

Keysight Technologies

Rapid Calibration of Area Function and Frame Stiffness with Express Test¹

Application Note

Introduction

The Keysight Technologies, Inc. G200 NanoIndenter performs a specific kind of mechanical test known as instrumented indentation. Instrumented indentation testing (IIT) involves pressing a hard indenter of known shape and elastic properties into a test material while continuously measuring both force and displacement. Mechanical properties, including Young's modulus and hardness, are derived from these fundamental measurements. Because IIT naturally lends itself to evaluating materials on the scale of nanometers, it is also commonly known as "nanoindentation".

A number of calibration processes are required in order to bring an instrumented indentation system online. For example, the mechanism for sensing the motion of the indenter must be calibrated by associating the output of the sensor with known dimensions (we use laser interferometry for this task). Most of these calibrations are performed at the factory and are transparent to the user. However, two calibrations deserve special attention. They are the "frame-stiffness" and "area-function" calibrations. These

calibrations are distinctive because they involve actually using the indenter to make indentations into a standard material. Thus, they are the last calibrations to be performed and they are often performed by the user. These calibrations are not unique to Keysight systems—they are a necessary aspect of testing with *any* instrumented indentation system.

Conceptually, one may think about the instrument *frame* as a spring in series with the contact as illustrated schematically in Figure 1. Physically, the instrument frame includes the indenter column, fixturing for the indenter tip and sample, translation mechanisms, gantry, and connections between all these parts. The objective of the *frame-stiffness calibration* is to determine a single value, K_f , which characterizes the composite elastic stiffness of the testing equipment. Once the frame stiffness, K_f , is known, then we can determine the deformation which occurs in the equipment and subtract it from the total measured displacement so as to isolate the deformation which occurs in the test sample. This is our purpose behind the calibration of frame stiffness. (Note: "Frame stiffness" is also commonly referred to as "machine stiffness" or "instrument stiffness".)

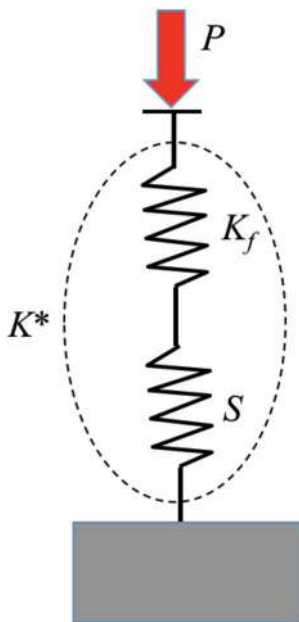


Figure 1. Schematic representation of frame (K_f) and contact (S) as two springs in series with an effective stiffness K^* .

1. A video presentation of this material is available online:
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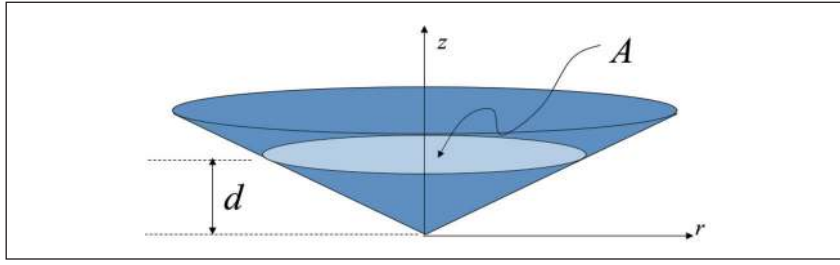


Figure 2. Schematic representation of indenter area function, $A = f(d)$.

As depicted schematically in Figure 2, the area function, $A = f(d)$, is a mathematical description of the geometry of that part of the indenter which is designed to be in contact with the test material. It expresses the relationship between the distance (d) from the apex of the indenter (along the indenter axis) and the cross-sectional area (A) of the indenter at that distance. Although we determine the area function by making indents into a standard material, it is important to note that the area function is a property of the indenter tip alone. Knowledge of the area function is required in order to be able to calculate contact area from the fundamental measurements of force and displacement. (Note: “Area function” is also commonly referred to as a “tip function,” “tip area function”, and “tip geometry”.)

Although the frame-stiffness and area-function calibrations are independent, they are typically accomplished simultaneously using a single set of experiments. In the past, it took several hours to perform the testing required for these calibrations. Most users set up the calibration batch in the evening and let the instrument run unattended through the night in order to acquire the necessary data. However, with the advent of our new Express Test option for the G200, both these calibrations can be completed simultaneously in less than 15 minutes, since Express Test performs one complete indentation cycle per second, including approach, contact detection, load, unload, and movement to the next indentation site[1, 2]. With Express Test, 400 indentations can be

performed at 400 different sites in less than 7 minutes. In addition to the obvious benefit of speed, ultra-fast testing actually *improves* calibration accuracy by allowing more indents to be included (since they are so fast) and by rendering the influence of thermal drift inconsequential².

Theory of Calibrating Frame Stiffness

With reference to Figure 1, it is clear that what is actually measured during an indentation test is the composite stiffness, K^* . The composite stiffness, K^* , is related to its components through a summation of compliances:

$$1/K^* = 1/S + 1/K_f. \quad \text{Eq. 1}$$

From elastic contact mechanics [3–5], we know that the contact stiffness, S , is related to reduced modulus (E_r) and contact area (A) through the relation

$$\frac{1}{S} = \frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}}, \quad \text{Eq. 2}$$

and that the reduced modulus is related to the Young’s modulus (E) and Poisson’s ratio (ν) of the sample (no subscript) and the indenter (subscript i) through the relation

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i}. \quad \text{Eq. 3}$$

Substituting the right-hand side of Eq. 2 in for $(1/S)$ in Eq. 1 yields

$$\frac{1}{K^*} = \frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}} + \frac{1}{K_f}. \quad \text{Eq. 4}$$

2. “Thermal drift” is the dimensional change in the equipment and sample during testing due to changing temperature. Thermal drift adversely affects the accuracy of measured displacements, with the potential for adversity being directly related to the testing time.
3. Frame compliance is the inverse of frame stiffness.

Eq. 4 expresses a linear relation between the independent variable $A^{-1/2}$ and the dependent variable $1/K^*$; the intercept of this line is $1/K_f$. In theory, one could determine frame compliance³ from a series of indentations as the intercept of a plot of $(1/K^*)$ vs. $A^{-1/2}$. However, in practice, this leads to a problem of interdependence between the frame-stiffness calibration and the area-function calibration — the area function is required in order to obtain frame stiffness by Eq. 4, but we cannot calibrate the area function without first knowing the frame stiffness (next section). To resolve this circularity, we use the fact that hardness (H) is defined as force (P) divided by contact area (A):

$$H = P/A \quad \text{Eq. 5}$$

Solving Eq. 5 for A and substituting in Eq. 4 yields

$$\frac{1}{K^*} = \frac{\sqrt{\pi H}}{2E_r} \frac{1}{\sqrt{P}} + \frac{1}{K_f}. \quad \text{Eq. 6}$$

Thus, if we indent a material having uniform hardness and reduced modulus, using a variety of applied forces, we can determine the frame compliance as the intercept of a plot of $(1/K^*)$ vs. $P^{-1/2}$. It should be noted that the hardness and reduced modulus of the standard material need not be known in order to determine frame stiffness. These properties must be merely uniform throughout the material.

The highest-force indents are the most useful for the purpose of determining frame stiffness. They have the strongest influence on the value of the intercept, and they are least subject to experimental problems (tip anomalies, surface contamination, etc.) Thus, it is common practice to only use high-force indents for the calibration of frame stiffness.

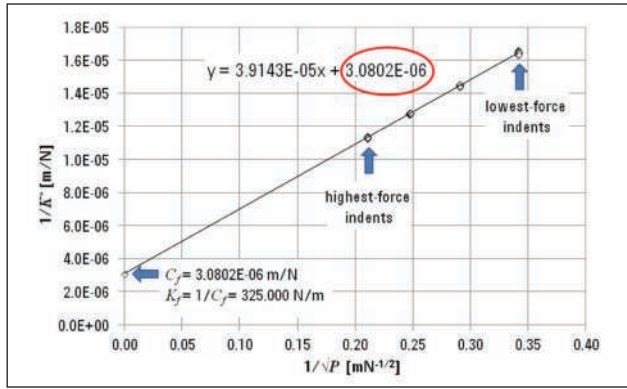


Figure 3. Frame stiffness calibration accomplished with Express Test (simultaneously with area-function calibration) in less than 15 minutes.

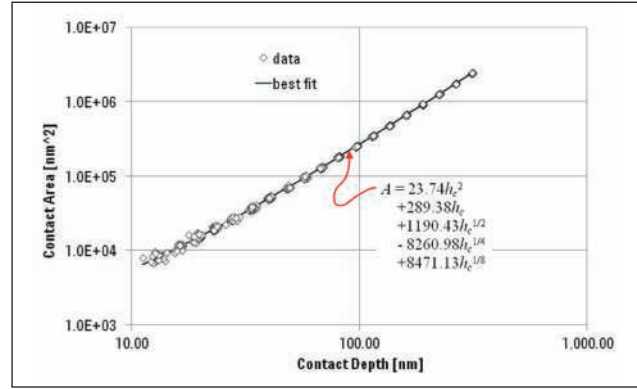


Figure 4. Area function calibration for a Berkovich indenter accomplished with Express Test in less than 15 minutes.

Theory of Calibrating Area Function

The theory presented here assumes that the frame stiffness has been accurately calibrated and that displacement (h) equals that deformation which occurs in the test material as a result of indentation. The calibration process involves curve-fitting a set of ordered pairs of contact depth (h_c) and area (A) with a functional form that is appropriate to the kind of indenter being used (Berkovich, conical, spherical, etc.). The ordered pairs (h_c , A) are determined experimentally by indenting a standard material using a variety of applied forces. We know from elastic contact mechanics [3,5] that the contact depth for each indentation can be calculated as

$$h_c = h - 0.75P/S, \quad \text{Eq. 7}$$

where the stiffness is determined as the slope of the force-displacement curve acquired just as the indenter begins to withdraw from the sample⁴:

$$S = \left. \frac{dP}{dh} \right|_{h=h_{\max}}. \quad \text{Eq. 8}$$

The contact area for each indentation is calculated by solving Eq. 2 for A :

$$A = \frac{\pi}{4} \frac{S^2}{E_r}. \quad \text{Eq. 9}$$

Because indents are performed in a standard material, the reduced modulus is known. For example, the

reduced modulus of fused silica is 69.9 GPa. Thus, everything on the right-hand side of Eq. 9 is known, and contact area A can be calculated for each indentation.

For a Berkovich indenter, the area-function is achieved by curve fitting the (h_c , A) data from the indentations performed in the standard material to the form

$$A = m_0 h_c^2 + m_1 h_c + m_2 h_c^{1/2} + m_3 h_c^{1/4} + m_4 h_c^{1/8}, \quad \text{Eq. 10}$$

where m_i are best-fit constants. The first term in Eq. 10 has the form of the ideal area function for a Berkovich indenter ($A = 24.56d^2$). The second term is the ideal form for a rounded indenter; this term accurately captures the effect of finite rounding at the apex of every real indenter. The remaining terms in Eq. 10 may or may not be necessary for a particular indenter, depending on the physical shape of the indenter. Conventional curve-fitting wisdom dictates how many constants should be used in Eq. 10. More constants may provide a smaller χ^2 , but at the sacrifice of physical relevance and applicability of the resulting fit. The fit may behave in unexpected ways between data points, or outside the range of data included in the fit. If only two terms

are used, the fit may be used reliably over any displacement range. If more terms are used, the fit (and the tip) should be used only within the displacement range for which the fit was determined.

Experimental Method

All indentations required for the determination of frame stiffness and area function are performed with a Keysight G200 NanoIndenter having Express Test, which includes NanoVision and a DCM II fitted with a Berkovich indenter. The standard material is fused silica.

The test method “Express Test Tip Calibration” automatically performs all the indentations necessary for calibrating frame stiffness and area function. Within an area of $100 \mu\text{m} \times 100 \mu\text{m}$, the method performs 20 vectors of indents, each vector comprising 20 indents to a particular force for a total of 400 indents. The first vector is performed to the maximum force of 30 mN. The purpose of this first vector is to clean the indenter. The process of making the indents pushes debris up the faces of the indenter so that it is not in the way of subsequent testing. Results from these first 20 indents are not used for

- Express Test determines contact stiffness from the slope of the unloading curve as expressed by Eq. 8. However, there are other ways to determine contact stiffness which are also valid. Our CSM option, for example, determines contact stiffness by means of an applied oscillation as the real part of the force amplitude divided by the resulting displacement amplitude. If the test material has negligible viscoelasticity, then these two methods of determining stiffness yield statistically identical results.

either calibration, because they may be compromised by debris. The next vector is performed to a maximum force of 22.5 mN, and subsequent vectors are performed to progressively smaller forces, with the last vector having a maximum force of 67 μ N. The total time required to perform these 400 indents is about 7 minutes.

Once the data are acquired, the NanoSuite software performs the theoretical analysis (Eqs. 1–10) required to obtain the frame stiffness and area function. For the calibration of frame stiffness, only those indents for which the applied force exceeds 8 mN are included in the calculation (a total of 80 indents). All but the first 20 indents (a total of 380 indents) are included in the calculation of area function.

Results and Discussion

Figure 3 shows the results of the frame-stiffness calibration. For this system, the frame stiffness is determined by inverting the y-intercept of the best linear fit which gives $K_f = 3.25E5$ N/m. This value is typical for the Express Test option, which employs the DCM II indentation head and the NanoVision positioning stage. A total of 80 indents were included in this calibration. In future testing, the displacement occurring in the instrument is calculated as P/K_f and subtracted from the total measured displacement in order to obtain the displacement of the indenter into the surface of the test sample.

Figure 4 shows the results of area-function calibration. The (h_c, A) data are plotted as distinct points, and the empirical area function (solid line) is obtained by curve fitting

these data. In future testing with this particular diamond, the contact area is calculated by evaluating this area function at the calculated contact depth: $A = A(h_c)$. The contact area so determined is used directly in the calculation of Young's modulus and hardness.

Conclusions

Two calibrations are regularly performed by the user of any nanoindentation system: the frame-stiffness and area-function calibrations. With traditional nanoindentation, hours of instrument time were required in order to perform the necessary indents for these calibrations. With the advent of Express Test – an option for the Keysight G200 NanoIndenter – these calibrations take only 15 minutes, so that the user can begin proper testing almost immediately.

References

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