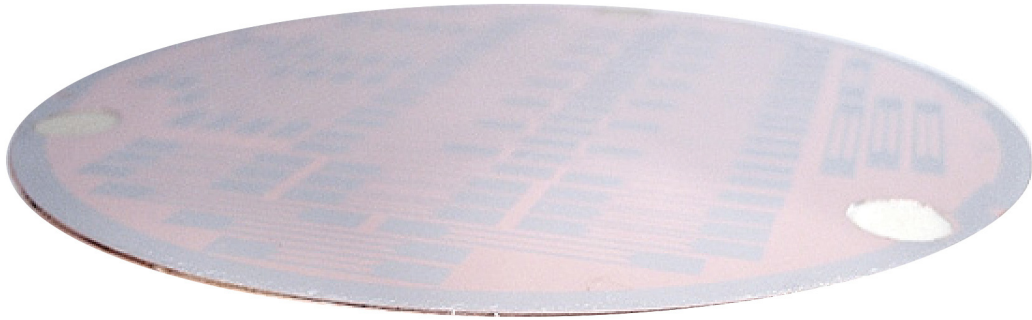


**Bridging Nanoindentation
And Macro Tensile Testing**



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INTRO:

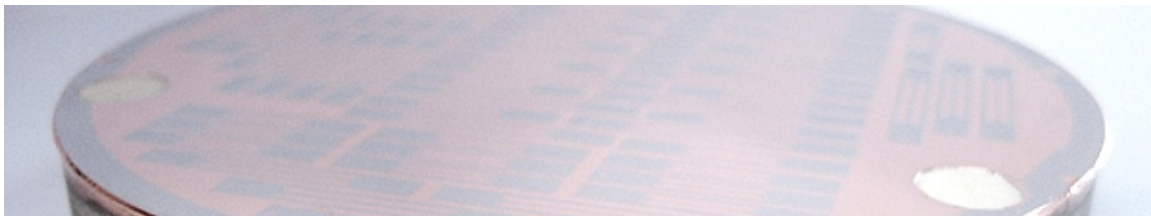
Beginning at the end of the 19th century, thin film coatings have become a large interest in several different industries today. Thin film coatings are used in many active and passive applications, such as optical coatings and electronic devices. Through this technique, materials are able to obtain characteristics that cannot be acquired through any treatments to the material itself, such as wear and corrosion resistant coatings. Also, the benefits for the thin film coatings are its ability to be used in small scale devices (i.e. integrated circuits and magnetic storage systems), physical properties that are dependent on its small scale (i.e. optics), and cost benefits. In the electronic devices, for example, thin film coatings are widely used on inductors because of its versatility in applying coatings on their diverse, but necessary, geometries. The requirements to go to smaller dimensions and more complex geometry have created a gap between knowledge of materials in a macro range versus what is accessible through somewhat newer technique such as nanoindentation.

IMPORTANCE BETWEEN NANOINDENTATION AND MACRO TENSILE TESTING

The properties of a material, such as yield strength, elastic modulus, stress and strain values, and ultimate tensile strength are usually found by using the method of tensile testing. Unfortunately, thin film coatings are impossible to test using these conventional methods. Tensile testers are normally used for bulk samples of at least millimeter dimensions. Thin film coatings of a nanometer to several micrometers are more fragile and easily deformable. They are also most of the time attached to a more bulky substrate. It is therefore impossible to get direct standard mechanical properties using tensile technique. In some cases, even bulk samples cannot be destroyed or easily tested using this technique. In this application, we have used nanoindentation technique to provide a data that closes the gap to standard macro tensile data.

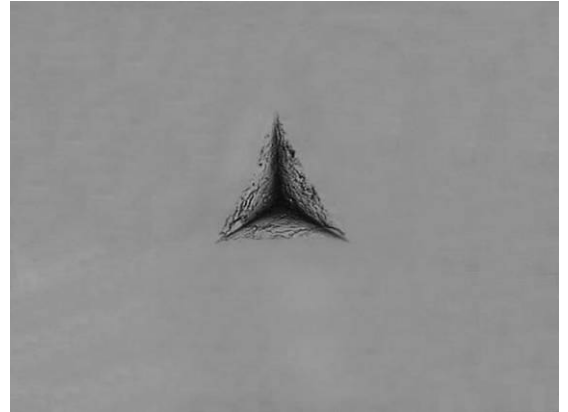
MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester in its Nanoindentation mode is used to provide measurements of hardness, elastic modulus and stress-strain data of the thin film coating. Each sample has a thickness of 30 μm on a very thick metal wafer. A 10 μm spherical tipped stylus is used at a maximum load of 140mN to create an indent on the coating. The depth and load measurements are taken to produce the hardness, elastic modulus and stress-strain values of the samples. Five tests were done on each sample to show the reproducibility of the results.



MEASUREMENT PRINCIPLE

The Nanoindentation test is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until partial or complete relaxation occurs. This procedure is performed repetitively; at each stage of the experiment the position of the indenter relative to the sample surface is precisely monitored with a linear variable differential transformer (LVDT).



For each loading/unloading cycle, the applied load value is plotted with respect to the corresponding position of the indenter. The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data.

The NHT is especially suited to load and penetration depth measurements at nanometer scales and has the following specifications:

Maximum displacement	:	25 μm
Depth Resolution (Theoretical)	:	0.003 nm
Depth Resolution (Noise Level)	:	0.05 nm
Maximum force	:	400 mN
Load Resolution (Theoretical)	:	0.08 μN
Load Resolution (Noise Floor)	:	2 μN

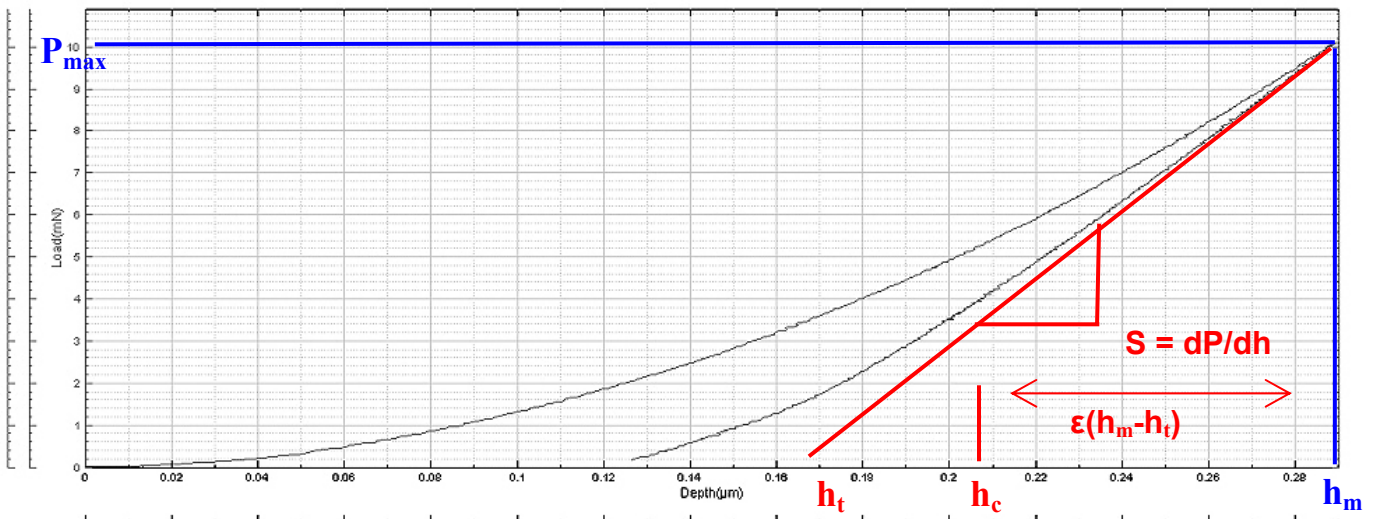
Analysis of Indentation Curve

A typical load/displacement curve is shown below, from which the compliance $C = 1/S$ (which is the inverse of the contact stiffness) and the contact depth h_c are determined after correction for thermal drift.

A simple linear fit through the upper 1/3 of the unloading data intersects the depth axis at h_t . The stiffness, S , is given by the slope of this line. The contact depth, h_c , is then calculated as:

$$h_c = h_m - \varepsilon(h_m - h_t)$$

where ε depends on the investigated material.



In practice, a more meticulous approach is used where a power law function is used to describe the upper 80% of the unloading data.

$$P = P_{\max} \left(\frac{h - h_0}{h_m - h_0} \right)^m$$

where the constants m and h_0 are determined by a least squares fitting procedure. The contact stiffness S ($=1/C$) is given by the derivative at peak load:

$$S = \left(\frac{dP}{dh} \right)_{\max} = mP_{\max} \left[\frac{(h_m - h_0)^{m-1}}{(h_m - h_0)^m} \right] = mP_{\max} (h_m - h_0)^{-1}$$

and the tangent depth, h_t , is thus given by:

$$h_t = h_m - \frac{P_m}{S}$$

The contact depth, h_c , is then:

$$h_c = h_m - \varepsilon(h_m - h_t)$$

where ε now depends on the power law exponent, m . Such an exponent can be summarized for different indenter geometries:

Elastic Indentation Behavior (indenter geometry)	m (power law exponent)	ε
Flat	1	1
Paraboloid	1.5	0.75
Conical	2	0.72

Calculation of Young's Modulus and Hardness

(i) Young's Modulus

The reduced modulus, E_r , is given by:

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} = \frac{\sqrt{\pi}}{2} \frac{1}{C} \frac{1}{\sqrt{A_c}}$$

which can be calculated having derived S and A_c from the indentation curve using the area function, A_c being the projected contact area.

The Young's modulus, E , can then be obtained from:

$$\frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

where E_i and ν_i are the Young's modulus and Poisson coefficient of the indenter and ν the Poisson coefficient of the tested sample.

(ii) Hardness

The hardness is determined from the maximum load, P_{\max} , divided by the projected contact area, A_c :

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

Test conditions and procedure

The following indentation parameters were used:

Test Parameters

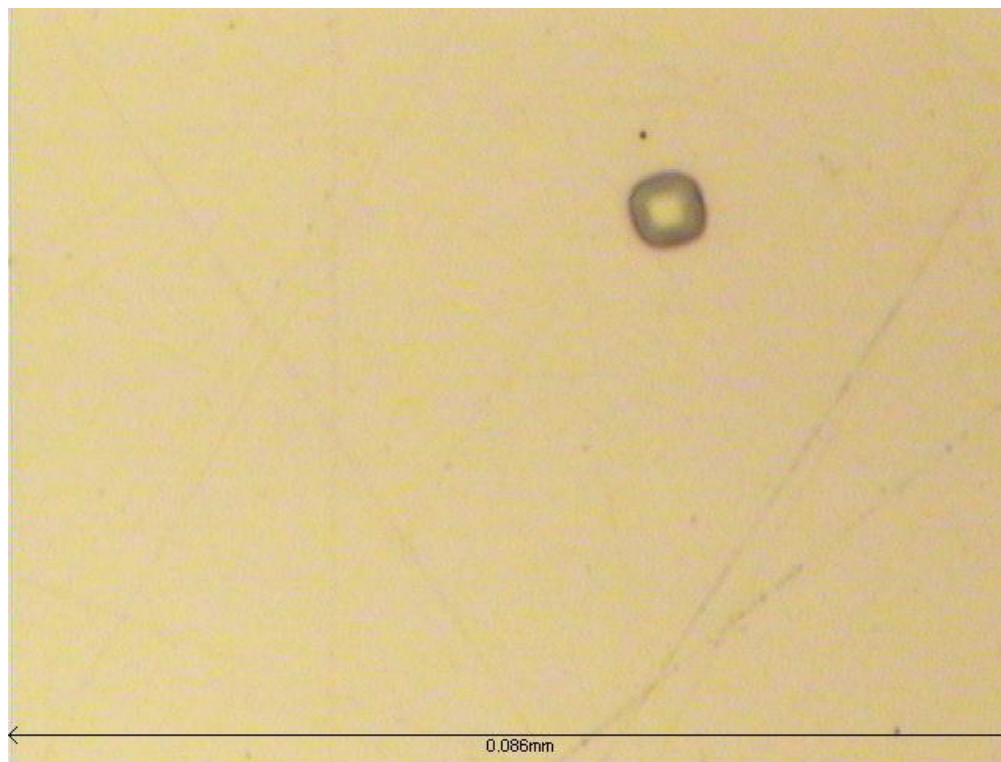
Maximum force (mN)	140
Loading rate (mN/min)	280
Unloading rate (mN/min)	280
Pause (s)	30
Computation Method	ASTM E-2546 & Martens Hardness
Indenter type	Conical 10micron 90Degree Diamond

Results

This section includes a summary table that compares the main numerical results for the different samples, followed by the full result listings, including each indentation performed, accompanied by micrographs of the indentation, when available. These full results present the measured values of Hardness and Young's modulus as the penetration depth (d) with their averages and standard deviations. It should be considered that large variation in the results can occur in the case that the surface roughness is in the same size range as the indentation.

Full result tables

Metal Type 1		
	H [GPa]	E [GPa]
1	2.683	135.528
2	2.697	124.344
3	2.717	128.854
4	2.752	129.924
5	2.657	129.741
Average	2.701	129.678
Std deviation	0.036	3.982



**Figure 1 : Micrograph of indentation – Metal Type 1
500x magnification (image width 0.086mm)**

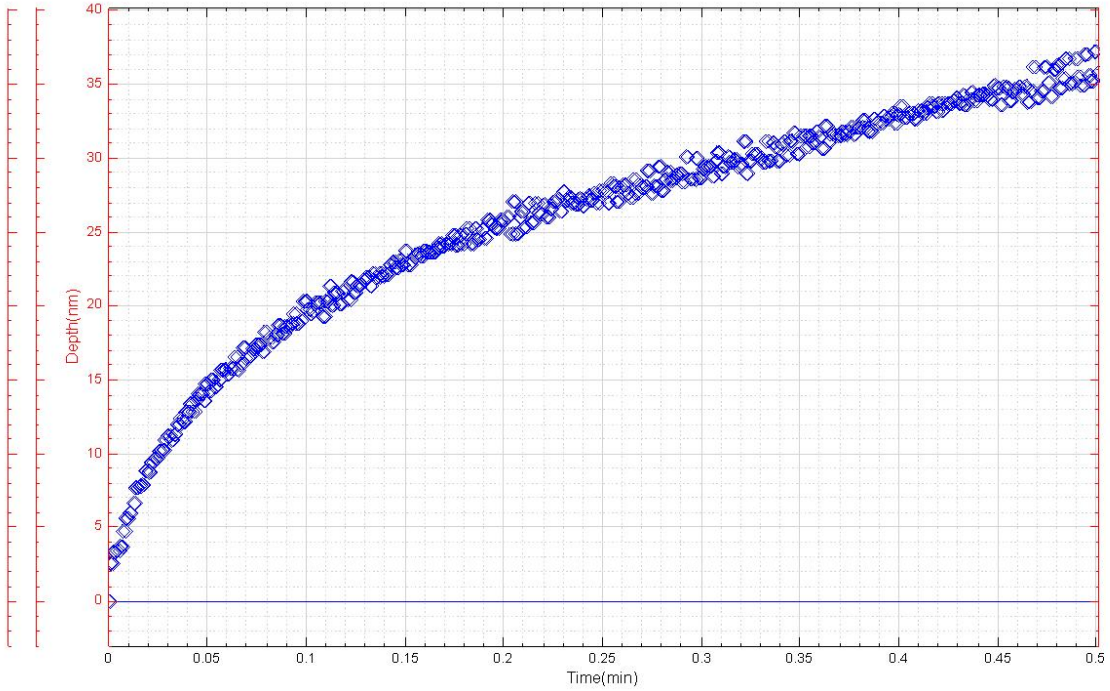


Figure 2 : Creep Graph - Metal Type 1

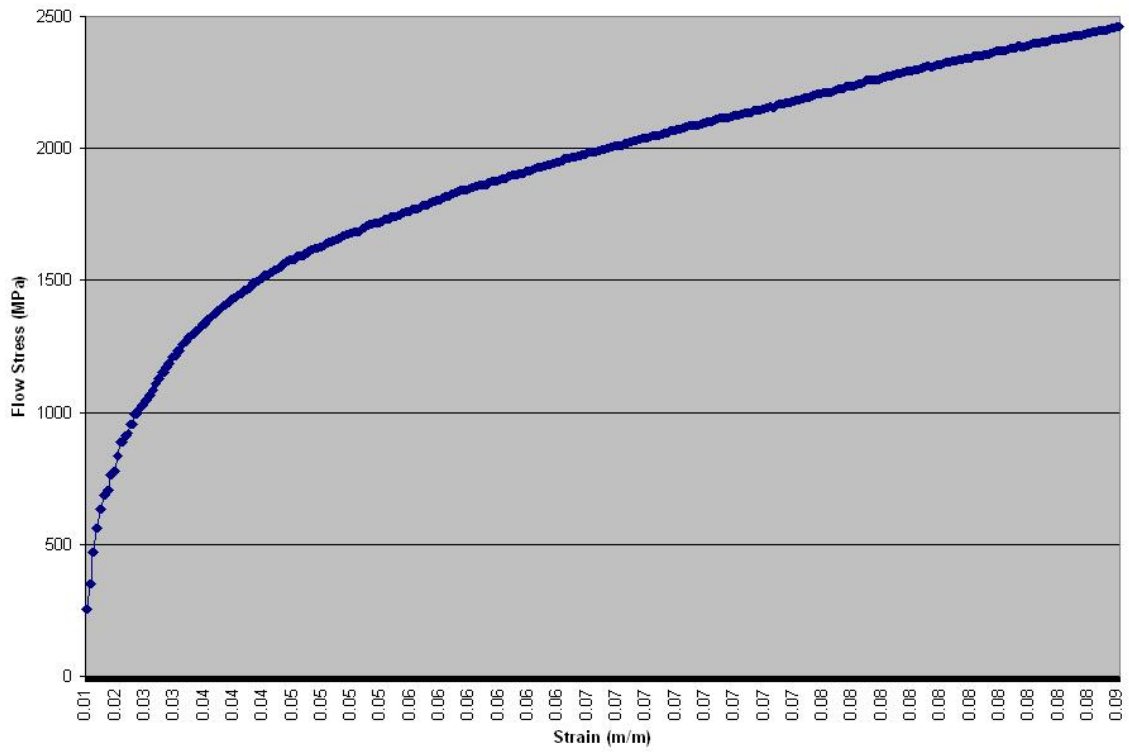
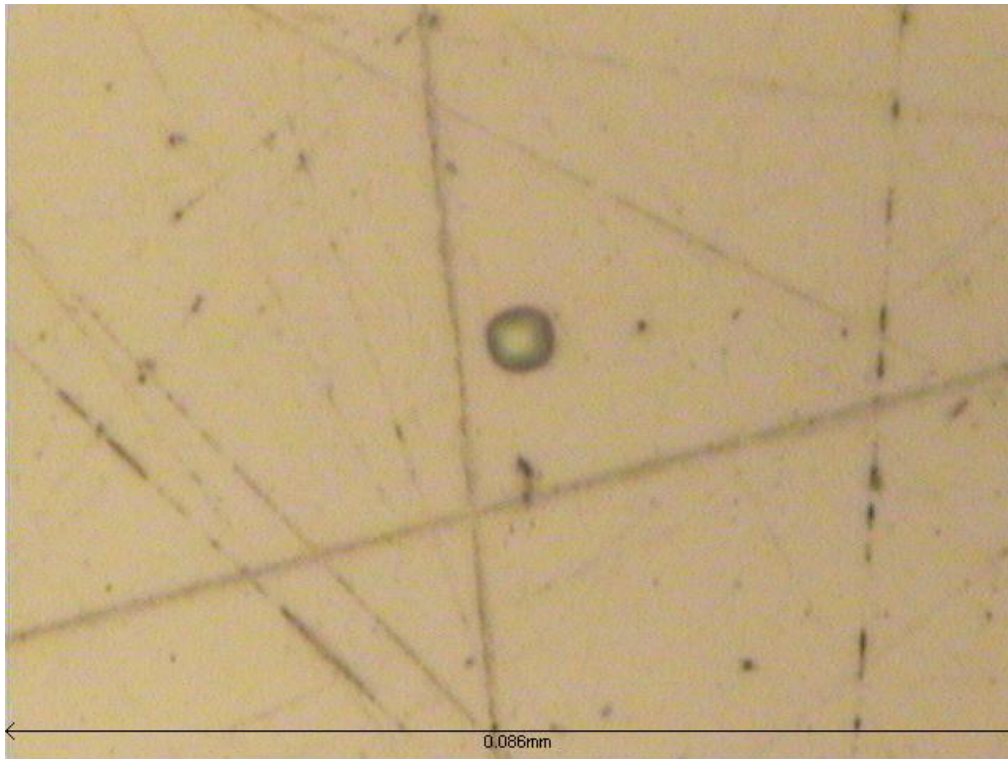


Figure 3 : Stress-Strain Curve - Metal Type 1

Metal Type 2		
	H [GPa]	E [GPa]
1	3.505	125.438
2	3.584	169.705
3	3.581	167.741
4	3.521	161.034
5	3.644	152.636
Average	3.567	155.311
Std deviation	0.056	17.986



**Figure 4 : Micrograph of indentation - Metal Type 2
500x magnification (image width 0.086mm)**

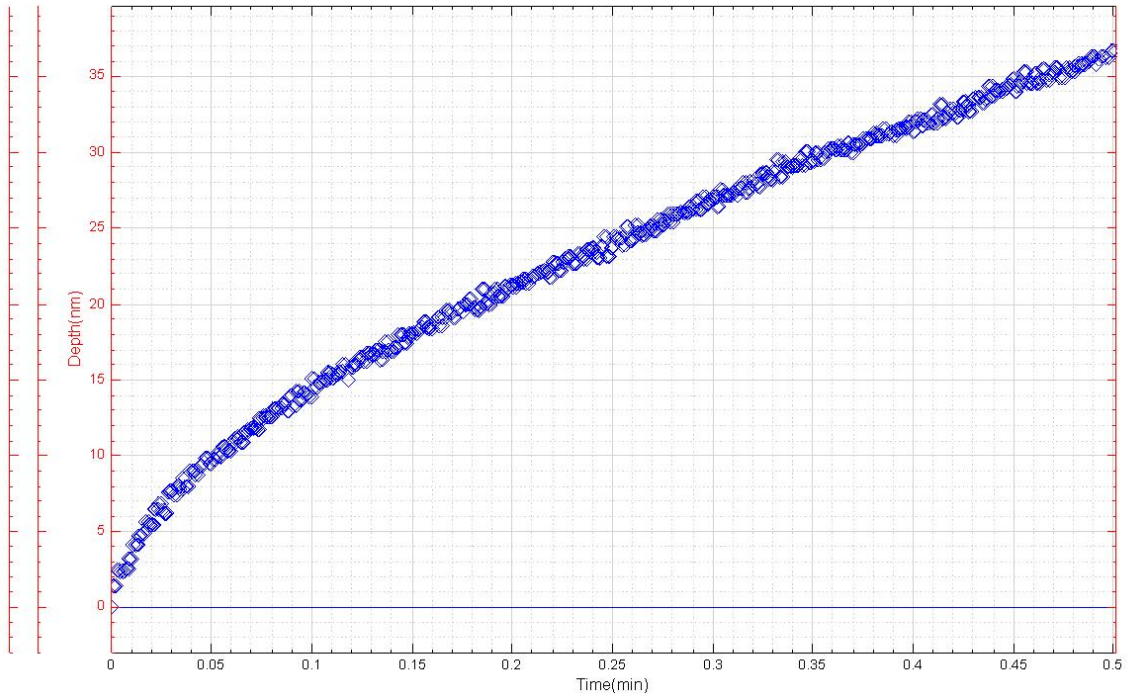


Figure 5 : Creep Graph - Metal Type 2

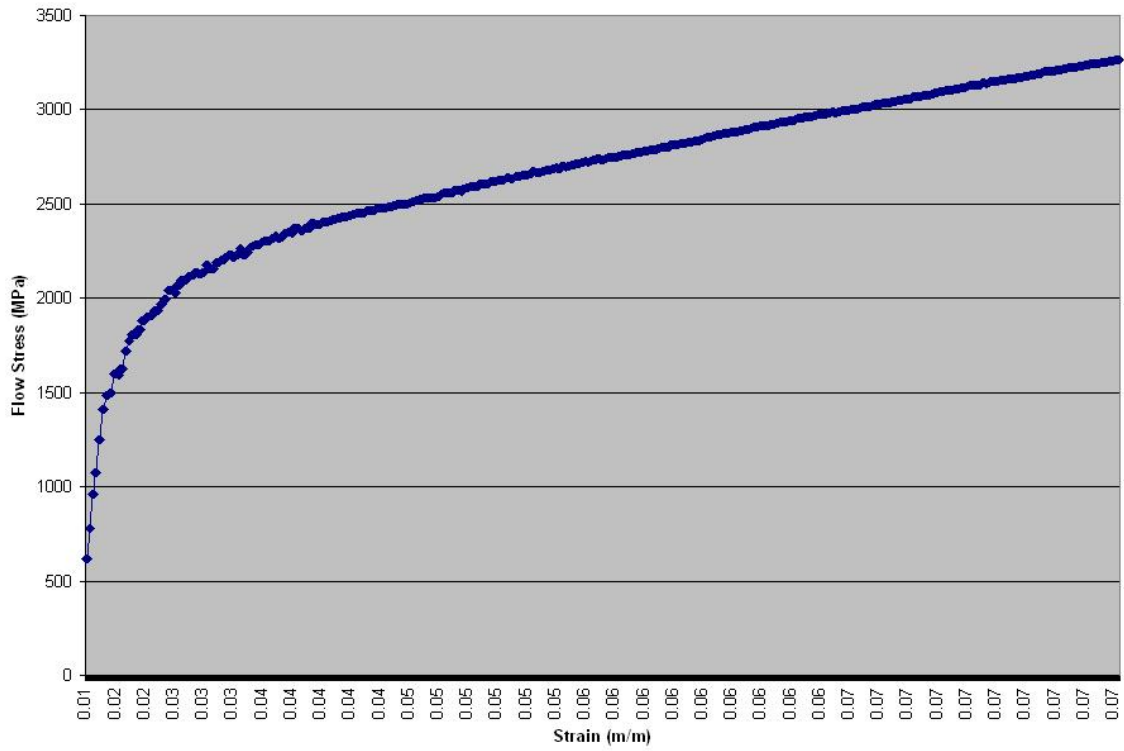


Figure 6 : Stress-Strain Curve - Metal Type 2

Summary table of main numerical results

Hardness and Elastic Modulus

Sample	Hardness [GPa]	Young's Modulus [GPa]
Metal Type 1	2.701 ± 0.036	129.7 ± 4.0
Metal Type 2	3.567 ± 0.056	155.3 ± 19.8

Rate of creep after initial large movement.

This factor is directly related to the ductility measured by Tensile measurements.

Sample	Linear Creep (nm/min)
Metal Type 1	34
Metal Type 2	50

Discussion:

Taking the rate of creep when it becomes more linear is a good indicator of ductility as it is measured by tensile measurements. We believe that measuring this creep at various loads, during a single loading, may allow to see a change in the change of rate that could be linked to the ultimate tensile strength. This is another parameter measured during tensile testing. To facilitate this test, the Nanoindentation software will be modified to allow this automatically and we will investigate this possibility in a future application note.

Yield Point from Stress-Strain Curve

Sample	Yield Point (MPa)
Metal Type 1	1550
Metal Type 2	2100

Discussion: We believe that the inflection point seen in the stress strain curve can be related to the yield point of a tensile test. Further testing will be needed to prove this correspondence.

CONCLUSION:

In addition to proving hardness data, Nanoindentation can provide some of the important tensile test parameters such as elastic modulus and quantitative information on ductility. We have also shown some promising options for deriving links to parameters such as yield point and ultimate tensile strength. Of course, further testing will be required for full correlation to tensile data but nanoindentation technique is definitively a powerful quantitative method of obtaining mechanical properties for thin film coatings research. Learn more about [Nanoindentation](#)