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(54) **METHOD FOR MEASURING ELASTIC PROPERTIES**

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(51) **Int. Cl.^{7}** **G01N 3/42**
(52) **U.S. Cl.** **73/81**; 73/78; 73/85; 73/76; 73/788
(58) **Field of Search** 73/78–85, 104, 73/10, 5, 760, 783, 787, 788, 826, 841, 847

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(57) **ABSTRACT**

This invention relates to a method for measuring elastic modulus of a sample object. This invention also relates to a method for correcting creep effects in the modulus measurement. The error due to creep in the apparent contact compliance is equal to the ratio of the indenter displacement rate at the end of the load hold to the unloading rate. Determination of this error term and deduction of it from the measured contact compliance can be easily done at a low cost by modifying the data analysis software in any commercial depth-sensing indentation system.
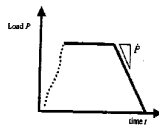
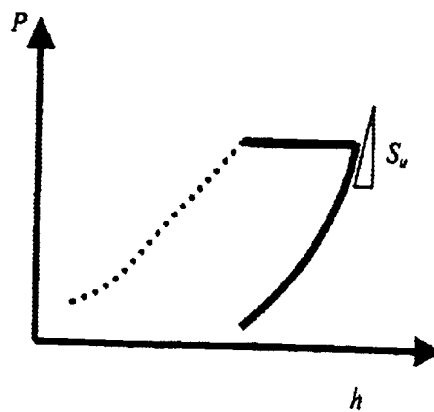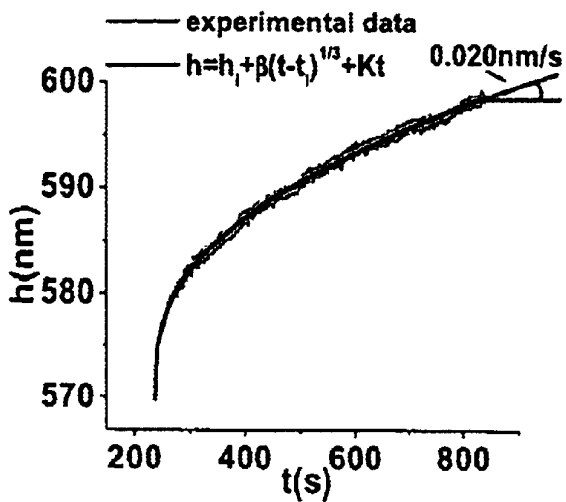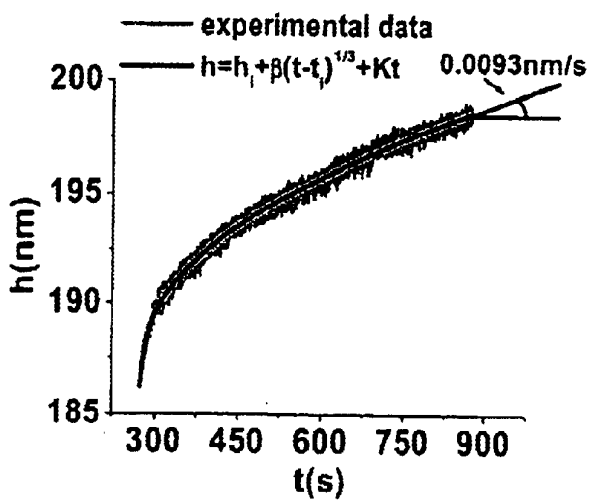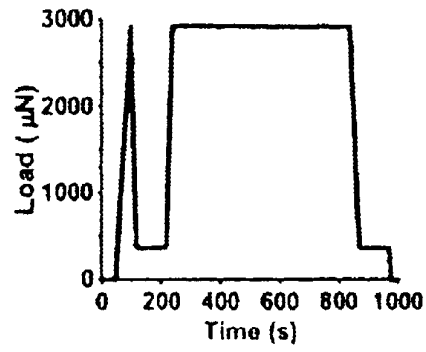**19 Claims, 5 Drawing Sheets**
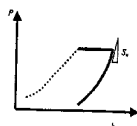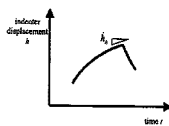
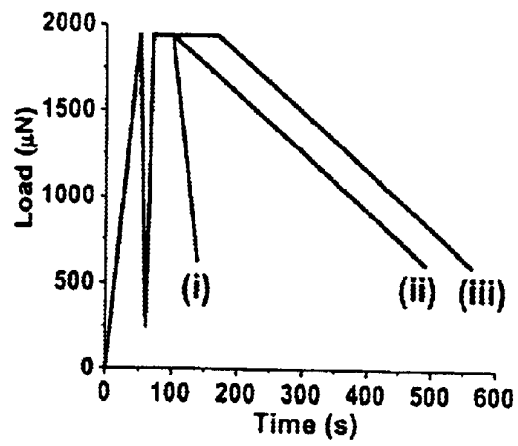Fig. 1a
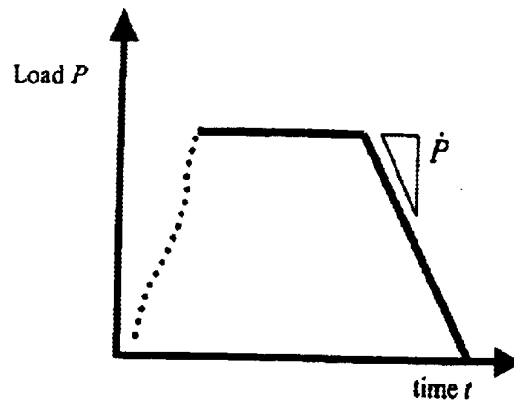
Fig. 1b
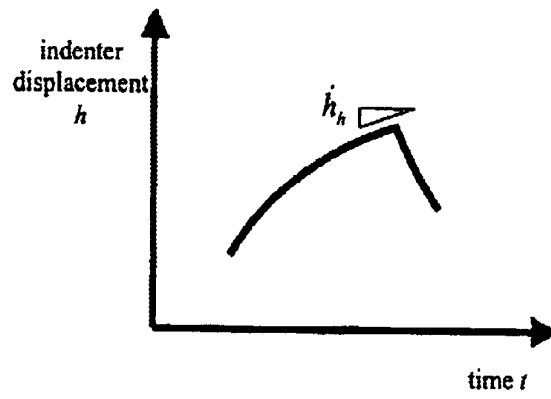
Fig. 1c

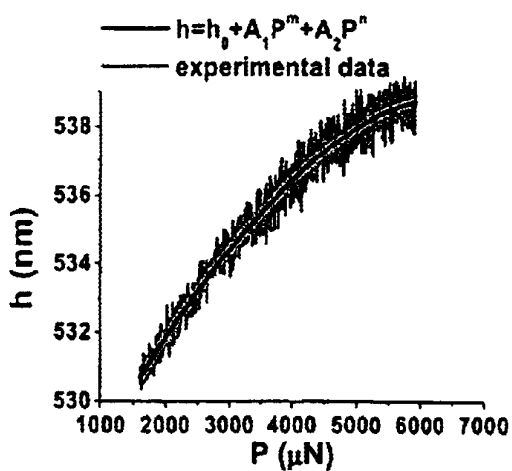**Fig. 2a**

Fig. 2b

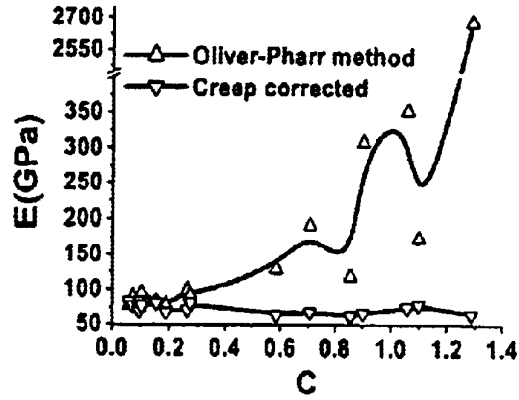Fig. 3a

Fig. 3b

Fig. 4a
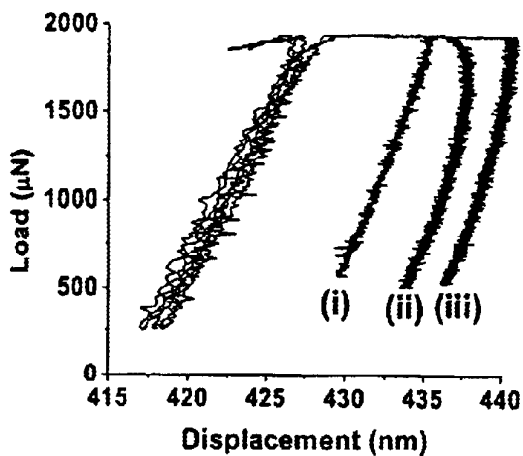
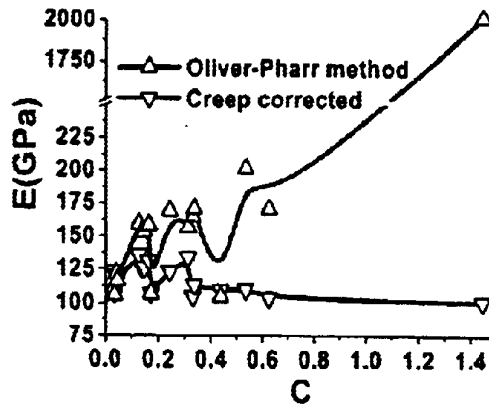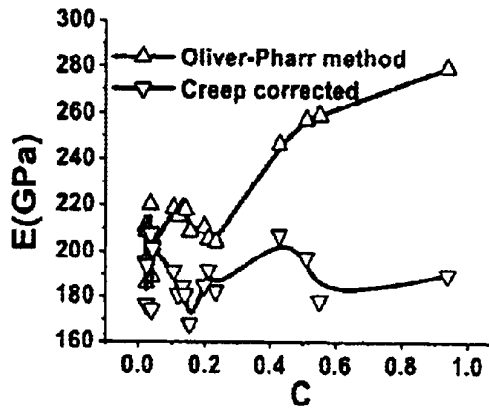Fig. 4b

Fig. 5a

Fig. 5b

Fig. 5c

METHOD FOR MEASURING ELASTIC PROPERTIES

CROSS-REFERENCE TO RELATED APPLICATION

This patent application claims priority to U.S. Provisional Patent Application No. 60/331,751 filed Nov. 20, 2001.

FIELD OF THE INVENTION

This invention relates generally to a method for measuring elastic properties of a material. More specifically, this invention relates to a method for measuring elastic modulus of a solid material by depth-sensing indentation. In addition, the present invention relates to a method for correcting for the effects of creep when measuring the elastic modulus of a solid material by depth-sensing indentation.

BACKGROUND OF THE INVENTION

Depth-sensing indentation systems have been used for measuring modulus of various materials. Most commercially available depth-sensing indentation systems are also known as "nanoindentators". When a nanoindentator is used, the indent size can be made to fall in the sub-micron range. As a result, ultra-small volumes of materials, e.g., second-phase particles in a matrix, thin-films deposited on substrates, etc., can be tested.

Unlike other conventional hardness testers, depth-sensing indentation systems use a depth-sensing machine to record the indenter displacement (or the "depth") data as the indentation proceeds. Many well-known brand names of commercial nanoindenters, such as those manufactured by MTS Systems Corporation, Hysitron Inc. and CSEM Instruments, all use the Oliver-Pharr scheme in their analysis packages. In the Oliver-Pharr scheme, the load and the depth data can be analyzed to give the contact area as well as the contact stiffness between the indenter and the sample. The contact area can be used to compute the hardness of the sample, while the contact stiffness can be used to compute the elastic modulus of the sample.

In the Oliver-Pharr scheme, the contact stiffness between the indenter tip and the sample is estimated in an unloading process from the peak load assuming that the material recovery during the unloading process is purely elastic. The reduced modulus E_r for the contact between the indenter tip and the sample is calculated from the contact stiffness S at the onset of the unloading process as follows:

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

wherein A_c can be the contact area at full load. The contact area A_c is calculated from the contact depth h_c by assuming a shape function of the indenter, i.e.,

$$A_c = f(h_c),$$

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S} \tag{2}$$

wherein ε is a constant depending on the indenter geometry (e.g., ε=0.75 for the Berkovich tip). The reduced modulus E_r is related to the sample Young's modulus as follows:

$$\frac{1}{E_r} = \frac{1 - v_s^2}{E_s} + \frac{1 - v_t^2}{E_t} \tag{3}$$

wherein E_s=Young's modulus of the sample object, v_s=Poisson's ratio of the sample object, E_t=Young's modulus of the indenter tip, v_t=Poisson's ratio of the indenter tip. In the Oliver-Pharr scheme, the contact stiffness to be used in the above equations (1) and (2) is the observed (or apparent) contact stiffness S, at the onset of the unloading process.

In reality, however, significant creep can occur at the peak load even for metals at room temperature after long holding periods. As shown in FIGS. 2(a) and 2(b), the indenter displacement continues to creep in both cases even after a long holding period of ten (10) minutes. The displacement rate appears to settle to a steady value of about 0.020 nm/s in Al and about 0.009 nm/s in Ni$_3$Al. Such a significant creep effect at the peak load can strongly affect the subsequent unloading behavior, especially when the unloading rate is not high enough.

In the extreme case where a creep dominates an elastic recovery at the onset of the unloading process, the load-displacement curve can even exhibit a "nose", such as shown in FIG. 4a for an aluminum sample object. Load schedules (i) to (iii), such as those shown in FIG. 3b, represent three similar indentation experiments on the same aluminum sample. Load schedule (i) has a very rapid unloading rate. Load schedules (ii) and (iii) have the same unloading rate, which is slower than the unloading rate in load schedule (i). On the other hand, load schedules (i) and (ii) have a short load holding period before the unloading process, while load schedule (iii) has a longer load holding period. A conspicuous "nose", such as that shown in FIG. 4a, appears in the unloading curve for load schedule (ii). The "nose" disappears when the unloading rate is increased, such as shown in the unloading curve for load schedule (i), or when the load holding period before unloading is lengthened, such as shown in the unloading curve for load schedule (iii).

When a "nose" occurs, the elastic modulus cannot be accurately calculated using the Oliver-Pharr scheme, because the apparent contact stiffness S_u becomes negative. Moreover, even if a "nose" does not occur, the presence of creep can cause serious errors in the estimation of the elastic modulus, if the creep effect is not corrected. Moreover, creep effects can become significant in certain circumstances, such as where an indentation is performed at a temperature of a significant fraction (e.g., more than 40%) of the absolute melting temperature or where a nano-sized indentation is performed by exerting a very large pressure on the sample.

The present invention provides a method for measuring elastic properties, such as the elastic modulus of an object, that avoids the above problems. Moreover, the present invention provides a method for measuring the elastic modulus of a solid material by depth-sensing indentation where the creep effect is corrected. Furthermore, the present invention provides a method for correcting a creep effect occurred when measuring elastic properties of a solid material by depth-sensing indentation.

SUMMARY OF THE INVENTION

This invention provides a method for measuring elastic properties of an object, such as by depth-sensing indentation. The present invention also provides a method for correcting for the effects of creep occurred in measuring the

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elastic modulus of various materials by depth-sensing inden-
tation. The method can comprise defining a correction term
in the contact compliance between an indenter tip and an
object to be measured. In one embodiment, the correction
term can be defined by equation (4) below. The method can
also comprise quantifying the correction term by measuring
the related quantities from the indentation data. In one
embodiment, curve fitting can be employed in quantifying
the correction term. The creep correction term can be
removed from the apparent contact compliance in the cal-
culation of the sample modulus. Such a correction procedure
can be incorporated easily into the analysis software of any
existing depth-sensing indentation system based on the
Oliver-Pharr scheme.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features, aspects, and advantages of the
invention will become much more apparent from the fol-
lowing description and accompanying drawings, in which:

FIGS. 1a to 1c show the P-t curve, the h-t curve, and the
P-h curve respectively in one embodiment of the invention
for modulus measurement using depth-sensing indentation;

FIGS. 2a and 2b are the displacement-time curves of the
load holding process after correction of thermal drift in (a)
Al, load=2918.1±1.5 μN and (b) Ni$_3$Al (111), load=
4964.6±2.1 μN, respectively;

FIGS. 3a and 3b show load schedules used in the
experiments, in which FIG. 3b shows the three different load
schedules used on a sample material of aluminum;

FIG. 4a is an enlarged view of the unloading portions of
the load-displacement curves showing creep effects thereon
after correcting thermal drift in aluminum; and FIG. 4b
illustrates the fitting by equation (5) of the unloading curve
for Cu (111) with large creep effects, wherein unloading
rate=12.47 μN/s, and creep factor C=60.3%; and

FIGS. 5a to 5c show different modulus-creep factor
curves obtained by the Oliver-Pharr scheme and the inven-
tion for sample materials of (a) Al, (b) Cu (111), and (c)
Ni$_3$Al (111), respectively.

DETAILED DESCRIPTION OF THE INVENTION

Exemplary methods embodying the principles of the
present invention are shown throughout the drawings and
described in the various embodiments below.

The invention is contemplated to measure various prop-
erties of various materials. For example, the invention can
measure the elastic properties, such as elastic modulus of a
sample material. In the method of the invention, one or more
correction term can be obtained and used to correct one or
more measurement errors occurred during the measurement
of the sample material properties. In one embodiment, a
depth-sensing indentation system can be used for measuring
the elastic modulus of a sample material. In such an
embodiment, the contact stiffness between the depth-sensing
indenter and the sample material, after correcting for the
effects of creep, can be obtained and used as a corrected
term. In another embodiment, one or more corrected terms
can be used to obtain the properties, such as the elastic
modulus, of the sample material.

According to one aspect of the invention, a method is
provided to measure elastic properties of various materials
using one or more corrected terms. Additionally or
alternatively, the method can comprise quantifying the cor-
rected terms.

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In one embodiment, a method is provided to measure the
elastic modulus of a material during the unloading process
in depth-sensing indentation. The method can comprise one
or more of the following:

(a) measuring the indenter displacement drift rate $\dot{h}_h$ just
prior to the unloading process;

(b) measuring the apparent contact stiffness S_u (the rate of
change of load with respect to indenter displacement) at
the onset of the unloading process;

(c) obtaining the contact stiffness S based on one or more
of the indenter displacement drift rate $\dot{h}_h$, the apparent
contact stiffness S_u, and $\dot{P}$, wherein $\dot{P}$ is the imposed
unloading rate at the onset of the unload process;

(d) using the value of S obtained from step (c) to calculate
the contact area A_c based on equation (2); and

(e) using the value of S obtained from step (c) above and
the value of A, from step (d) above to calculate the
reduced modulus E_r based on equation (1).

According to another aspect of the invention, a method is
provided to correct various measurement errors, such as
occurred when measuring various properties of a sample
material. In an embodiment where a depth-sensing indenta-
tion system is used for measuring the elastic properties of a
sample material, the method of the invention is capable of
correcting various measurement errors, such as caused by
creep and/or thermal drift, as will be described in details
below.

In one embodiment, a method is provided to correct
various measurement errors during an unloading process in
depth-sensing indentation. The method can comprise one or
more of the following:

(a) measuring the indenter displacement drift rate $\dot{h}_h$ just
prior to the unloading process;

(b) measuring the apparent contact stiffness S_u (the rate of
change of load with respect to indenter displacement) at
the onset of the unloading process; and

(c) obtaining a correction term based on one or more of $\dot{h}_h$,
S_u, and $\dot{P}$, wherein $\dot{P}$ is the imposed unloading rate at
the onset of the unload process.

In an embodiment where the measurement is carried out
by using a depth-sensing indentation system, the correction
term can concern the contact stiffness and/or the contact area
as will be described in details below.

The contact stiffness S can be obtained in various man-
ners. For example, the contact stiffness S can be obtained by
taking one or more of the following factors into consider-
ation: the observed (or apparent) contact stiffness S_u, the
indenter displacement rate $\dot{h}_h$, and the unloading rate $\dot{P}$. In an
exemplary embodiment, the contact stiffness S to be used in
equation (1) can be given by the following equation:

$$\frac{1}{S} = \frac{1}{S_u} + \frac{\dot{h}_h}{|\dot{P}|} \tag{4}$$

wherein the correction term is capable of correcting creep
and/or thermal drift. In an exemplary embodiment such as
shown in FIG. 1, $\dot{h}_h$ can be the indenter displacement rate
recorded at the end of the load holding period preceding the
unloading process. In another exemplary embodiment, $\dot{P}$ can
be the unloading rate at the onset of the unloading process.
In a further exemplary embodiment, S_u can be the contact
stiffness at the onset of the unloading process. The observed
(or apparent) contact stiffness. So, the indenter displacement
rate $\dot{h}_h$, and/or the unloading rate $\dot{P}$ will be described in detail
below.

In one embodiment, to evaluate the apparent contact stiffness S_u at the onset of the unloading process from the unloading portion of the load-displacement P-h curve, the load-displacement P-h curve can be first fitted by the following equation:

$$h=h_o+A_1P^m+A_2P^n \qquad (5)$$

wherein h_0, A_1, A_2, m, and n can be fitting constants. For example, h_0 can be about 520.55±0.15; A_1 can be about 0.254±0.003; A_2 can be about 7.7E-23±4.8E-22; m can be about 0.5; and/or n can be about 5.90±0.71. Equation (5) can provide very good fit to the onset portion of the unloading process. In another exemplary embodiment, R^2 can be about 0.97785. In one exemplary embodiment, the curve fitting of the load-displacement P-h curve can be carried out during an initial part of the unloading process. In another embodiment, the contact stiffness at the onset of the unloading process can be defined as $S_u=dP/dh|_u$, wherein "u" denotes the onset of the unloading process. In an exemplary embodiment, S_u can be calculated, such as by differentiating equation (5).

In another embodiment, the indenter displacement rate $\dot{h}_h$ in equation (4) can be obtained by fitting the displacement-time h-t curve during the load holding period or prior to the unloading process by the following empirical law:

$$h(t)=h_f+\beta(t-t_f)^{1/3}+Kt \qquad (6)$$

where h_f, β, t_f, and K can be fitting constants. For example, h_f can be about 568.66±0.03; β, can be about 2.94±0.01, t_f can be about 237.32±0.05; and/or K can be about 0.0062±0.0001. Alternatively, h_f can be about 184.754±0.022; β can be about 1.320±0.010, t_f can be about 274.927±0.096; and/or K can be about 0.00315±0.00009. The above equation is capable of providing very good fittings to the displacement-time h-t curves. In an exemplary embodiment, R^2 can be about 0.99753. In another exemplary embodiment, R^2 can be about 0.99013. In one embodiment, the indenter displacement rate $\dot{h}_h$ can be obtained as follows: $\dot{h}_h=dh/dt|_h$, wherein "h" can denote the end of the load holding period. In a further embodiment, the indenter displacement rate $\dot{h}_h$ can be obtained by differentiating equation (6). It will be appreciated that various alternate embodiments of calculating the contact stiffness S, the observed (or apparent) contact stiffness S_u, the indenter displacement rate $\dot{h}_h$, and/or the unloading rate $\dot{P}$ are within the scope of the invention.

Additionally or alternatively, the correction term can be based in part on the contact depth h_c. In an exemplary embodiment, the contact depth h_c can be determined from the Oliver-Pharr scheme after thermal drift correction and contact stiffness correction due to creep effects. In another exemplary embodiment, the corrected contact stiffness S obtained from the above equation (4) can be used in equation (2) to obtain a contact depth h_c. The corrected contact depth h_c can then replace the uncorrected contact depth to obtain the elastic modulus of the sample material as in the Oliver-Pharr scheme. It will be appreciated that various alternate embodiments of obtaining the contact depth h_c are within the scope of the invention.

The method of the invention will now be described through the various experiments. The invention can be used to measure elastic properties of various materials. In the embodiments such as shown in FIGS. 1 and 2, the following three materials can be used: a single crystal of Ni_3Al, a single crystal of copper, and a polycrystalline Al. In an exemplary embodiment, the Ni_3Al single crystal can have various compositions. For example, the composition of the

Ni_3Al single crystal can be of about 75 at. % Ni, about 16.7 at. % Al, about 8.0 at. % Cr, and about 0.3 at. % B. In another exemplary embodiment, the copper single crystal can have a purity of up to 99.99%. In a further exemplary embodiment, the polycrystalline Al can have a grain size of approximately 1 to 2 mm. It will be appreciated that various alternate embodiments of sample materials are within the scope of the invention.

Optionally, the sample materials can be subjected to various processes prior to the measurement. In one embodiment, the crystal material can be homogenised prior to nanoindentation. For example, the crystal can be homogenised at about 1250° C. for about 120 hours. In another embodiment, the copper single crystal can be annealed for about 5 hours at about 800° C. In a further embodiment, the polycrystalline Al can be in the as-cast state. It will be appreciated that various alternate embodiments of processing the sample materials are within the scope of the invention.

In one embodiment, the indentation surfaces of the various sample objects can be prepared by various methods. For example, the crystal materials can be prepared by having their (111) surface for indentation cut by various means. In an exemplary embodiment, the Ni_3Al or the Cu crystal can have its (111) surface for indentation cut such as by a spark machine. Additionally or alternatively, the (111) surface for indentation can be subjected to various processes, such as grinding and electropolishing. In one embodiment, the Al surfaces can be electropolished. It will be appreciated that various alternate embodiments of processing the indentation surfaces of the sample materials are within the scope of the invention.

The indentation process can be carried out using various conventional indentation equipment. For example, the indentation process can be carried out with nanoindenters or atomic force microscopy (AFM) setups with a Berkovich tip, such as those manufactured by Hysitron Inc. or TM Microscopes. In one exemplary embodiment, the indentation process can be carried out at room temperature.

Various load schedules can be employed in the indentation process. For example, the load schedules can be similar to that as shown in FIG. 3a or FIG. 3b. In one embodiment, the load can be ramped up quickly to the peak value. In an exemplary embodiment, the load can reach its peak value within a short period of time, such as within about 50 seconds. The peak value of the load can vary depending on the sample material. For example, the peak value of the load can be from about 3000 μN to about 5000 μN. In one exemplary embodiment, the peak load value for Al can be 2918.1±1.5 μN. In another exemplary embodiment, the peak value for Ni_3Al (111) can be 4964.6±2.1 μN.

In another embodiment, the load can be held at its peak value for a predetermined period of time. In an exemplary embodiment, the load holding period can be up to about 100 seconds. In another exemplary embodiment, the load holding period can be more than 50 seconds. In a preferred embodiment, the load can be ramped up quickly to the peak value followed by a rapid unloading process. Such a load schedule is capable of initiating the indent shape so that time-independent plasticity can be made negligible in subsequent stages.

In a further embodiment, the load can be ramped up again to the peak value after the initial unloading process. In an exemplary embodiment, the load can be held for a load holding period after being ramped up again to the peak value. The contact stiffness S for modulus calculation can be measured from the final unloading process. It will be appre-

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ciated that various alternate embodiments of load schedules of the indentation process are within the scope of the invention.

Various methods can be adopted to correct the measurement error caused by thermal drift. In one embodiment, low-load holds can be applied to the sample material during the indentation process. In an exemplary embodiment, low-load holds, such as about 15% of the peak load, can be placed towards the end of the final unloading process. In anther exemplary embodiment, low-load holds can be placed between the cycles to measure the thermal drift rate. Such low-load holds can be used to assess the fluctuation of the thermal drift rate during the entire measurement. For example, when the thermal drift rate changes beyond a predetermined value during the measurement, the results gathered under such circumstances can be discarded.

Creep effects can have significant impact on the modulus measured. For example, the following creep factor C can be used to represent the importance of creep over elasticity:

$$C = \frac{\dot{h}_h^c S}{|\dot{P}|} \tag{7}$$

wherein $\dot{h}_h^c$ is the creep portion of $\dot{h}_h$. In an exemplary embodiment, $\dot{h}_h^c$ can be obtained by subtracting the thermal drift rate from $\dot{h}_h$. According to equation (7), a large creep factor C can result from a slow unloading rate and/or a short load holding period, which can cause a high creep displacement rate at the end of the load holding period.

FIG. 4b shows a "nose" occurrence in the load-displacement P-h curve during an unloading process in Cu. The indentation conditions can be such that the creep factor defined in equation (7) can be about 60% so that the creep effect is significant. According to one embodiment of the invention, equation (5) can provide very good fit to the onset portion of the unloading process. In an exemplary embodiment, the value of S_u fitted by equation (5) can be about 3454 μN/nm. In another exemplary embodiment, $R^2 = 0.97785$.

FIGS. 5a to 5c show modulus-creep factor E-C curves containing modulus data measured from three sample materials. The creep factors C in FIGS. 6a to 5c can be defined by equation (7). All modulus results shown in FIGS. 5a to 5c, whether corrected for creep or not, have been corrected for thermal drift and machine compliance. As shown in FIGS. 5a to 5c, modulus values obtained without correcting for the effects of creep increase when the creep factor C increases. For example, the apparent modulus for Al and Cu is dramatically high, such as over 2000 GPa, when the creep factor C is higher than about 1.4.

FIGS. 5a to 5c also show the modulus values obtained based on equations (1) and (2) wherein the contact stiffness S is corrected by equation (4). After the creep effect is corrected by equation (4), the calculated modulus values tend towards a constant value. According to one embodiment, the corrected modulus is about 72.3±7.5 GPa for Al, about 116.9±11.1 GPa for Cu, and about 189.9±11.9 GPa for Ni₃Al. The theoretical values of the reduced modulus calculated using the method of Vlassak and Nix are 74.8 GPa for polycrystalline Al, 125.9 GPa for Cu (111), and 201.9 GPa for Ni₃Al (111). Such theoretical values of the reduced modulus all fall within the corresponding modulus value ranges measured by the method of the invention.

It will be appreciated that the various features described herein may be used singly or in any combination thereof. Therefore, the present invention is not limited to only the

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embodiments specifically described herein. While the foregoing description and drawings represent a preferred embodiment of the present invention, it will be understood that various additions, modifications, and substitutions may be made therein without departing from the spirit and scope of the present invention. In particular, it will be clear to those skilled in the art that the present invention may be embodied in other specific forms, structures, arrangements, proportions, and with other elements, materials, and components, without departing from the spirit or essential characteristics thereof. One skilled in the art will appreciate that the invention may be used with many modifications of structure, arrangement, proportions, materials, and components and otherwise, used in the practice of the invention, which are particularly adapted to specific environments and operative requirements without departing from the principles of the present invention. The presently disclosed embodiment is therefore to be considered in all respects as illustrative and not restrictive, the scope of the invention being not limited to the foregoing description.

What is claimed is:

1. A method for measuring the contact stiffness during an unloading process in depth-sensing indentation, comprising:

measuring the indenter displacement drift rate $\dot{h}_h$ just prior to the unload process;

measuring the apparent contact stiffness S_u at the onset of the unloading process; and

obtaining the contact stiffness S based on one or more of the indenter displacement drift rate $\dot{h}_h$, the apparent contact stiffness S_u, and P;

wherein P is the imposed unloading rate at the onset of the unload process.

2. The method of claim 1, wherein the contact stiffness S is obtained based on the following equation:

$$\frac{1}{S} = \frac{1}{S_u} + \frac{\dot{h}_h}{|\dot{P}|}.$$

3. The method for obtaining a correction term for correcting for the effects of creep, comprising determining a correction term based in part on one or more of the indenter displacement drift rate $\dot{h}_h$, the apparent contact stiffness S_u, and the imposed unloading rate P wherein:

the indenter displacement drift rate $\dot{h}_h$, is measured prior to the unloading process;

the apparent contact stiffness S_u is measured at the onset of the unloading process; and

the imposed unloading rate P is measured at the onset of the unloading process.

4. The method of claim 3, wherein the correction term comprises a first correction term, which is based in part on a contact stiffness S.

5. The method of claim 4, wherein the contact stiffness S is obtained by the following equation:

$$\frac{1}{S} = \frac{1}{S_u} + \frac{\dot{h}_h}{|\dot{P}|}.$$

6. The method of claim 3, wherein the correction term is based in part on a contact depth h_c.

7. The method of claim 4, wherein the correction term further comprises a second correction term based in part on one or more of the following: the maximum indenter displacement, the indenter geometry, the maximum load

imposed, the apparent contact stiffness measured at the onset of the unloading process, the indenter displacement drift rate measured prior to the unloading process, and the imposed unloading rate measured at the onset of the unloading process.

8. The method of claim 6, wherein the second correction term is based in part on the contact depth h_o.

9. The method of claim 8, wherein the contact depth h_c is obtained based on the following equation:

$$h_c = h_{max} - \varepsilon P_{max}\left(\frac{1}{S_u} + \frac{h_h}{|\dot{P}|}\right)$$

wherein h_{max} is the maximum indenter displacement, ϵ is a constant depending on the indenter geometry, P_{max} is the maximum load imposed, S_u is the apparent contact stiffness measured at the onset of the unloading process, h_h is the indenter displacement drift rate measured prior to the unloading process, and P is the imposed unloading rate measured at the onset of the unloading process.

10. The method of claim 3, wherein the indenter displacement drift rate h_h is obtained by curve fitting of the data of indenter displacement-time h-t curve prior to the unloading process by the following equation:

$$h(t)=h_i+\beta(t-t_i)^{1/3}+Kt$$

wherein h_i, β, t_i, and K are fitting constants.

11. The method of claim 3, wherein the apparent contact stiffness Su is obtained by curve fitting of the data of load-indenter displacement P-h curve during an initial part of the unloading process by the following equation:

$$h=h_o \pm A_1 P^m + A_2 P^n$$

wherein h_o, A_1, A_2, m, and n are fitting constants.

12. A method for correcting for the effects of creep in the measurement of a material property during an unloading process in the depth-sensing indentation, comprising:

measuring the indenter displacement drift rate h_h prior to the unloading process;

measuring the apparent contact stiffness S_u at the onset of the unloading process;

measuring the imposed unloading rate P at the onset of the unload process;

obtaining a correction term based on one or more of the indenter displacement drift rate h_h, the apparent contact stiffness S_u, and P; and

determining a corrected material property using the correction term.

13. The method of claim 12, wherein the correction term comprises a contact stiffness S.

14. The method of claim 13, wherein the contact stiffness is calculated according to the following equation:

$$\frac{1}{S} = \frac{1}{S_u} + \frac{h_h}{|\dot{P}|}.$$

15. The method of claim 13, further comprising obtaining a second correction term.

16. The method of claim 15, wherein the second correction term is based in part on one or more of the following: the maximum indenter displacement, the indenter geometry, the maximum load imposed, the apparent contact stiffness measured at the onset of the unloading process, the indenter displacement drift rate measured prior to the unloading process, and the imposed unloading rate measured at the onset of the unloading process.

17. The method of claim 15, wherein the second correction term is based in part on the contact depth h_o based on the following:

$$h_c = h_{max} - \varepsilon P_{max}\left(\frac{1}{S_u} + \frac{h_h}{|\dot{P}|}\right)$$

wherein h_{max} is the maximum indenter displacement, P_{max} is the maximum load imposed, ϵ is a constant depending on the indenter geometry, S_u is the apparent contact stiffness measured at the onset of the unloading process, h_h is the indenter displacement drift rats measured prior to the unloading process, and P is the imposed unloading rate measured at the onset of the unloading process.

18. The method of claim 14, wherein the indenter displacement drift rate h_h is obtained by curve fitting of the data of indenter displacement-time h-t curve prior to the unloading process by the following equation:

$$h(t)=h_i+\beta(t-t_i)^{1/3}+Kt$$

wherein h_i, β, t_i, and K are fitting constants.

19. The method of claim 14, wherein the apparent contact stiffness S_u is obtained by curve fitting of the data of load-indenter displacement P-h curve during an initial part of the unloading process by the following equation:

$$h=h_o \pm A_1 P^m + A_2 P^n$$

wherein h_o, A_1, A_2, m, and n are fitting constants.

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