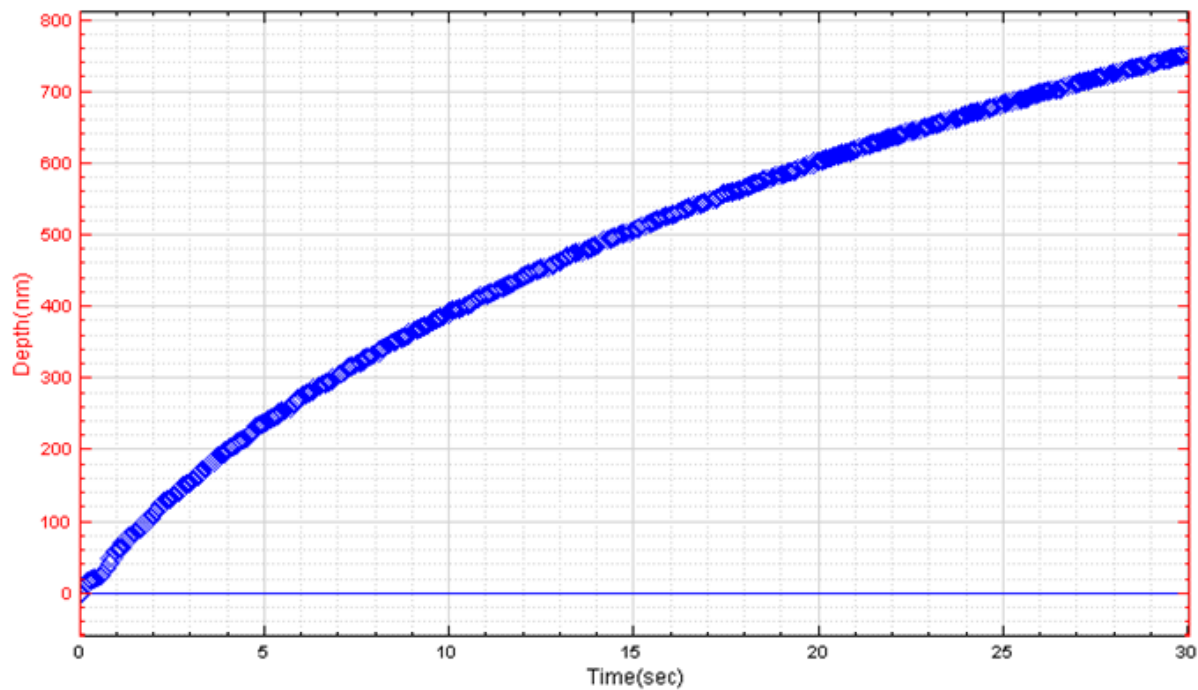


NANOINDENTATION CREEP MEASUREMENT



Prepared by
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INTRO

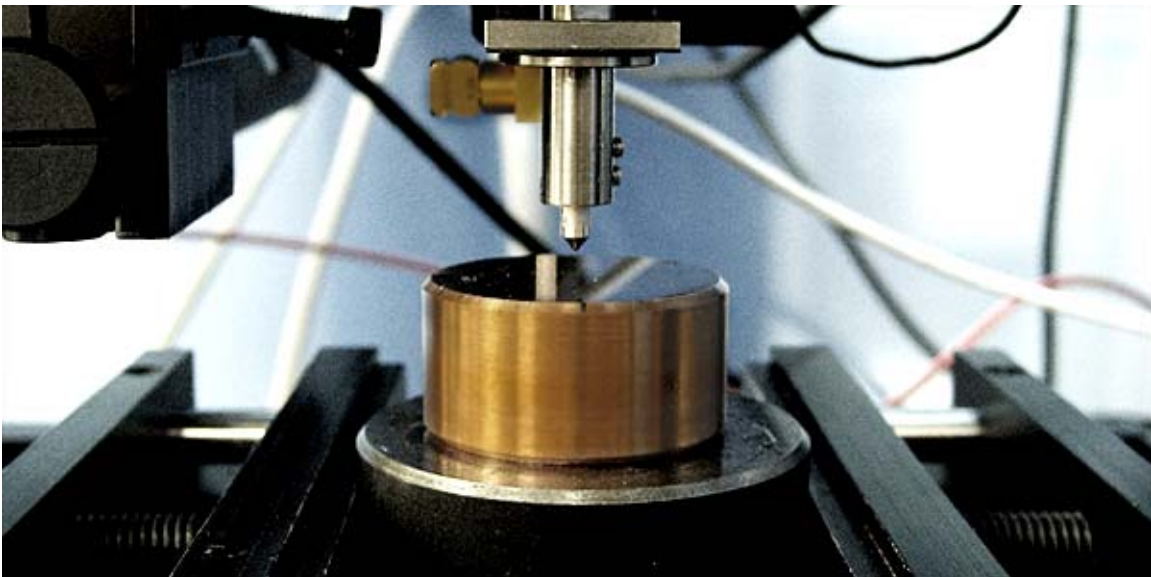
Creep can be characterized as the result of a solid material that is slowly and permanently deformed under the influence of stress. Deformation results from the consistent stress below the yield strength of the material. The amount of applied stress and its duration can eventually lead to the failure of the given material. Ultimately, it is for this reason that Creep measurement provides crucial information to study material behavior. More importantly, nano & micro advances with materials require mechanical characterization unattainable through traditional Creep measurement methods intended for large bulk materials.

IMPORTANCE OF NANOINDENTATION FOR QC AND R&D APPLICATIONS

Nanoindentation Creep measurement is critical when Creep measurement data is limited to a small area or feature, such as thin films and small micro features. The highly controlled applied loads of the Nanoindentation system can provide sensitive Creep displacement measurements on material with extremely low Creep rates. Therefore also providing crucial Creep information on large bulk materials traditionally measured at the macro scale. Nanoindentation provides a reliable and user-friendly method to quickly investigate Creep data in addition to an endless list of additional measurements including: hardness, elastic modulus, compression, fracture toughness and many others.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Nanoindentation mode, is used to study the comparative Creep between a polymer, copper and steel sample. The samples were chosen for their commonly recognized Creep data to display the control and accuracy at the nano scale. A Berkovich tip was used for the test but other tip geometries could have been used.



MEASUREMENT PRINCIPAL

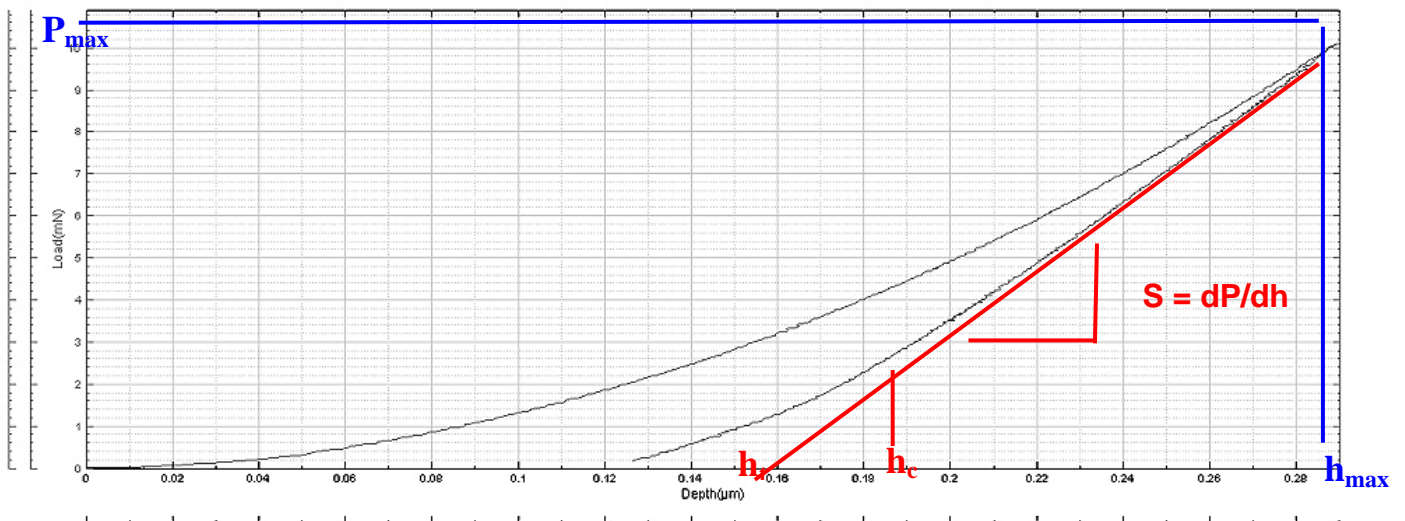
Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set 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 μm or 250 μm |
| Depth Resolution (Theoretical) | : 0.003 nm |
| Depth Resolution (Noise Level) | : 0.05 nm |
| Maximum force | : 400 mN |
| Load Resolution (Theoretical) | : 0.03 μN |
| Load Resolution (Noise Floor) | : 1.5 μN |

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 :

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

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 ν_i are the Young's modulus and Poisson coefficient of the indenter and ν 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 σ is the stress, E is the elastic modulus of the material, and ϵ 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

relationship can be given as,
$$\sigma = \eta \frac{d\epsilon}{dt}$$
 where σ is the stress, η 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.

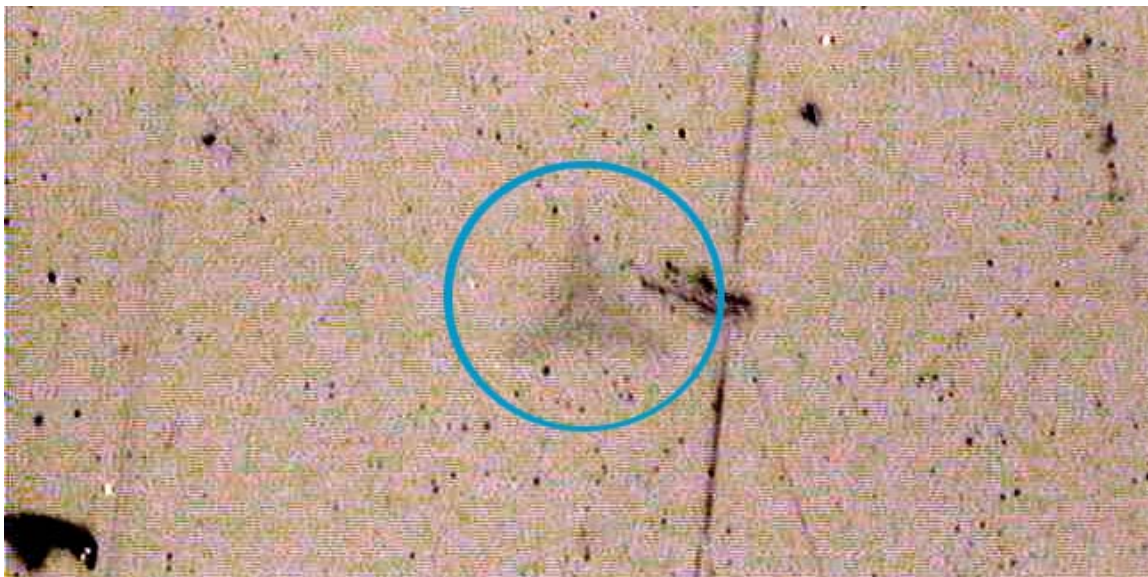
Other tests possible includes the following:

Stress-Strain & Yield Stress, Fracture Toughness, Compression strength and Fatigue testing and many others.

TEST CONDITIONS & PROCEDURES

The following indentation parameters were used:

| | |
|-----------------------------------|------------------------------|
| Maximum force (mN) | 20 |
| Loading rate (mN/min) | 40 |
| Unloading rate (mN/min) | 40 |
| Creep (s) at maximum force | 30 |
| Computation Method | ASTM E-2546 & Oliver & Pharr |
| Indenter type | Berkovich Diamond |



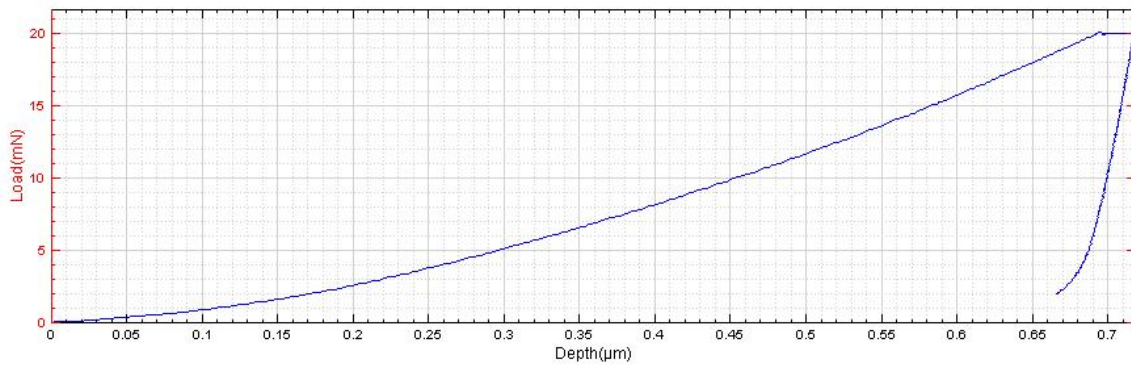
RESULTS

Summary table of main numerical results

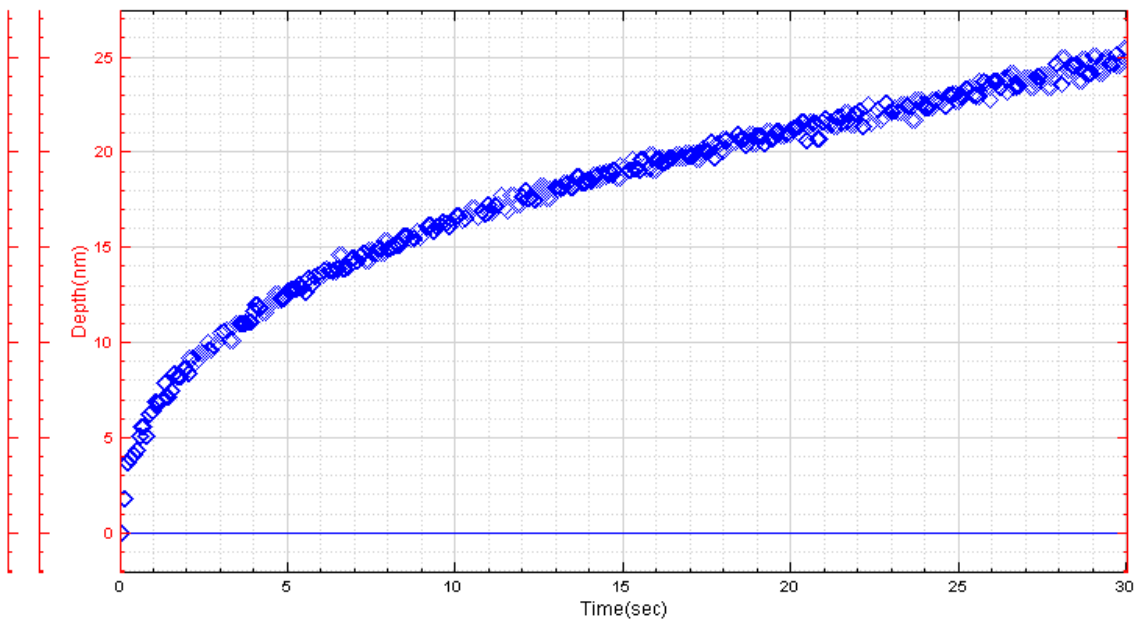
| Sample | Max Depth [μm] | Creep Depth [μm] | Creep/Max Depth [$\mu\text{m}/\mu\text{m}$] % |
|---------------|---|---|---|
| Copper | 0.698 ± 0.012 | 0.023 ± 0.002 | 3.3 ± 0.2 |
| Polymer | 6.28 ± 0.12 | 0.762 ± 0.007 | 12.1 ± 0.3 |
| Steel | 0.285 ± 0.004 | 0.007 ± 0.002 | 2.6 ± 0.6 |

Copper

| | Hv [Vickers] | H [GPa] | E [GPa] | Max Depth [μm] | Creep Depth [μm] | Creep/ Max Depth [$\mu\text{m}/\mu\text{m}$] |
|-----------------------|-------------------|--------------|---------------|-----------------------------------|-------------------------------------|--|
| 1 | 189.77 | 2.01 | 165.96 | 0.688 | 0.021 | 0.031 |
| 2 | 177.01 | 1.87 | 168.96 | 0.711 | 0.025 | 0.035 |
| 3 | 185.63 | 1.96 | 174.18 | 0.694 | 0.023 | 0.033 |
| Average | 184.14 | 1.95 | 169.70 | 0.698 | 0.023 | 0.033 |
| Standard deviation | 6.51 | 0.07 | 4.16 | 0.012 | 0.002 | 0.002 |



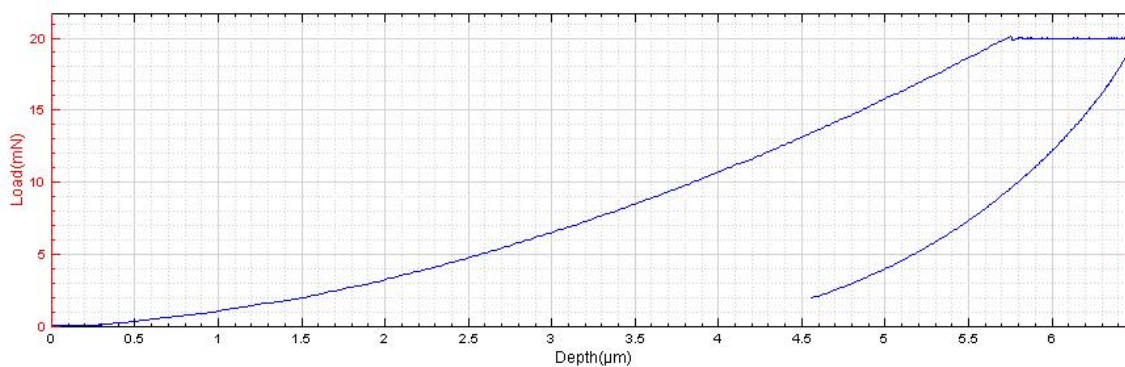
Loading Curve – Copper, Indent 2



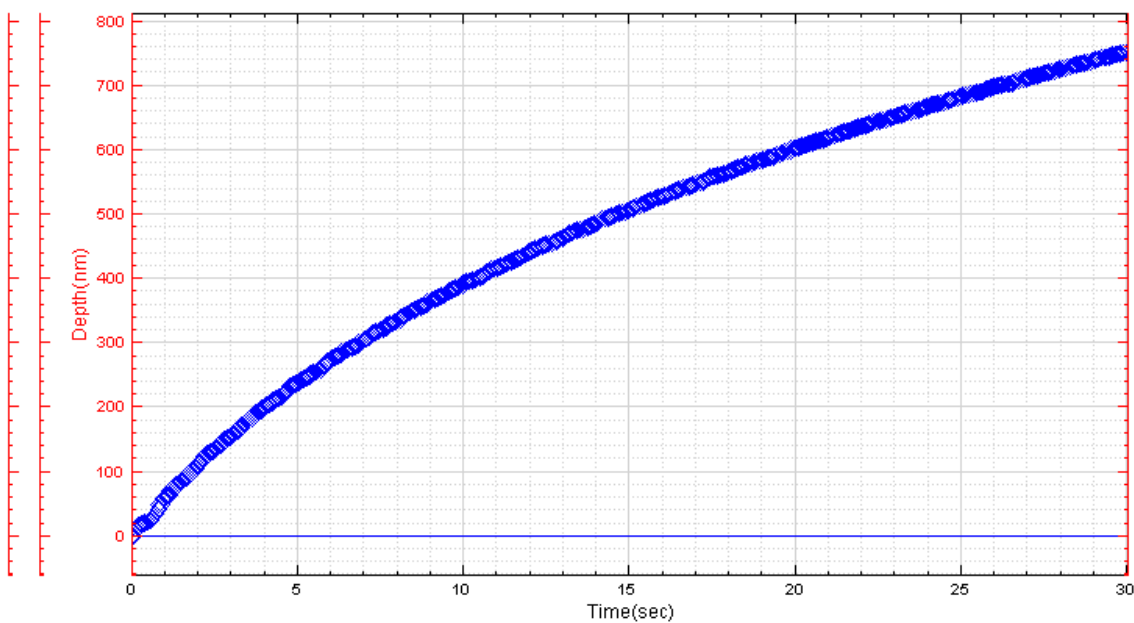
Creep Curve – Copper, Indent 2

Polymer

| | Hv [Vickers] | H [GPa] | E [GPa] | Max Depth [μm] | Creep Depth [μm] | Creep/ Max Depth [$\mu\text{m}/\mu\text{m}$] |
|-----------------------|-------------------|--------------|--------------|-----------------------------------|-------------------------------------|--|
| 1 | 3.14 | 0.033 | 0.367 | 6.42 | 0.761 | 0.119 |
| 2 | 3.21 | 0.034 | 0.368 | 6.20 | 0.770 | 0.124 |
| 3 | 3.18 | 0.034 | 0.370 | 6.21 | 0.756 | 0.122 |
| Average | 3.17 | 0.034 | 0.369 | 6.28 | 0.762 | 0.121 |
| Standard deviation | 0.03 | 0.000 | 0.002 | 0.12 | 0.007 | 0.003 |



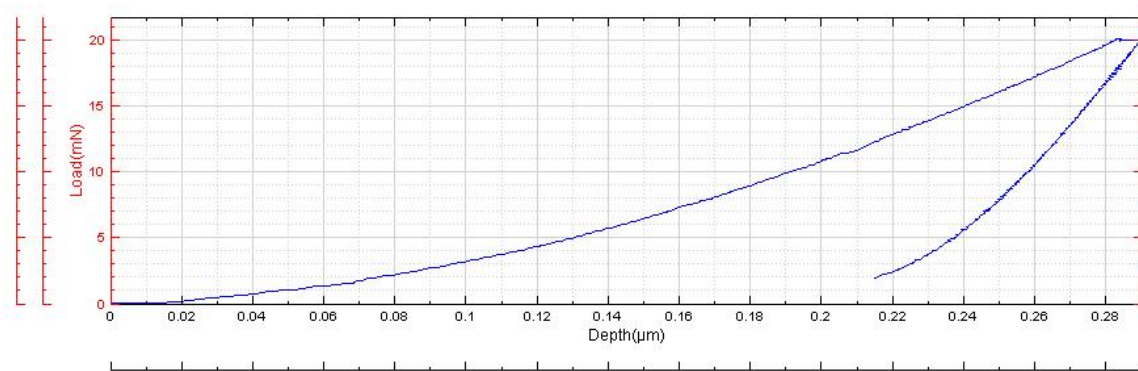
Loading Curve – Polymer, Indent 1



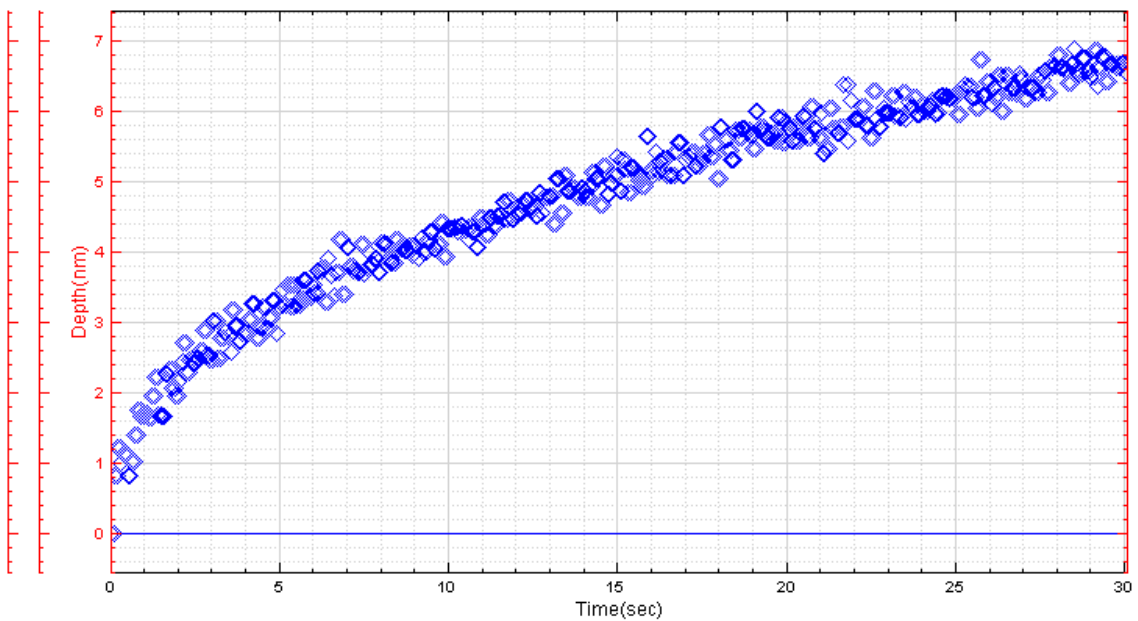
Creep Curve – Polymer, Indent 1

Steel

| | Hv [Vickers] | H [GPa] | E [GPa] | Max Depth [μm] | Creep Depth [μm] | Creep/ Max Depth [$\mu\text{m}/\mu\text{m}$] |
|-----------------------|-------------------|--------------|---------------|-----------------------------------|-------------------------------------|--|
| 1 | 1281.51 | 13.56 | 296.89 | 0.280 | 0.009 | 0.032 |
| 2 | 1224.70 | 12.96 | 274.18 | 0.288 | 0.007 | 0.024 |
| 3 | 1230.82 | 13.03 | 280.26 | 0.286 | 0.006 | 0.021 |
| Average | 1245.68 | 13.18 | 283.78 | 0.285 | 0.007 | 0.026 |
| Standard deviation | 31.18 | 0.33 | 11.76 | 0.004 | 0.002 | 0.006 |



Loading Curve – Steel, Indent 2



Creep Curve – Steel, Indent 2

CONCLUSION

In conclusion, we have shown that the Nanovea Mechanical Tester, in Nanoindentation Mode, can be used to quickly provide reliable Creep data at nm levels. Viscoelastic Creep is important when considering long-term structural design. Given loading and temperature conditions, designers can choose materials that best suit component lifetimes. Viscoelastic materials can be modeled in order to determine their temporal dependencies. These models, which include the Maxwell model, the Voigt model are used to predict a material's response under different loading conditions. Viscoelastic behavior has elastic and viscous components modeled. Each model arranges these elements in their own way. The elastic component stores energy while the viscosity one dissipates energy. It is to be noted that the non-linear response in the displacement versus time is best modeled by a Voigt model while a linear response is best modeled by a Maxwell model. The Steel sample quickly moves (in about 4seconds) from a non-linear to a linear behavior. Copper takes about 10seconds while the polymer keeps the non-linear behavior for the 30 seconds of Creep. Using these models and the test results, we believe that it is possible to fully understand the creep behavior of materials and to obtain parameters such as information on viscosity even for solid materials. We have tested Creep here while loading at a fairly slow speed. This was to show the sensitivity of the instrument since the full creep of the steel for 30seconds was only 7nm. This allows the material to adjust to the loading. In some cases, a fast loading might be more appropriate to study the full Creep behavior. Spherical tip is a well known geometry to use for these tests but it is possible to test Creep under different indenter geometry.

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