

WHITE PAPER | SEMICONDUCTOR

Extracting Intrinsic Mechanical Properties of Thin Low-Dielectric Constant Materials with iTF Analysis

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I. Introduction

Continuous scaling of integrated circuits has led to the replacement of the interconnect insulator SiO_2 by porous low dielectric constant (low-k) materials to reduce the RC delay.¹ However, the inferior mechanical properties of these low-k materials has raised concerns regarding their integration and reliability.² These concerns are further aggravated with the necessity of integrating thinner and more porous materials at each advancing technology node. Consequently, there has been a growing interest in evaluating and monitoring the mechanical properties of low-k materials, specifically their elastic modulus.³ For this purpose, nanoindentation has been the mostly widely used technique. This is largely due to its simple sample preparation requirements, its straightforward measurement technique, its ability to obtain statistical information, and its use of a rich history of mathematical formulations based on contact mechanics to derive properties.

The conventional approach for determining the elastic modulus of bulk materials by nanoindentation is based on the ISO-14577-1 standard. For thin low-k film measurements however, the technique is not valid due to the influence of substrate⁴ (usually Si-wafer). This issue is known as the *substrate effect* and it constitutes a major challenge to obtain intrinsic properties by nanoindentation, as the elastic modulus obtained at a particular depth is the result of a composite response from both the thin film and the substrate (Figure 1).

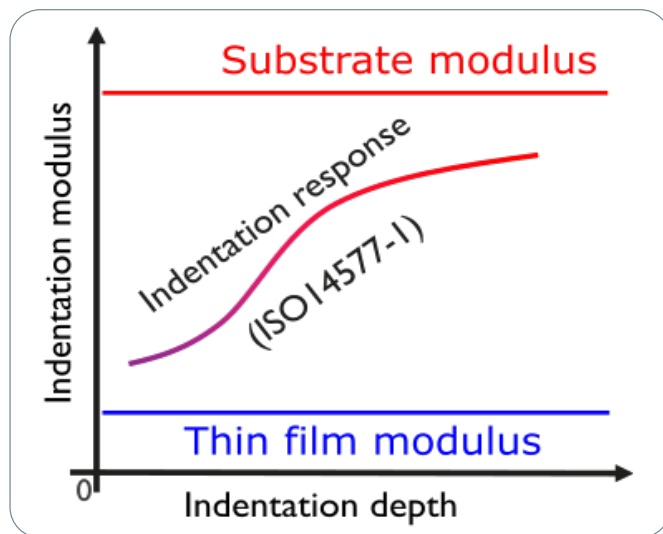


Figure 1. Schematic representation for indentation modulus calculated by the ISO 14577-1 standard, as a function of indentation depth for a compliant, thin film on a stiff substrate.

Efforts to obtain substrate-independent properties of thin films from nanoindentation measurements has been a major research focus. The vast majority of these studies are linked to the interpretations of relationships between *contact stiffness* (S) and *contact radius* (a).⁵⁻⁸ Among these, the iterative solution scheme proposed by Li and Vlassak⁸ has been shown to work accurately within the

elastoplastic regime and is applicable for a wide range of indentation depths and elastic mismatch ratios.^{8,9} The solution uses two measures of S - a curves (experimental and theoretical) and iterates the elastic modulus of the thin film until the error between the curves is minimized. This methodology is patented by Harvard University,¹⁰ with an exclusive license granted to Bruker for the technique. Through this license agreement, Bruker has developed the intrinsic thin film (iTf) analysis package. This software package provides a user-friendly environment to the complex mathematical solution for extracting the intrinsic mechanical properties of thin films in a convenient and time-efficient manner.

As the solution works through comparison of the experimental and theoretical measures of S : a , there are several critical inputs for the iTf analysis to extract an accurate modulus value; area function, iteration ranges, indentation depth range, substrate modulus. Failing to supply proper values may lead to inconsistent output conclusions. It is important that the output be independent of instrument or the probe geometry.

This paper focuses on the optimization and use of Bruker's iTf software package for the extraction of intrinsic (substrate independent) mechanical properties, particularly for thin, low-k materials. These considerations are split into two main parts: Measurement procedure (Section II) and iTf execution (Section III). The former outlines important aspects of acquiring proper experimental data, which is later fed into the iTf software. It should be noted that the reader is assumed to have a preliminary understanding of the Li-Vlassak solution and basic nanoindentation principles.

II. Measurement and Procedure

The iTf software package utilizes data from nanoindentation measurements. For best results follow the guidelines below for tip area function, indenter geometry selection, load function, and data analysis.

a. Area function calibration

The area function is a required input for iTf, the accuracy of which directly influences the resulting output. Therefore, performing regular air indentations (daily), machine compliance calibrations by a Berkovich indenter (when the tip or sample mounting scheme is changed) and obtaining a proper area function of the indenter by calibration measurements on a reference sample are the first steps towards achieving accurate results. The tip area function calibration should always be performed when a new tip is mounted and should be monitored as frequently as possible. Tip wear is less of an issue on softer test specimens, but frequent use will dull the probe, changing both the tip area function, and eventually the strain/geometry of the contact.

The low-k films typically found in semiconductors are usually thin (25–400 nm), and require shallow contact depths. Therefore, it is of paramount importance to have

a proper tip area function calibration, especially at these shallow indentation depths, where tip imperfections result in significant deviations from ideal area function (e.g., ideal $A_c = 24.5h_c^2$ for a Berkovich indenter). Additionally, the iTF solution requires an experimental S - a curve obtained through an indentation depth interval. Hence, the measurements conducted to perform area function fitting (usually on fused quartz) should cover at least the whole range of indentation depth planned to be used in actual thin film measurements. Using the same type of load function (quasistatic, partial unloading or nanoDMA) for area function calibration and the related actual thin film indentations is recommended. For quasistatic and partial unloading measurements, a fine displacement resolution is advised (preferably 1 data point per 1 nm of displacement at shallow depths, e.g., 3-30 nm). For nanoDMA measurements, the area function should be built with nanoDMA III CMX tests with a frequency of 90 Hz.

Another potential issue for the area function calibration is the negative A_c values occasionally observed in the fitting at the shallow contact depths required for indentation of these thin films (e.g., <20 nm). These negative values are an artifact from over specifying the fitting parameters, and should be avoided. If the A_c yields negative values at low depths, it is advised to reduce the number of coefficients and/or enforce only positive terms for area coefficients (C_n). More detailed information on machine compliance calibration, indenter area function calibration, and nanoDMA III application can be found in Bruker's Hysitron TribolIndenter user manuals.^{11,12}

b. Indenter tip selection

Geometries sharper than the Berkovich include the cube corner and the 60° pyramidal. Sharper tips increase local plastic strain, requiring lower forces to penetrate to a given displacement, due to the increase in plastic strain. They also exhibit a decrease in the extent of the substrate effects when using the standard nanoindentation analysis due to these lower forces. Figure 2 shows the indentation moduli extracted by the ISO 14577-1 method from indentations performed by three different indenters with varying sharpness on the same 183 nm thick low- k film. The Berkovich shows the largest substrate effect due to the steep curvature in modulus versus depth. Therefore, applying a substrate correction methodology, such as the iTF, is necessary in to obtain *intrinsic* thin film properties.

Using a sharp indenter reduces the effect of the substrate on the modulus measurements, but increases error due to radial or outward displacement that deviates from the Sneddon punch approximation. In addition, as the indenter sharpness increases (e.g., for cube corner and 60° pyramidal), the probability that there will be "cutting" or fracture increases dramatically, while the substrate effects are reduced considerably. Therefore, while sharp tips may help in obtaining substrate-independent properties directly from conventional measurements, a method such as iTF is more reliable and still preferred.

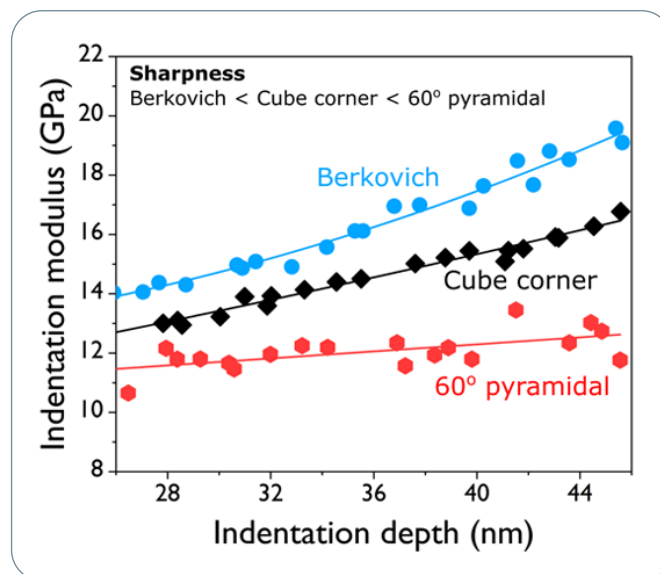


Figure 2. Indentation moduli extracted for three different indenters using conventional data analysis from measurements on a 183 nm thick low- k film.

For measuring thin-low- k film properties with the iTF method, a sharp Berkovich tip is recommended. It is acknowledged that the substrate effects will be more pronounced with a Berkovich tip as compared to sharper tips, such as cube corner or 60° pyramidal tips. Despite this, the smaller spherical penetration regime combined with the lower stresses induced with a Berkovich indenter (delaying densification and fracture events) help increase the usable indentation depth interval for iTF fitting.

c. Load function

To produce the required experimental S - a versus depth curves that will be compared to the theoretical values during the iTF analysis, data from a range of indentation depths is required. It is recommended that the measurements should be automated starting from 1 nm to the total film thickness. For quasistatic indentations, a basic trapezoid load function with loading (5 sec), holding (5 sec) and unloading (5 sec) with thermal drift monitoring prior to the indentation (40 sec) should be sufficient. One parameter to note on the load function is the pre-load or contact threshold force. This value is used for contact determination and should be kept below 0.5 μ N to avoid potential damage to the porous structure prior to the actual measurement. Alternatively, a small lateral offset (sideways motion) can be set after contact detection using the 'approach offset' functionality of the Bruker software to ensure a virgin surface area is tested.

d. Data analysis

In a typical nanoindentation test, there is usually some offset at the contact point of nanoindentation load-displacement curves (usually a few nanometers). Although this would have a negligible influence for high-load micro-indentations, it can be a significant portion of the total contact depth for thin film indentations. Therefore, it is

important to off-set the (load, displacement) values to (0,0) at the beginning point of the tests as accurately as possible before proceeding with any data analysis.

Another important aspect for data analysis is related to the application of widely used analytical solution that relates contact stiffness to the contact area for axisymmetric indenters.¹³

$$(Eq. 1) \quad S = \frac{2}{\sqrt{\pi}} E_r \sqrt{A_c}$$

Pyramidal indenters such as Berkovich and cube corner are not axially symmetric, therefore Eq. (1) was modified¹⁴ to include a geometry correction factor, β . Later, an additional correction factor, γ , was introduced¹⁵ to rectify finite radial inward displacements which arise from Sneddon's solution.¹⁶ A frequent overall correction factor used for Berkovich indenter is 1.08, but there is no consensus among the researchers and the value of the correction factor may change depending on the indenter angle. On the other hand, the theoretical solution provided by Yu for bilayer indentation¹⁷ (is used in both the Li-Vlassak solution and the derived iTF software) was published prior to either of these correction factors. This means that the iTF platform uses Eq. (1) directly without any correction factors. Therefore, it is recommended that the experimental data analysis should be performed without correction factors as well (or as $\beta = \gamma = 1$), so that the relative comparison of theoretical and experimental S - a curves is not affected by user-selection of correction factors.

A demonstration for this issue is presented in Figure 3. A 291 nm low-k film with 15% porosity is indented with Berkovich and cube corner tips using quasistatic measurements up to 90 nm. When the data analysis is performed with varying values of the correction factors, the extracted values from iTF differ and the discrepancy between indenters increase.

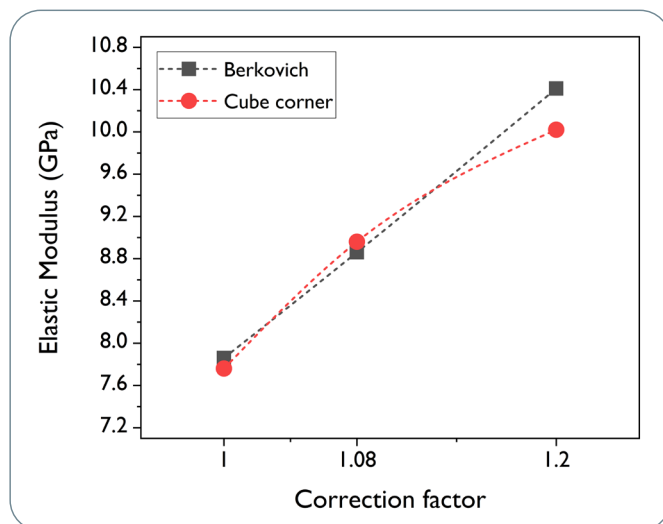


Figure 3. The impact of correction factors on the iTF results extracted for a 291 nm, 15% porous low-k film.

III. iTF Execution

There are four critical user input values required for the iTF fitting a thin film. They are the properties of the substrate, the minimum fitting depth, the maximum fitting depth, and the starting values for the plasticity parameter. A basic introduction into the importance of each parameter and how to optimize them for films is presented in each section.

a. Substrate properties

The theoretical S - a curves of the system calculated by iTF require an input for the elastic modulus of the substrate. However, low-k films are typically deposited on a silicon wafer, which has a high degree of anisotropy (directional dependence). The modulus dependence on crystal orientation is shown in detail by Hopcroft, et. al.¹⁸ Using the anisotropic stiffness coefficients of silicon, a stiffness matrix can be calculated for a variety of crystal orientations, based on the work of Zhang, et. al.¹⁹ Then, for a known crystal orientation, the indentation modulus of silicon in the direction of indentation can be approximated using the explicit anisotropic indentation formulations provided by the work of Delafargue and Ulm for conical indenters.²⁰ Following this procedure, indentation moduli of Si for commonly encountered (100), (110) and (111) orientations are estimated to be 163 GPa, 172 GPa, and 177 GPa, respectively.

Alternatively, if the crystal orientation is unknown, standard nanoindentation tests can be performed on the backside of the wafer to extract the mechanical property of the substrate. While doing so the unloading curve fitting range should be adjusted to avoid the phase transformation induced kinks/pop-outs.²¹

b. Minimum indentation depth

When setting up the analysis, the iTF package requires a user determined input for the **minimum** indentation depth to be used for the analysis (i.e., ignore displacements less than...). For thin low-k films, the main criteria for this selection are the tip imperfections, such as the tip radius for a pyramidal indenter.

The theoretical solution of iTF originates from the work of Yu,¹⁷ who progressed upon the gradually established works of Sneddon,¹⁶ Lebedev-Ufliand,²² and Chen-Engel²³ for a bilayer indentation problem. In this theoretical solution, boundary conditions for displacements along the material surface are defined based on the indenter shape. Therefore, the iTF software package requires the input of tip geometry in the form of pyramidal or spherical shape. Although the conventionally used Berkovich and cube corner indenters are pyramidal shaped, they always possess some tip radius, which can be approximated by a spherical shape. If the low-k film is quite thin and the tip radius is large, a major portion of the S - a curve may be governed by the spherical portion, and the use of a pyramidal iTF solution would be improper in this range.

However, the pyramidal portion is preferred due to its constant strain approximation. Therefore, it is important to estimate the transition point from spherical to pyramidal, excluding the displacements below this threshold value from the iTF solution.

Ideally, the tip imperfections and pyramidal transitional depth are visualized using experimental imaging techniques such as atomic force microscopy,²⁴ scanning electron microscopy, or using the included piezo scanner to image an inverted AFM tip or calibration “spikes”. However, the analysis of calibration measurements performed on fused quartz can give insights to this transition point.

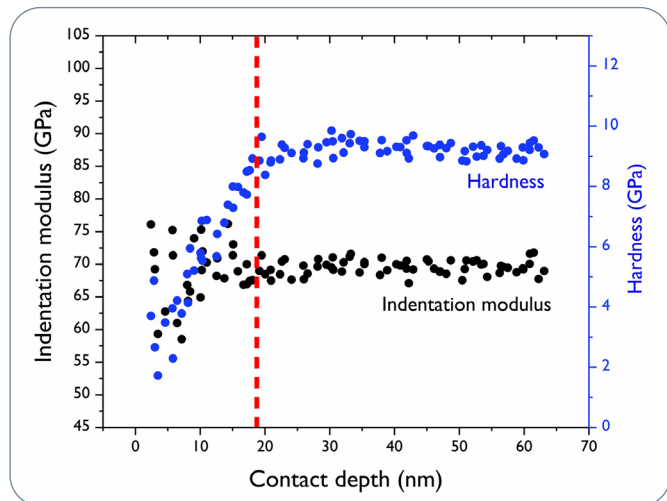


Figure 4. Indentation modulus and hardness of a fused quartz reference calibration sample fused, as measured by a Berkovich indenter.

The transition point from the spherical apex to the pyramidal portion of the indenter is reasonably approximated by the transition to a flat hardness in a fused quartz calibration. The flat hardness for depths greater than 18 nm in Figure 4 is indicative of a fully developed plastic zone. An overview and analysis of the various methods for determining the spherical portion of the probe can be found in Saringer, et. al.,²⁵ and schematics of the probe geometries can be found in the appendix of Fischer-Cripps.²⁶

The effect of tip imperfections on the iTF computation can be seen in Figure 5. Here, a low-k film of 291 nm thickness and 15% porosity was measured by both Berkovich and cube corner indenters. The pyramidal transition depths for these specific Berkovich and cube corner indenters are estimated to be ≈ 18 nm and ≈ 60 nm, respectively. The indentation depth interval used in iTF-pyramidal solution was altered from 10 nm to 90 nm. The results show that the extracted modulus is overestimated at low depths, with a significant depth-dependence. However, above the pyramidal transition point (≈ 60 nm), the computed results stabilize with depth. Still, there are some observable differences between the Berkovich and cube corner indenters. This is thought to be due to some portion of the fitting still belonging to the spherical penetration (< 60 nm) data for a cube corner indenter. If we further exclude the spherical part for both tips and fit only 60-90 nm for the

cube corner indenter, and 20-90 nm for the Berkovich indenter, the results are in very good agreement with only a 1.2% difference, as seen in Figure 6.

These results highlight the importance of continuous monitoring of probe wear. Newer and sharper probes, with smaller radii/imperfections may provide increased range of usable indentation depths for iTF analysis.

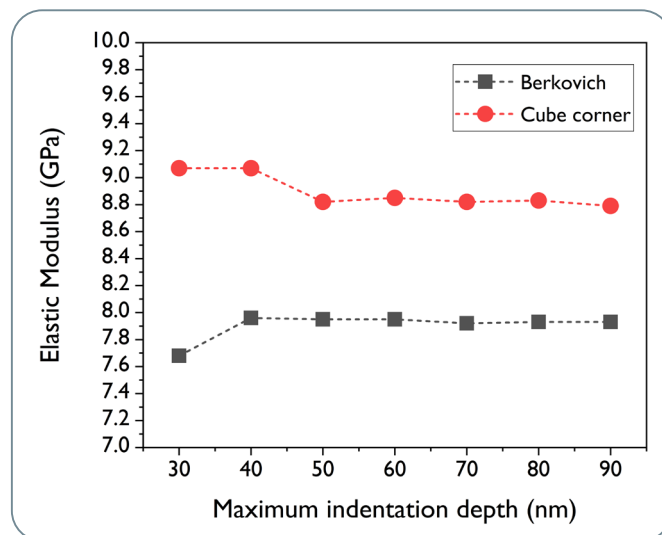


Figure 5. The impact of indentation depth interval on the iTF results extracted for a 291 nm thick, 15% porous low-k film.

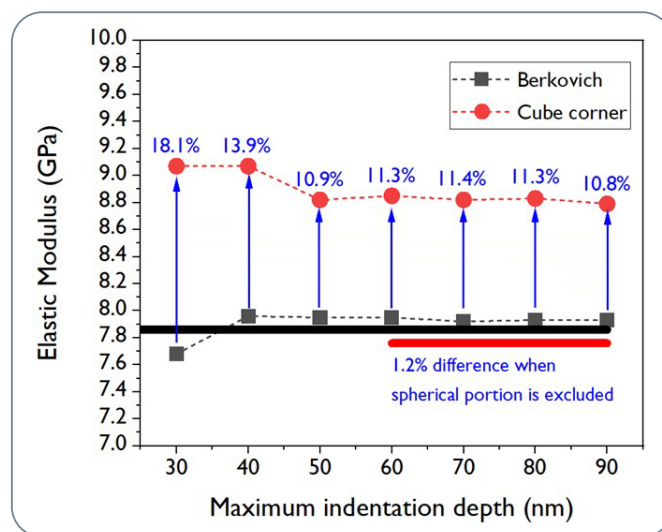


Figure 6. The impact of selecting the proper indentation depth interval by excluding spherical penetration regime from iTF-pyramidal analysis (continuous red and black lines show the iTF results for cube corner and Berkovich indenters, when only pyramidal indentations of both tips are included in the analysis).

c. Maximum indentation depth

The iTF software also requires that the user input the **maximum** indentation depth to be used for the analysis (i.e., ignore displacements greater than...). For thin, low-k films, the main criteria for this selection would be the occurrence of cracks and/or delamination from the substrate. As low-k films are typically deposited in such a way that develops an intrinsic tensile stress, the formation

of cracks on the thin film surface, or the delamination from the substrate is a frequent observation.²⁷ Once this happens, the stiffness used for the experimental S - a is no longer reliable. Therefore, only the displacements prior to these events should be used in the iTF analysis. Presence of cracks and/or delamination from the substrate can usually be detected by the kinks/pop-ins in the force-displacement curves, as shown in Figure 7.

As a summary, it is recommended that the indentation depth range selected for iTF analysis start from the spherical-pyramidal transition point, and end prior to any cracking/delamination event of the thin film/substrate system. In the absence of any cracking/delamination event, the depth at which the indenter penetrates the substrate becomes the maximum indentation depth for iTF.

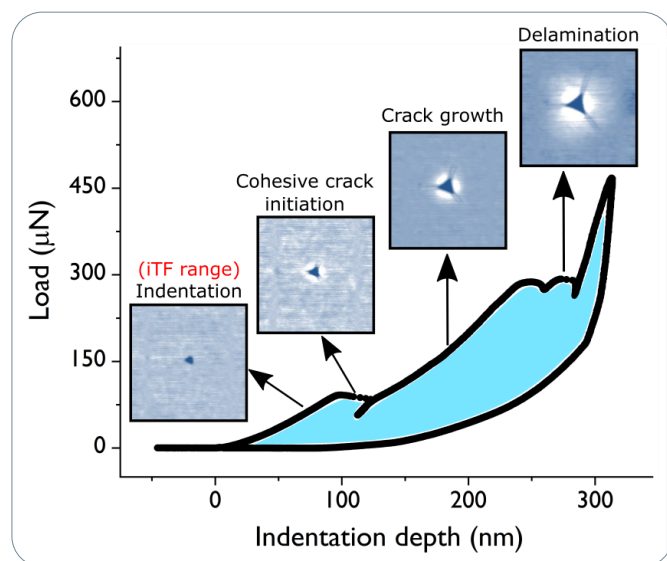


Figure 7. Possible fracture events during indentation of low-k thin films, detected by the kinks/jumps in force-displacement data.²⁸ The region marked as 'indentation' is suitable for iTF analysis.

d. Iteration range for the plasticity parameter, η

A key advantage of the iTF analysis is its ability to consider the thinning of the material due to plastic flow during indentation. This is realized by incorporating the plasticity parameter, η , as a free parameter into the fitting algorithm.

If a custom iTF solution is chosen by the user, the iteration range for η needs to be indicated. By definition ($t_{eff} = t - \eta h$), one would expect η to vary between '0' for fully elastic deformation and '1' for fully plastic deformation for nearly all cases. However, for porous low-k films, fitted η values greater than 1 is a frequent observation. This is thought to be related to the densification of the porous network during indentation, which could lead to stiffening with increasing indentation depth. The calculations using the theoretical solution indicate that the plasticity parameter, η , tends to shift the theoretical S - a curve more dominantly towards larger indentation depths, which agrees with potential densification-induced stiffening.

Therefore, if densification is suspected, it is recommended that the plasticity parameter should be iterated between 0 and 2 for low-k analysis using the iTF platform.

IV. Conclusions

This paper addresses the important aspects of measurement and execution of the iTF analysis, with examples given for thin, porous, low-k films. Through proper application of the presented procedures (see Appendix C for a summary), the process of obtaining substrate-independent properties of low-k materials is greatly simplified. Such properties are particularly valuable for optimizing the reliability and performance of these technologically relevant material. The iTF analysis can also be utilized in a similar manner for other materials with different elastic mismatch ratios (see Appendix B for some examples) with minor adjustments, such as to the plasticity parameter or substrate properties.

V. References

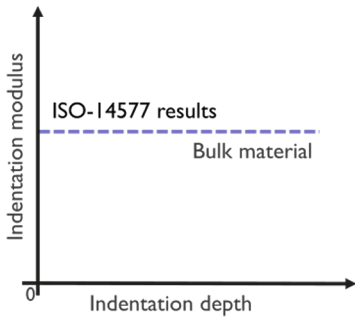
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VI. Appendix

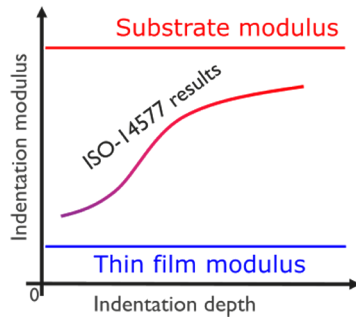
a. A brief overview of indentation and iTF Analysis for semiconductor applications

Bulk Sample Indentation

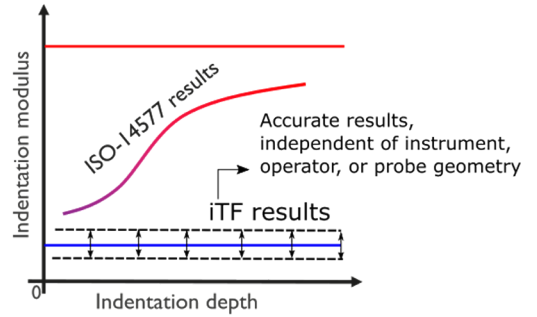


Constant modulus for a linear isotropic material.

Thin Film Indentation → Intrinsic Thin Film Analysis



Unable to retrieve intrinsic thin film modulus by conventional data analysis.



iTF analysis to extract substrate-independent properties of thin films.

b. Different combinations of layered systems

Figure 8 shows the iTF analysis applied for a variety of thin films with different elastic mismatch ratios with the substrate. Each group of materials include variations in thickness (as shown in parentheses), porosity and processing scheme. Measurements were performed by a Berkovich indenter.

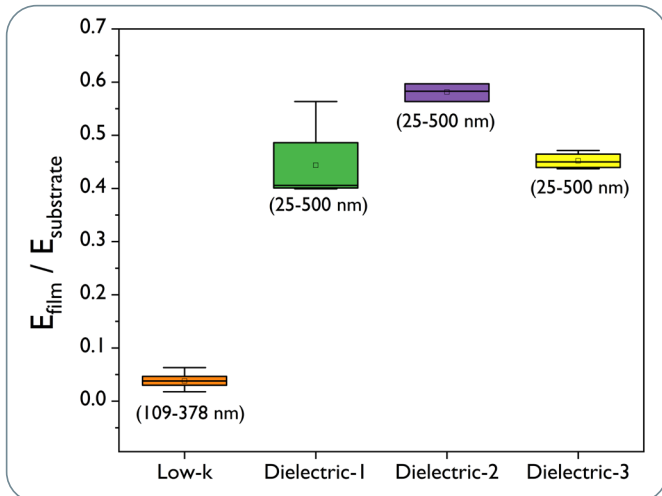
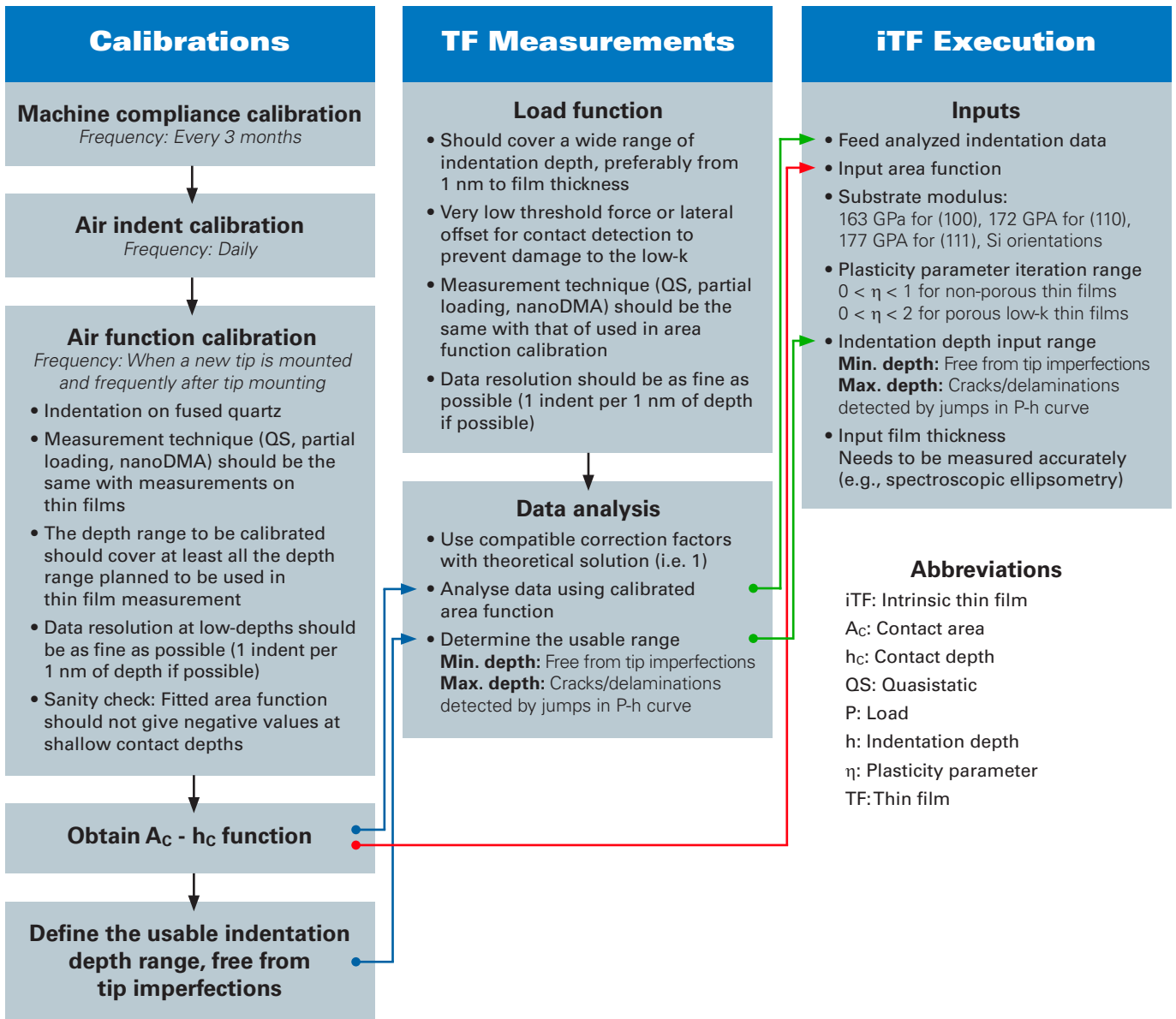


Figure 8. Different combinations of thin films on a Si substrate, analyzed by iTF to obtain intrinsic thin film properties.

c. Summary of iTF analysis procedure for low-k materials



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