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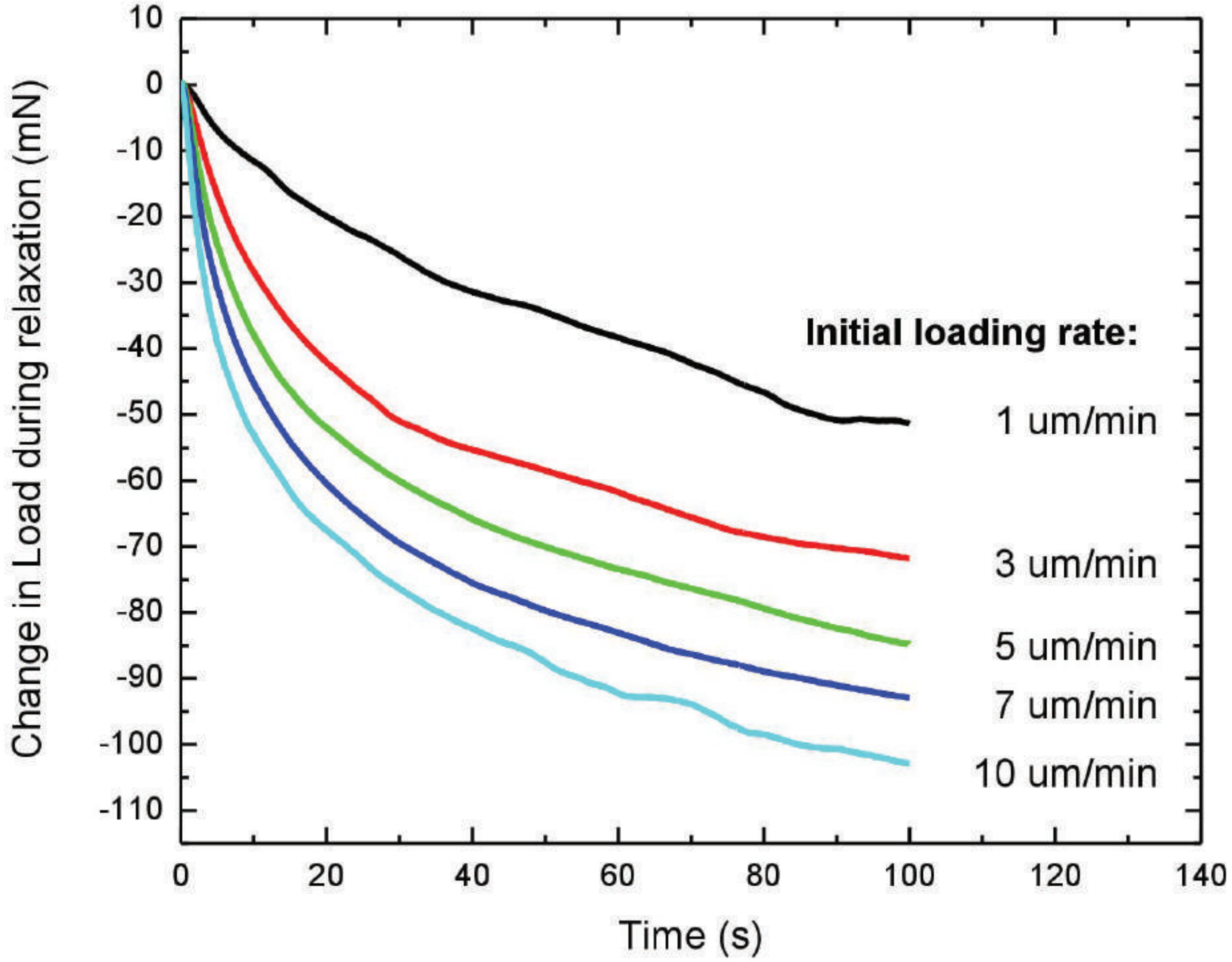
STRESS RELAXATION MEASUREMENT

BY

NANOINDENTATION



Prepared by

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Introduction

Viscoelastic materials are characterized as having both viscous and elastic material properties. These materials are subject to time-dependent stress decrease (stress 'relaxation') under constant strain, leading to a significant loss of initial contact force. Stress relaxation is dependent on the type of material, texture, temperature, initial stress, and time. Understanding stress relaxation is critical in selecting optimal materials that have the strength and flexibility (relaxation) required for specific applications.

Importance of Stress Relaxation Measurement

As per ASTM E328[i], "Standard Test Methods for Stress Relaxation for Materials and Structures", an external force is initially applied on a material or structure with an indenter until it reaches a predetermined maximum force. Once the maximum force is reached, the position of the indenter is held constant at this depth. Then the change in external force necessary to maintain the indenter's position is measured as a function of time. The difficulty in stress relaxation testing is maintaining the depth constant. The Nanovea Mechanical Tester's nanoindentation module accurately measures the stress relaxation by applying a closed (feedback) loop control of the depth with a piezo-electric actuator. The actuator reacts in real-time to keep the depth constant, while the change in load is measured and recorded by a highly sensitive load sensor. This test can be performed on virtually all types of materials without the need for stringent sample dimension requirements. Additionally, multiple tests can be performed on a single flat sample to ensure test repeatability

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester's nanoindentation module measures the stress relaxation behavior of an acrylic and copper sample. We showcase that the Nanovea Mechanical Tester is an ideal tool for evaluating the time-dependent viscoelastic behavior of polymer and metal materials.

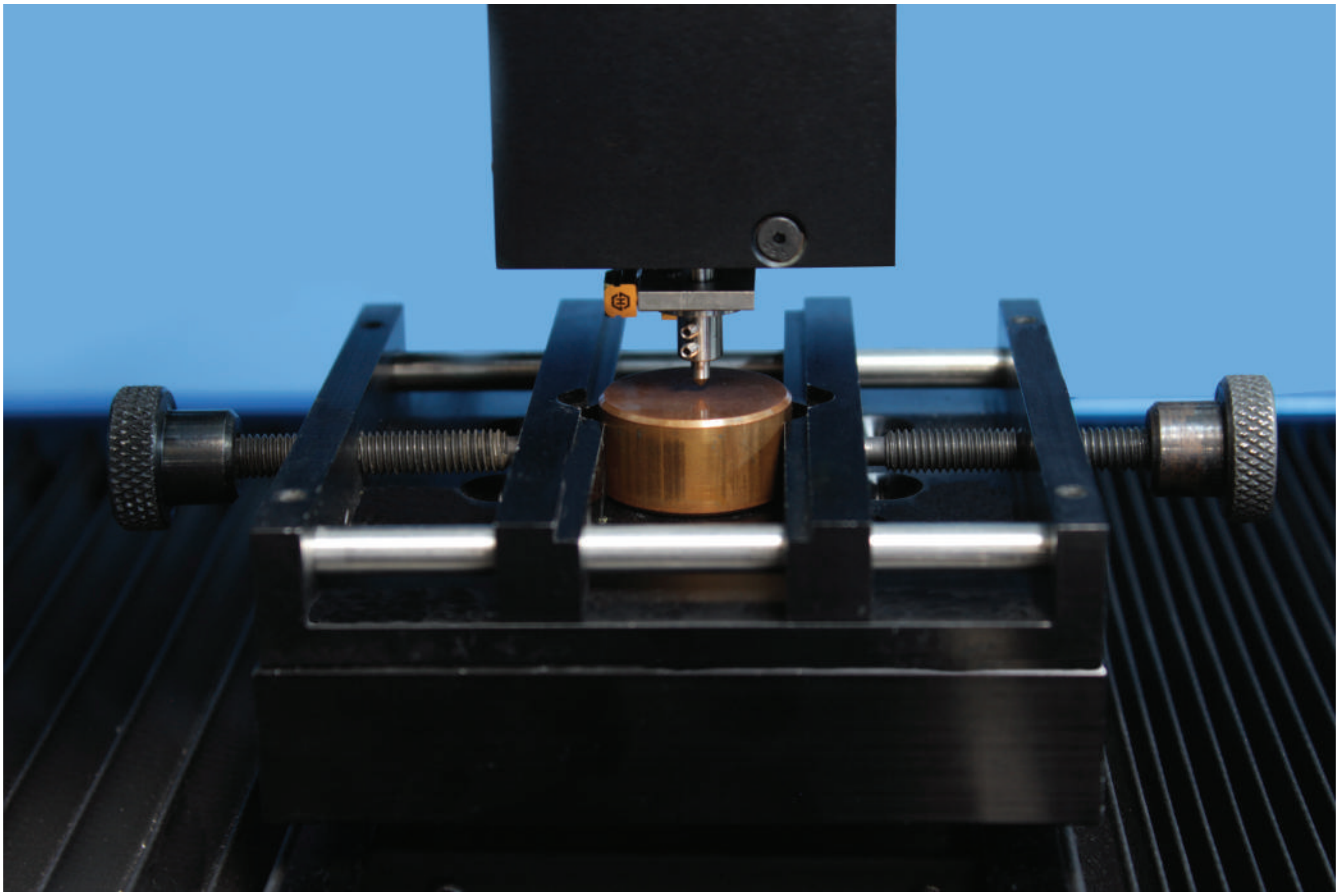

Figure 1: Indentation tip on the copper sample.

TEST CONDITIONS

The stress relaxation of an acrylic and a copper sample was measured by the Nanovea Mechanical Tester's nanoindentation module. Different indentation loading rates were applied ranging from 1 to 10 µm/min. The relaxation was measured at a fixed depth once the target maximum load was reached. A 100 second holding period was implemented at a fixed depth and the change in load was recorded as the holding time elapsed. All of the tests were conducted at ambient conditions (room temperature of 23 °C) and the indentation test parameters are summarized in **Table 1**.

Loading and unloading rate (µm/min)	1, 3, 5, 7, 10
Maximum Load	300mN for Acrylic, 80mN for Copper
Relaxation time (s)	100
Indenter type	20µm radius diamond conical tip

Table 1: Indentation test parameters for the stress relaxation measurement.

RESULTS AND DISCUSSION

Figure 2 shows the evolution of displacement and load as a function of time during the stress relaxation measurement of an acrylic sample and an indentation loading rate of 3 μm/min as an example. The entirety of this test can be broken down into three stages: Loading, Relaxation and Unloading. During the Loading stage, the depth linearly increased as the load progressively increased. The Relaxation stage was initiated once the maximum load was reached. During this stage a constant depth was maintained for 100 seconds by using the closed feedback loop depth control feature of the instrument and it was observed that the load decreased over time. The entire test concluded with an unloading stage in order to remove the indenter from the acrylic sample.

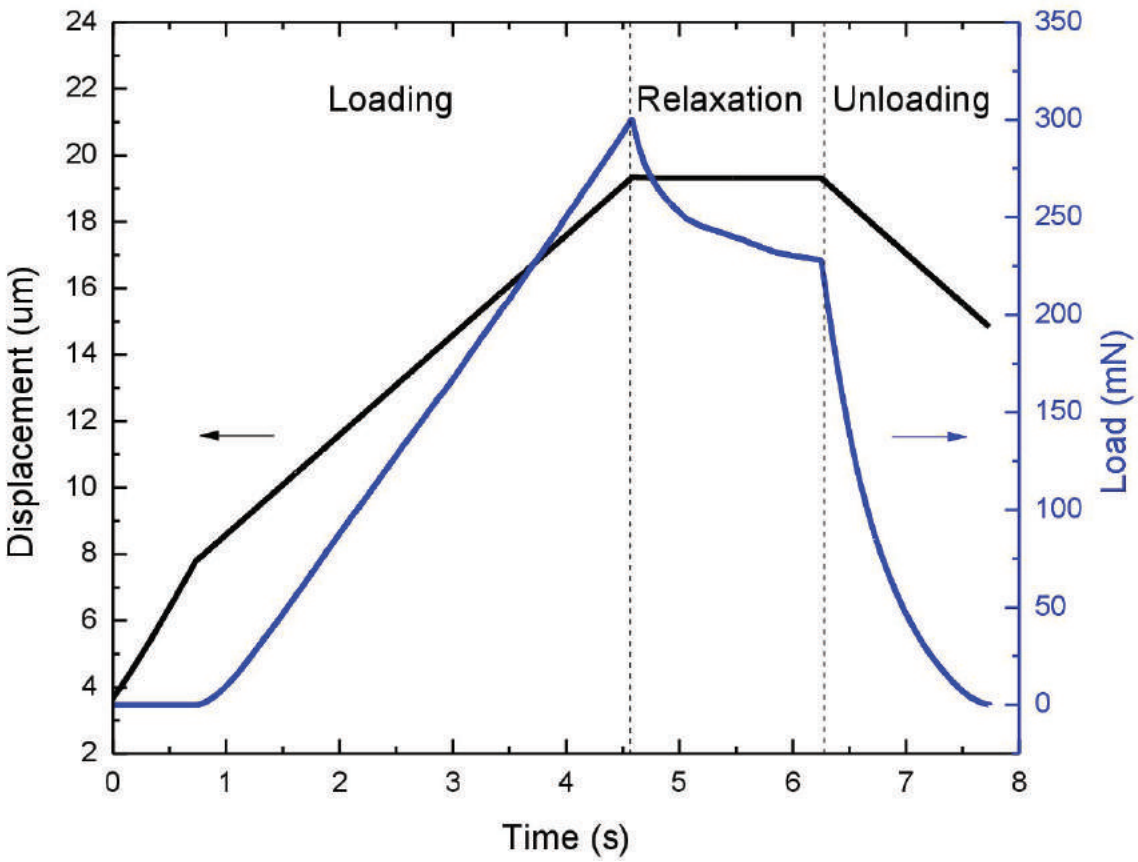

Figure 2: The evolution of displacement and load as a function of time during the stress relaxation measurement.

Additional indentation tests were conducted using the same indenter loading rates but excluding a relaxation (creep) period. Load vs. displacement plots were acquired from these tests and were combined in the graphs in Figure 3 for the acrylic and copper samples. As the indenter loading rate decreased from 10 to 1 μm/min, the load-displacement curve shifted progressively towards higher penetration depths for both Acrylic and Copper. Such a time-dependent increase in strain results from the viscoelastic creep effect of the materials. A lower loading rate allows a viscoelastic material to have more time to react to the external stress it experiences and to deform accordingly.

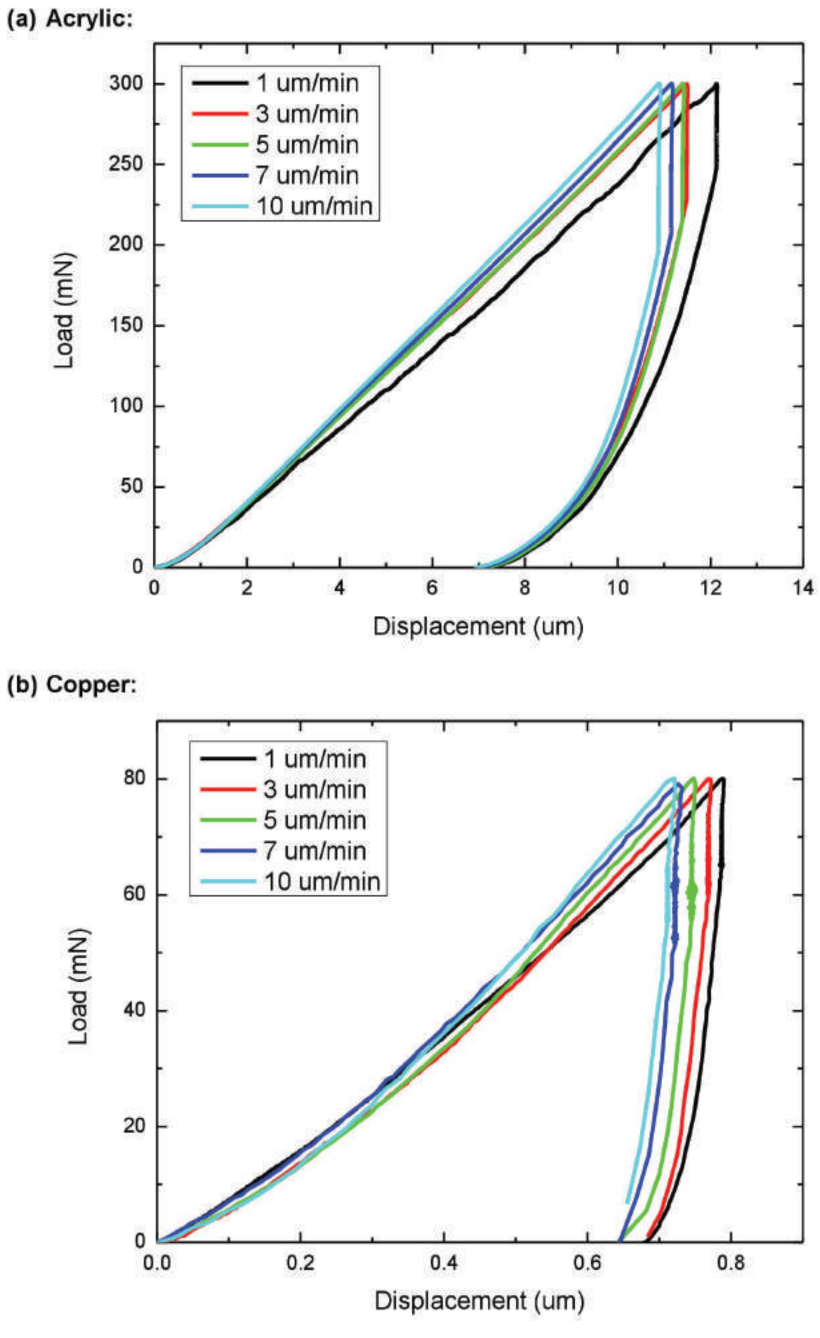

Figure 3: The load vs displacement curves of (a) the Acrylic and (b) the Copper samples.

The evolution of load at a constant strain using different indentation loading rates were plotted in Figure 4 for both materials tested. The load decreased at a higher rate in the early stages of the relaxation stage (100 second hold period) of the tests and slowed down once the hold time reached ~50 seconds. Viscoelastic materials, such as polymers and metals, exhibit greater load loss rate when they are subjected to higher indentation loading rates. The load loss rate during relaxation increased from 51.5 to 103.2 mN for Acrylic, and from 15.0 to 27.4 mN for Copper, respectively, as the indentation loading rate increased from 1 to 10 µm/min, as summarized in **Figure 5**.

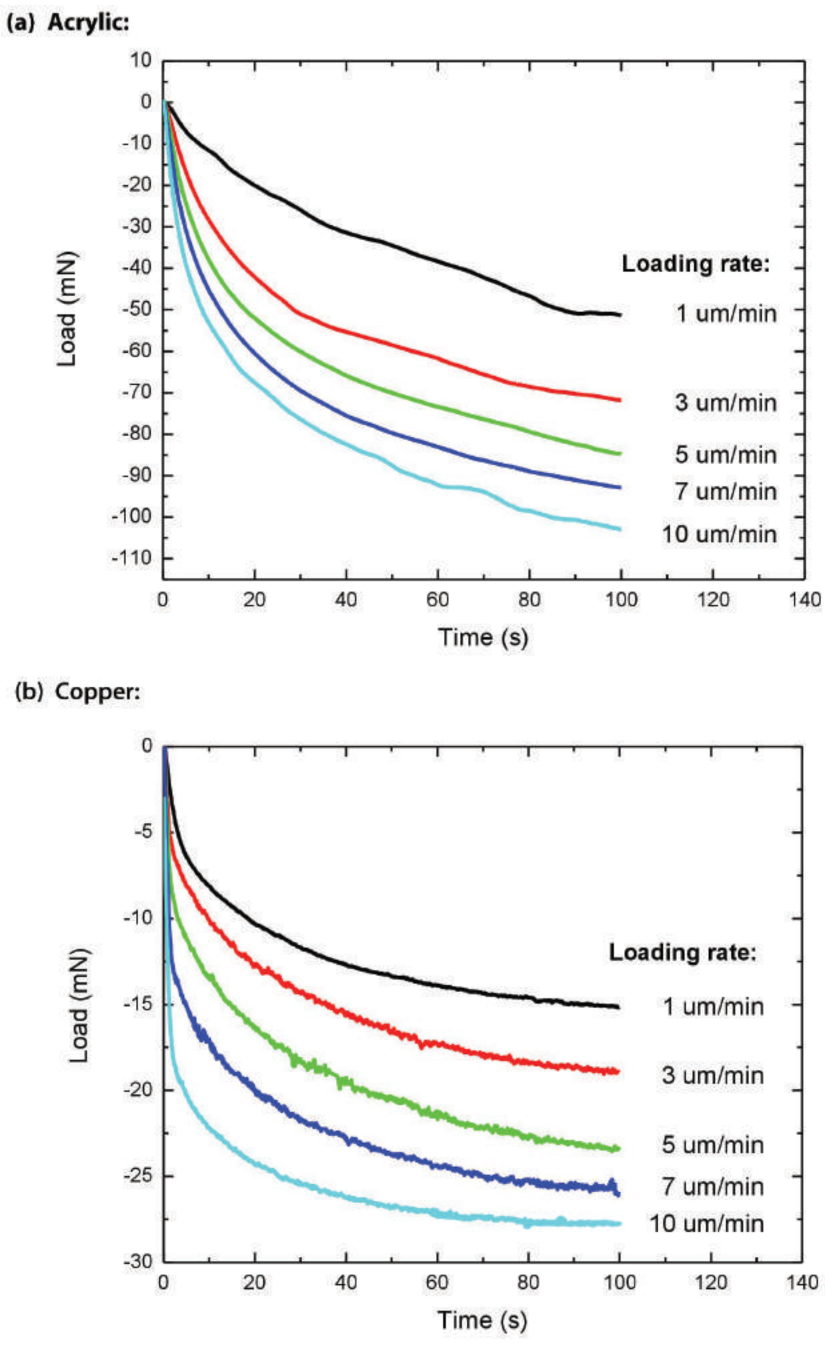

Figure 4: Evolution of the load at the maximum depth for nanoindentation tests at different loading rates.

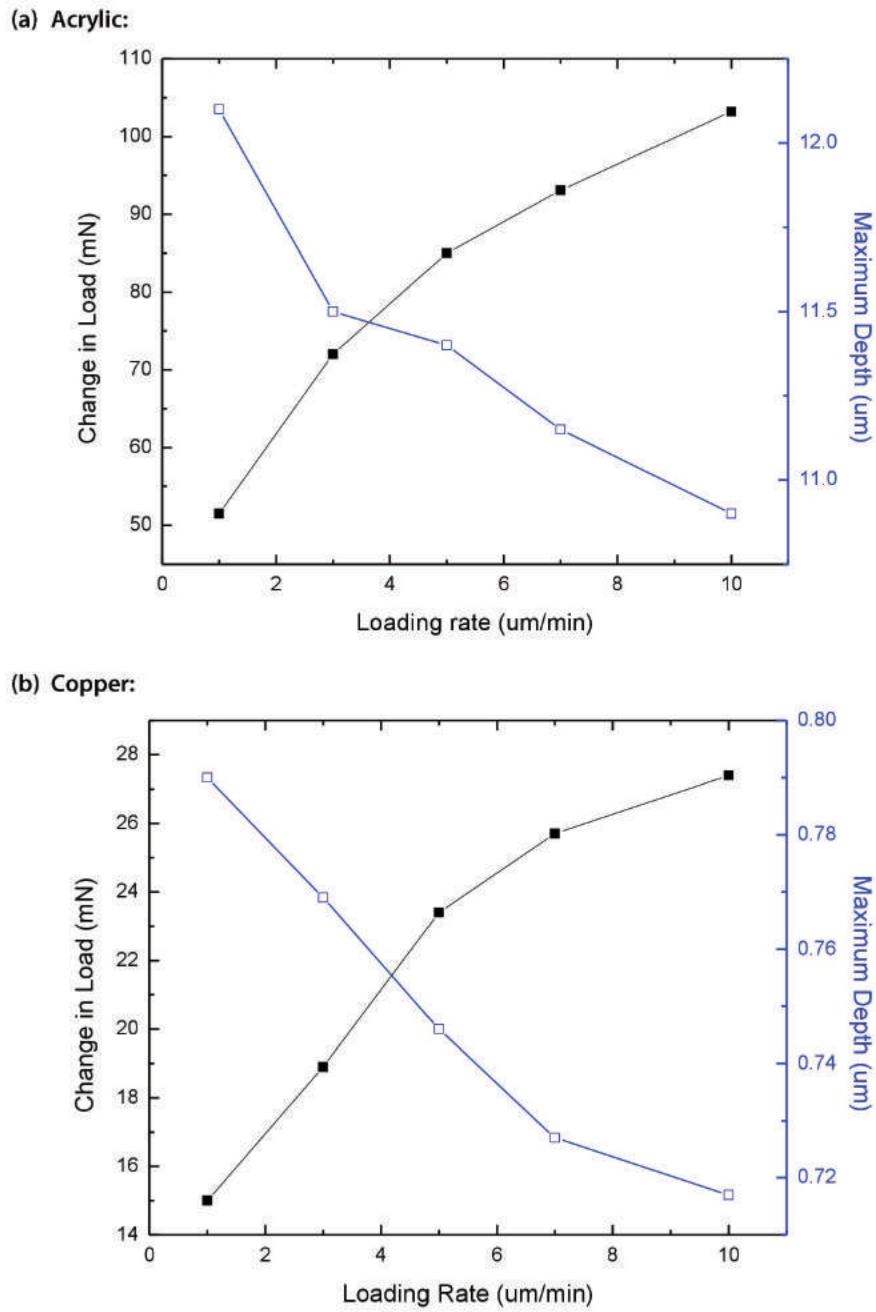

Figure 5: Change in load during the stress relaxation and Maximum Depth as a function of Loading Rates.

As mentioned In ASTM Standard E328[ii], the major problem encountered in stress relaxation tests is an instrument's inability of maintaining a constant strain/depth. The Nanovea Mechanical Tester provides excellent accurate stress relaxation measurements due to its ability to apply a closed feedback loop control of the depth between the fast acting piezo-electric actuator and the independent capacitor depth sensor. During the relaxation stage, the piezo-electric actuator adjusts the indenter to maintain its constant depth constraint in real-time while the change in load is measured and recorded by an independent high precision load sensor.

Conclusion

The stress relaxation of an acrylic and a copper sample were measured using the nanoindentation module of the Nanovea Mechanical Tester at different loading rates. A greater maximum depth is reached when indentations are performed at lower loading rates due to the creep effect of the material during loading. Both the acrylic and the copper sample exhibit stress relaxation behavior when the indenter position at a targeted maximum load is held constant. Larger changes in load loss during the relaxation stage were observed for the tests with higher indentation loading rates.

The stress relaxation test produced by the Nanovea Mechanical Tester showcase the instruments ability to quantify and reliably measure the time-dependent viscoelastic behavior of polymer and metal materials. It has an unmatched multi-function Nano and Micro modules on a single platform. Humidity and temperature control modules can be paired with these instruments for environmental testing capabilities applicable to a wide range of industries. Both the Nano and Micro modules include scratch testing, hardness testing, and wear testing modes, providing the widest and most user-friendly range of mechanical testing capabilities available on a single system.

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NANO MECHANICAL CHARACTERIZATION
OF
SPRING CONSTANTS

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