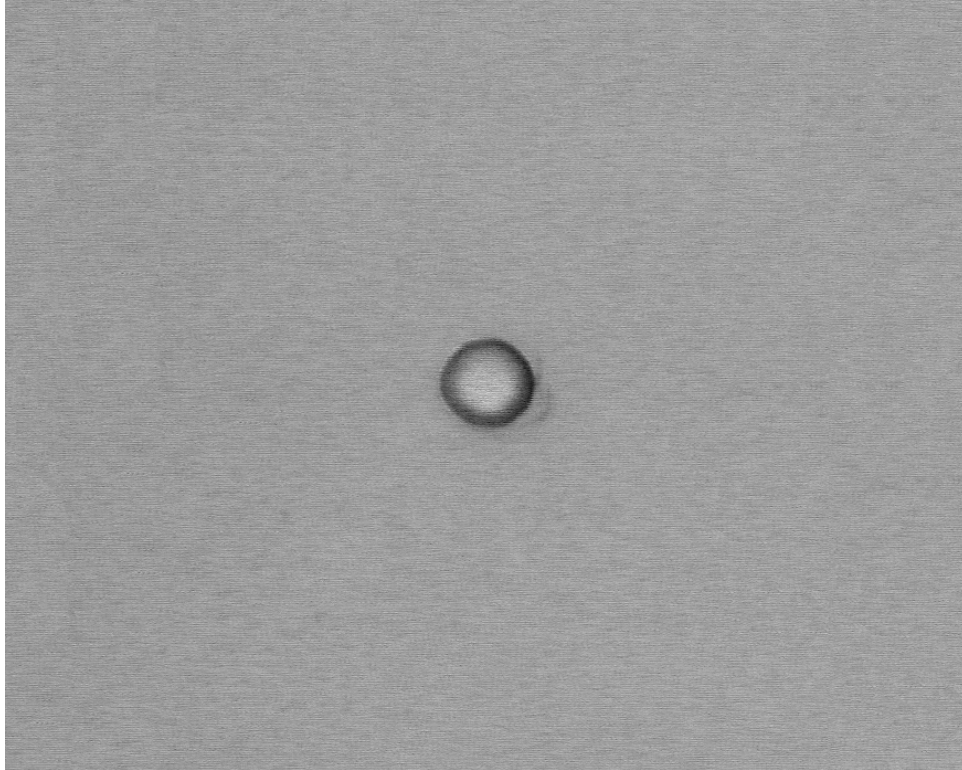


**CYCLICAL NANOINDENTATION  
STRESS-STRAIN MEASUREMENT**



Prepared by  
**Jorge Ramirez**

## INTRO

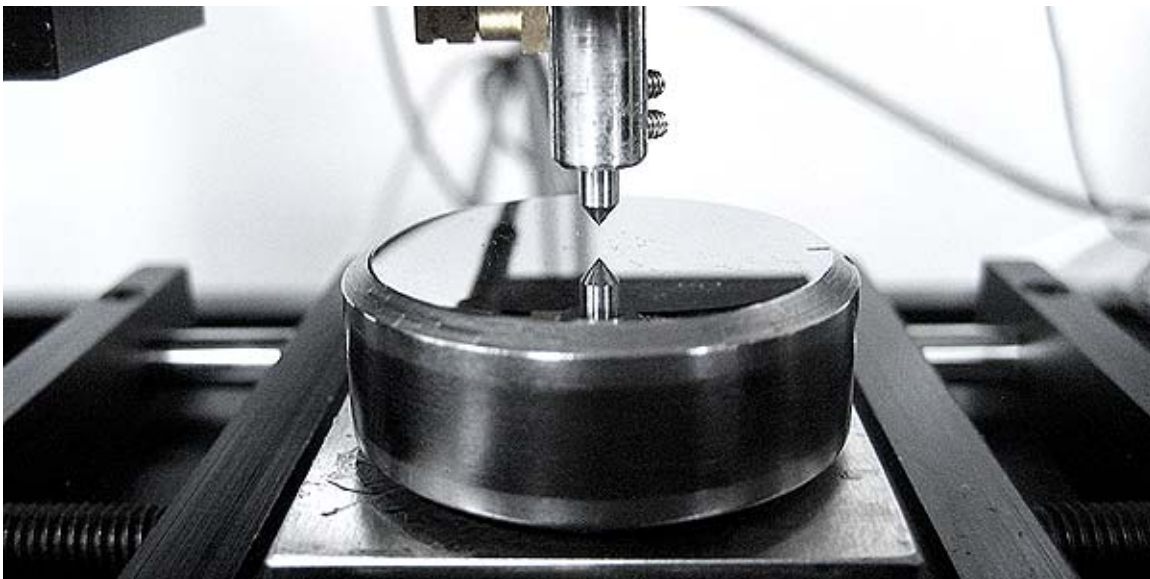
The Stress-Strain curve obtained by Nanoindentation reveals the correlation of "Stress" and "Strain" of a given material under nano controlled loads. Unlike the traditional Tensile Testing method of obtaining Stress-Strain curve data, which gives data at a macro level, the Nanoindentation method provides vital Stress-Strain curve data at nano level scale without the large and intense machinery. The Stress-Strain curve of various materials will vary widely. Ultimately, the Stress-Strain curve provides crucial information on the threshold between elastic and plastic-elastic behavior zone as the sample is subject to increasing loads.

## IMPORTANCE OF NANOINDENTATION FOR QC AND R&D APPLICATIONS

Stress-Strain data is an important study into the surface characteristic of bulk material. Nanoindentation provides a reliable and user-friendly method to quickly investigate stress-strain data. Furthermore, measuring stress-strain behavior on the nano scale opens the door to the study of important properties on coatings and particles in composite materials among others. Therefore, in addition to hardness, elastic modulus, creep information, fracture toughness etc, Nanoindentation can provide information on elastic limit and yield strength.

## MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode, is used to study Hardness and Elastic Modulus versus depth and Stress-Strain data on a standard steel sample. The steel sample was chosen for its commonly recognized Stress-Strain curve to display the control and accuracy of the nano scale Stress-Strain data. A spherical tip of 5micron in radius was used for the test because it could reach stresses high enough to be higher than the elastic limit for steel.



## MEASUREMENT PRINCIPAL

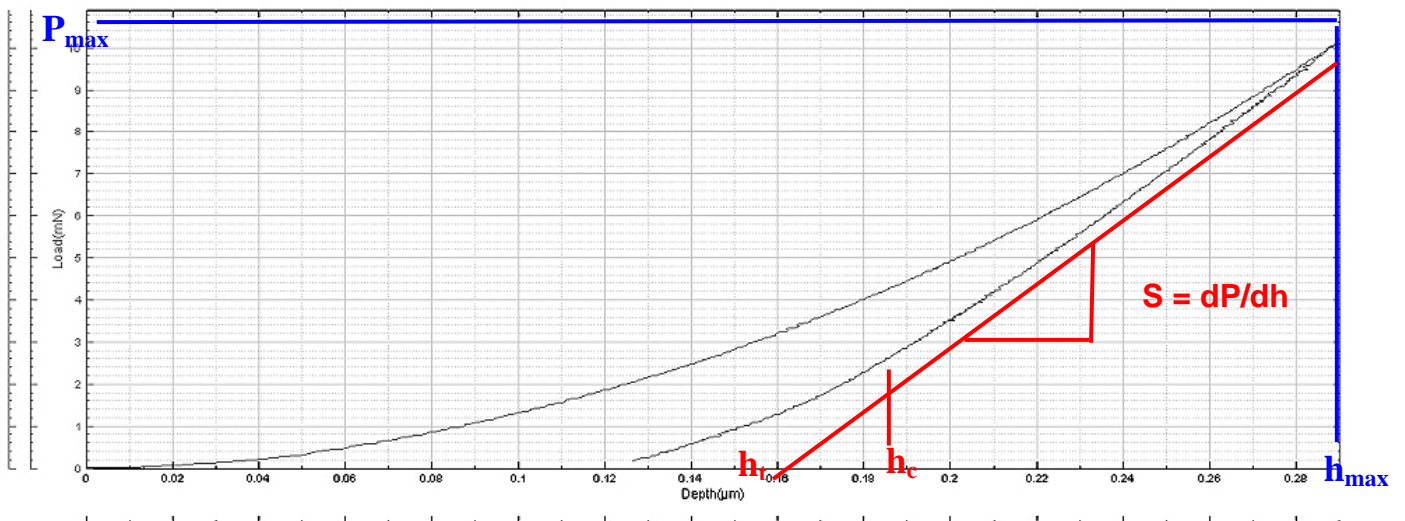
Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. The load is applied by a piezo actuator and the load is measured in a controlled loop with a high sensitivity load cell. During the experiment the position of the indenter relative to the sample surface is precisely monitored with high precision capacitive sensor.

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

Maximum displacement (Dual Range)	: 50 $\mu\text{m}$ or 250 $\mu\text{m}$
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 $\mu\text{N}$
Load Resolution (Noise Floor)	: 1.5 $\mu\text{N}$

## Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.



## Hardness

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

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

## Young's Modulus

The reduced modulus,  $E_r$ , is given by:

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

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  $\nu_i$  are the Young's modulus and Poisson coefficient of the indenter and  $\nu$  the Poisson coefficient of the tested sample.

### How are these calculated?

A power-law fit through the upper 1/3 to 1/2 of the unloading data intersects the depth axis at  $h_i$ . The stiffness,  $S$ , is given by the slope of this line. The contact depth,  $h_c$ , is then calculated as:

$$h_c = h_{\max} - \frac{3P_{\max}}{4S}$$

The contact Area  $A_c$  is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is  $A_c=24.5h_c^2$  For Cube Corner indenter, the area function is  $A_c=2.60h_c^2$  For Spherical indenter, the area function is  $A_c=2\pi Rh_c$  where  $R$  is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant  $E$ , given the formula:  $\sigma = E\epsilon$  where  $\sigma$  is the stress,  $E$  is the elastic modulus of the material, and  $\epsilon$  is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate

$$\sigma = \eta \frac{d\epsilon}{dt}$$

relationship can be given as,

where  $\sigma$  is the stress,  $\eta$  is the viscosity of the material, and  $d\epsilon/dt$  is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen. Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip might be a better choice.

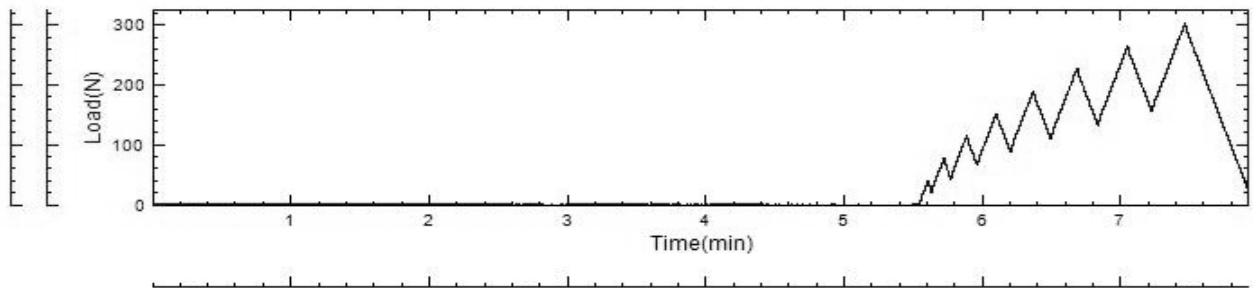
### Other tests possible includes the following:

Work of Indentation, Volume of indent, Plastic & Elastic Work, Visco Elastic , Creep, Compression and others.

## TEST CONDITIONS & PROCEDURES

The following indentation parameters were used:

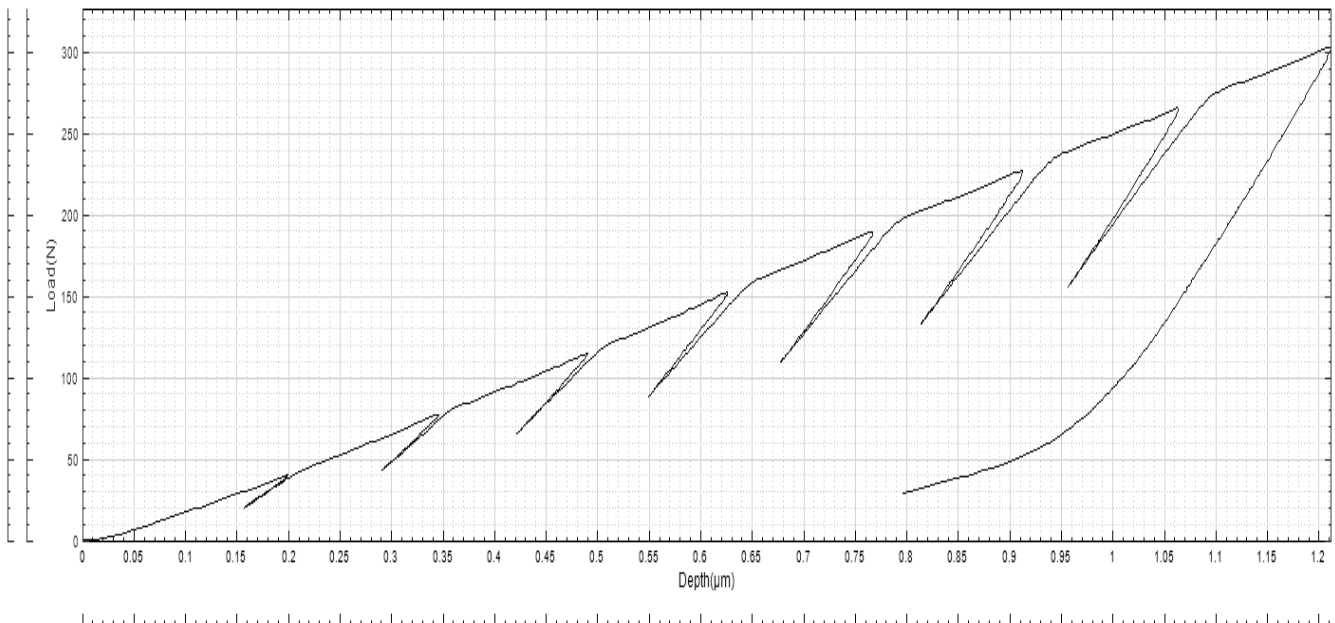
Maximum force (mN)	300
Loading rate (mN/min)	600
Unloading rate (mN/min)	600
Creep (s)	0
Computation Method	ASTM E-2546 & Martens Hardness
Indenter type	5micron Spherical tip
# Cycles	8



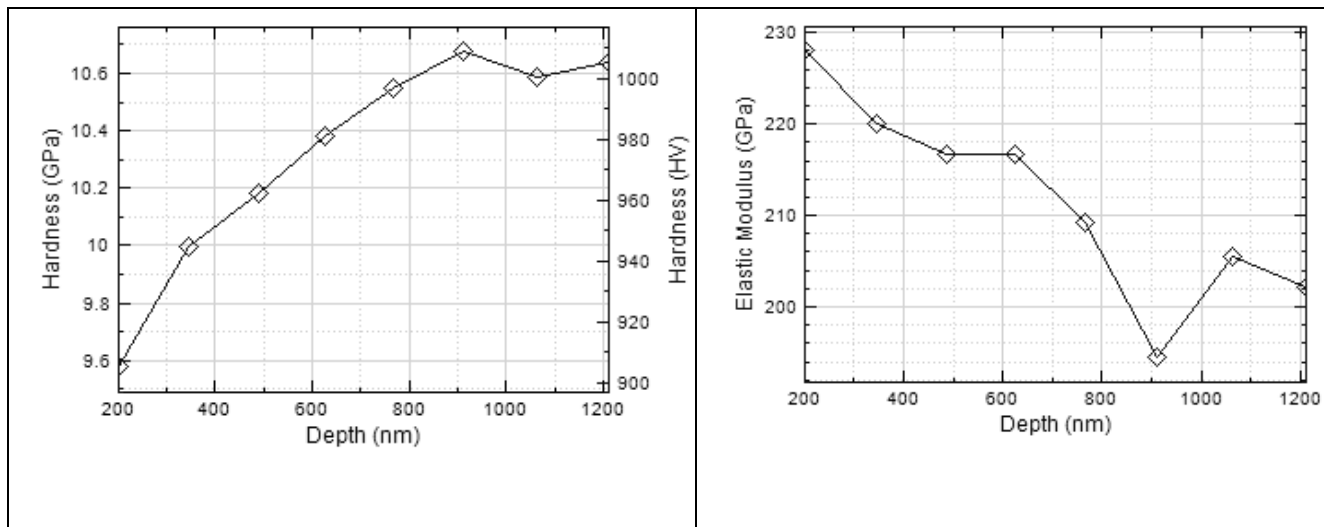
**Loading Versus Time Curve that was applied for this test**

## RESULTS

The increase in load cycling at the same position provides the following depth vs. load curve where each of the cycles are well identified.



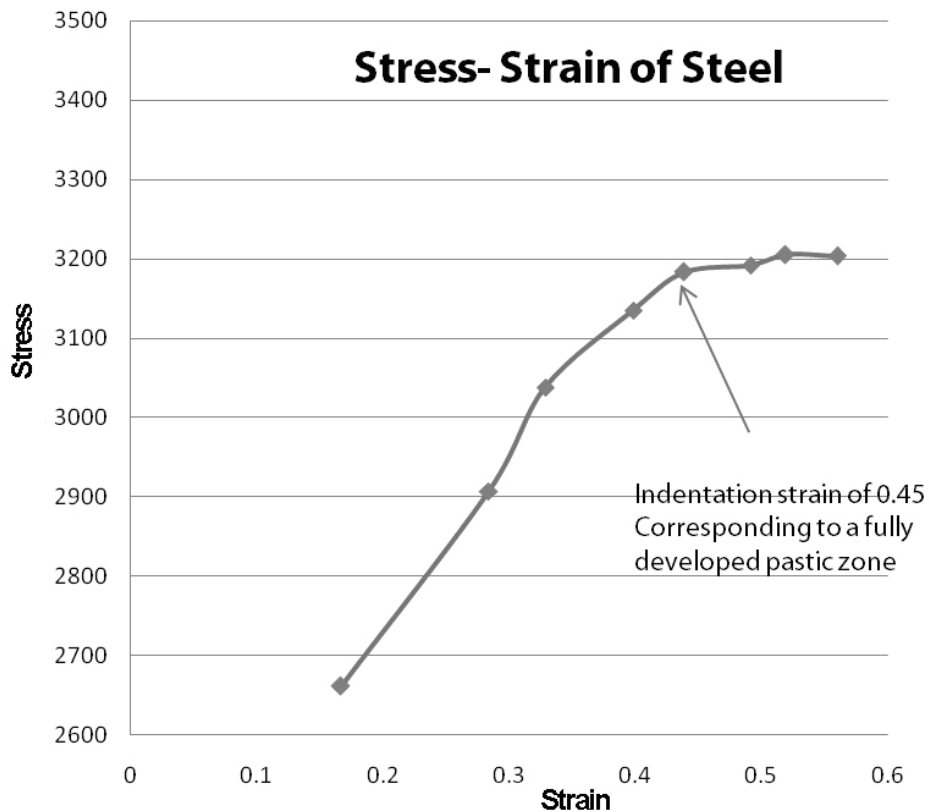
Each of the cycles can be considered as being one indent done at its own depth. Therefore hardness and elastic modulus versus depth can be plotted directly from the data obtained for each cycle.



It is also possible to plot stress vs. strain from the information obtained at each of the cycle. The maximum load applied at each cycle and the maximum depth at each cycle allows to calculate the actual maximum stress that was applied to each specific cycle. Strain is calculated from the residual depth at each cycle which is calculated from the partial unloading. This allows to calculate the radius of the residual imprint which is divided by the radius of the tip used to give the strain factor.

Cycle #	Load [ mN ]	Max Depth [ nm ]	Stress [ GPa ]	Residual Depth [ nm ]	Strain
1	40.34	193	2661	70	0.166745
2	77.4	339	2907	206	0.283947
3	115.03	482	3038	278	0.328799
4	152.44	619	3135	414	0.398427
5	189.8	759	3183	507	0.438769
6	227.18	906	3192	646	0.491637
7	265.61	1055	3205	727	0.519287
8	302.65	1203	3203	858	0.560137

Plotting the stress versus strain data provides a crucial set of data related to the material under test. First, it is possible to clearly see the elastic and plastic zone and measure the stress at this transition point. It is worth noting that 0.4 for strain is a value often found to be consistent with a plastic zone that is fully developed. 3150GPa can be taken as the elastic limit for this sample.



**CONCLUSION:**

In conclusion, we have shown that the Nanovea Mechanical Tester, in Nanoindentation Mode, can be used to provide reliable stress-strain data. Using a spherical tip and increasing cyclic loads allows measuring of the properties of the material under increased stress. The stress-strain data provided by Nanoindentation allow identifying the elastic limit of the material. It is worth noting that the information was gathered from the first 1.2micron of the surface. It is possible to change the radius of the probe and the loads to test various materials at controlled depths. The Nanovea Mechanical Tester can provide these tests from the sub mN range to 200N.

We have also shown that it is possible to use the increasing load cycles to measure mechanical properties versus depth. The main parameters are hardness and elastic modulus vs. depth. This can be very useful in measuring case hardening and it would eliminate the need to prepare a specific cut sample by only requiring one indent instead of multiple. At low load it could also be performed on the final product because the indent is small enough to not be seen by customers. The down side is that the hardness data will be difficult to be compared with traditional case hardening data. However, the change of hardness versus depth trend will match traditional case hardening data.

The multi-cycle test can also be very useful on measuring hardness and elastic modulus versus depth in the case of thin and thick coatings. A sharper Berkovich or Vickers tip would be recommended for these tests. It is worth noting also that the multi-cycle test can be performed at constant load. This can be useful to study fatigue in some materials or to eliminate the effect of porosity in some materials such as bones to obtain the true elastic modulus. To learn more about [Nanoindentation](#).