the results for E and H for this parameter is extremely low. Hence, despite our little knowledge (accuracy at least ± 15 %) concerning the actual support spring stiffness K_s value, the current knowledge of this parameter is sufficient enough to obtain correct measurement results.

Tip coefficients C_i

Measurements results are influenced by all tip coefficients, but the first one is the most important. In a first approximation it can be determined with an exactitude of 25 %. A variation of 25% for C_1 induces a small deviation of the result for Young's Modulus and a deviation of 3 % for the hardness.

The Impact of Temperature on the Quality of the Results

In order to investigate the impact of higher temperatures on the result's quality, the measurement's dispersion at 80 °C has been considered. As for the numerical model's validation, a great number of experiments was carried out on a SiO_2 sample. The observed dispersion is quite similar to the dispersion observed for measurements at room temperature. So, the temperature seems to have no effect on the result's dispersion.

Later on the influence of temperature on four parameters, the frame stiffness, the support spring stiffness, the tip coefficients and the thermal drift, has been considered:

Frame stiffness K_f

The frame stiffness K_f can be modified by temperature, particularly for enveloped materials but the knowledge of this parameter remains the same as at room temperature.

Support spring stiffness K_s

The support spring stiffness changes due to temperature are in a range of 5%. As already described in the last paragraph, such a variation has no important influence on the values of E and H and can be neglected.

Tip coefficients C_i

The material of the tip is diamond. Hence, one can assume that the tip is not deformed by a temperature up to 100°C and that the effect of temperature on the tip coefficients is irrelevant.

Thermal drift

The main effect of temperature on nanoindentation experiments concerns the thermal drift correction. The nanoindentation tests at 80°C showed that the thermal drift value depends strongly on temperature. However, the dispersion of measurement results remains similar. Further studies are ongoing to determine the influence of this value on the measurement results.

5. EXPERIMENTS

In order to validate the developed thermal nanoindenter tool, first tests have been performed on two different materials: SiO_2 and a polymer, the polymethyl methacrylate plastic (PMMA). For each material, two series of tests were realized, one at room temperature and one at 80°C. The test results were compared to literature values (for the SiO_2) and to experimental results obtained with an other tool (for the PMMA).

PMMA

Nanoindentation tests on the PMMA show a Young's Modulus of 5 GPa at 25 °C and of 3.5 GPa at 80 °C. The measured hardness is 0.3 GPa at 25 °C and 0.15 GPa at 80 °C. On the Fig. 3., one can see Young's Modulus dependent on the penetration depth of the tip. At low penetration depths, the dispersion is very big, even though at higher values the results are more and more constant and reliable.
The nanoindentation results are compared to the results of an other analysis, the Dynamic Mechanical Analysis (DMA). The DMA results are coherent with nanoindentation results.

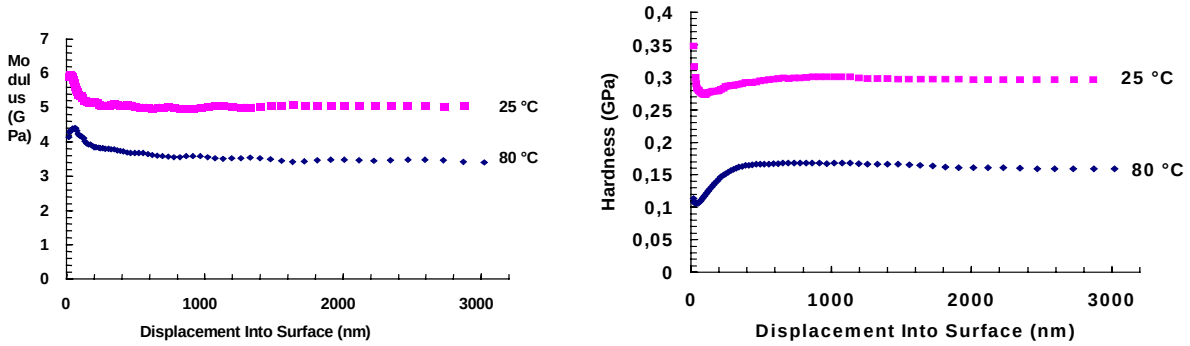

Fig. 3. Hardness and Young's modulus of PMMA versus displacement and temperature.

Siliconoxyde

Further measurements have been carried out on a SiO_2 sample once again with the aim to validate the successful use of the thermal nanoindenter tool. Each point correspond to the mean of a series of test. As one can see on Fig. 4. we observe an increase of Young's Modulus of 11 GPa between 25 °C and 100 °C and a hardness increase of 1,7 GPa in the same temperature range. These results seem surprising but the not-so-usual rise of mechanical properties in this temperature range for an SiO_2 sample has already been observed during other measurements and is described in literature [8].

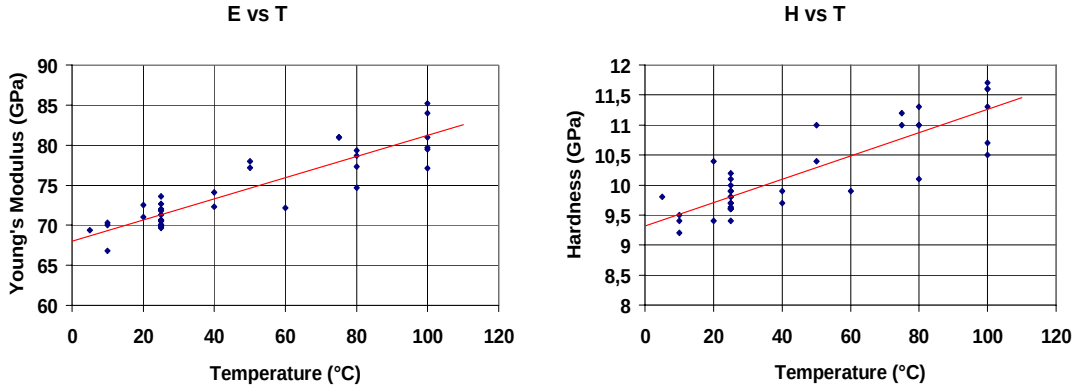

Fig.4. Hardness and Young's modulus versus temperature on SiO_2

In order to insure that this rise is due to physical properties of SiO_2 and not to a bias induced by temperature on the tool, another test has been performed on TA6V, a titanium alloy which is known for its thermal stability in this temperature range. The measurements show no modification of its mechanical properties. Hence, these results confirm the successful use of the thermal nanoindenter tool.

CONCLUSION

The material properties change with temperature. Hence it is very interesting to determine them at different temperatures. Thanks to the development of a new thermal nanoindenter tool, CNES has hugely improved its skills concerning the characterization of material properties. Until now, the mechanical microcaracterisation of MEMS and materials by nanoindentation could only be performed at room temperature. The newly developed thermal nanoindenter tool enables us to perform nanoindentation experiments between 0 and 100°C. With some care, experiments can be carried out between 0 and -25°C and we are working on tool enhancements that will enable us to carry out experiments between –25 °C and –50 °C.

An important point in the nanoindentation field is the quality of measurement results which depends on inputs and environmental parameters. The evaluation of the influence of these parameters on the values of Young's modulus *E* and the hardness *H* has been done by a sensitivity analysis. In order to carry out the sensitivity analysis, the software

algorithm used to calculate *E* and *H* based on the results of a load-unload cycle has been implemented in Matlab and after the validation of the chosen approach, the sensitivities of different parameters have been estimated thanks to numerical simulation. The results at room temperature are sensitive to the frame stiffness, Poisson's ratio and the tip coefficients. During temperature tests the most important parameter is the thermal drift coefficient.

The thermal indentation tool has been used to carry out tests on two different materials: a polymer, PMMA, and SiO_2. Both tests showed an evolution of E and H in the temperature range from 0 to 100 °C. The obtained results are coherent with literature values and results obtained with an other measurement method. Tests on MEMS are still ongoing.

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