

**NANOINDENTATION OF
SILICON CARBIDE WAFER COATINGS**



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INTRO

Single crystal silicon carbide (c-SiC) wafers have the potential to be used in the production and manufacturing of microelectronic devices such as integrated circuits (IC) or photovoltaic (PV) cells. Most wafers are formed from highly pure single crystals and can range in size from 25.4 mm to 300 mm. The wafer acts as the substrate for the micro-devices that are fabricated in, and over, the wafer. Doping or ion implantation, photolithographic patterning, deposition of different materials, and etching are all examples of the many micro-fabrication process steps a wafer can go through.

WAFER FABRICATION CONCERNS

The fabrication process for microelectronic devices can have over 300 different processing steps and can take anywhere from six to eight weeks. During this process the wafer substrate must be able to withstand the extreme conditions of manufacturing, since a failure at any step would result in the loss of time and money. The hardness and strength of a wafer must be much greater than the conditions imposed during manufacturing to insure a failure will not occur.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Nanoindentation Tester (seen below) is used to measure the Vickers hardness (Hv) and Young's Modulus (E) of coatings on silicon carbide wafers. The wafer has a 2 μm coating on top of a silicon carbide substrate. By determining the Vickers hardness and Young's Modulus of these coating the role the substrate plays in these properties can be examined.



NANOINDENTATION MEASUREMENTS (NHT)

Principle

The Nanoindentation test 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 partial or complete relaxation occurs. This procedure is performed repetitively; at each stage of the experiment the position of the indenter relative to the sample surface is precisely monitored with a linear variable differential transformer (LVDT).

For each loading/unloading cycle, the applied load value is plotted with respect to the corresponding position of the indenter. 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.

The NHT is especially suited to load and penetration depth measurements at nanometer scales and has the following specifications:

Maximum displacement	: 25 μm
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.08 μN
Load Resolution (Noise Floor)	: 2 μN

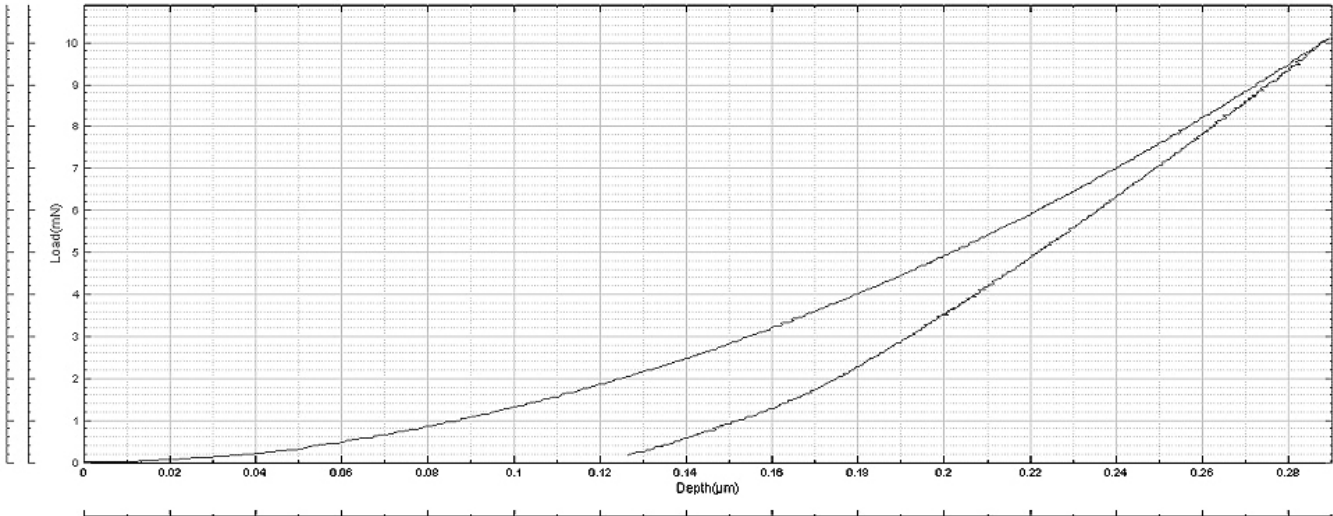
Analysis of Indentation Curve

A typical load/displacement curve is shown below, from which the compliance $C = 1/S$ (which is the inverse of the contact stiffness) and the contact depth h_c are determined after correction for thermal drift.

A simple linear fit through the upper 1/3 of the unloading data intersects the depth axis at h_t . The stiffness, S , is given by the slope of this line. The contact depth, h_c , is then calculated as:

$$h_c = h_m - \varepsilon(h_m - h_t)$$

where ε depends on the investigated material.



In practice, a more meticulous approach is used where a power law function is used to describe the upper 80% of the unloading data.

$$P = P_{\max} \left(\frac{h - h_0}{h_m - h_0} \right)^m$$

where the constants m and h_0 are determined by a least squares fitting procedure.

The contact stiffness S ($=1/C$) is given by the derivative at peak load:

$$S = \left(\frac{dP}{dh} \right)_{\max} = m P_{\max} \left[\frac{(h_m - h_0)^{m-1}}{(h_m - h_0)^m} \right] = m P_{\max} (h_m - h_0)^{-1}$$

$S = dP/dh$

and the tangent depth, h_t , is thus given by:

$$h_t = h_m - \frac{P_m}{S}$$

$\epsilon(h_m - h_t)$

$h_t \quad h_c$

The contact depth, h_c , is then:

$$h_c = h_m - \epsilon(h_m - h_t)$$

where ϵ now depends on the power law exponent, m . Such an exponent can be summarized for different indenter geometries:

Elastic indentation Behavior (indenter geometry)	m (power law exponent)	ϵ
Flat	1	1
Paraboloid	1.5	0.75
Conical	2	0.72

Calculation of Young's Modulus and Hardness

Young's Modulus

The reduced modulus, E_r , is given by:

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

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.

Hardness

The hardness is determined from the maximum load, P_{max} , divided by the projected contact area, A_c :

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

Test conditions and procedure

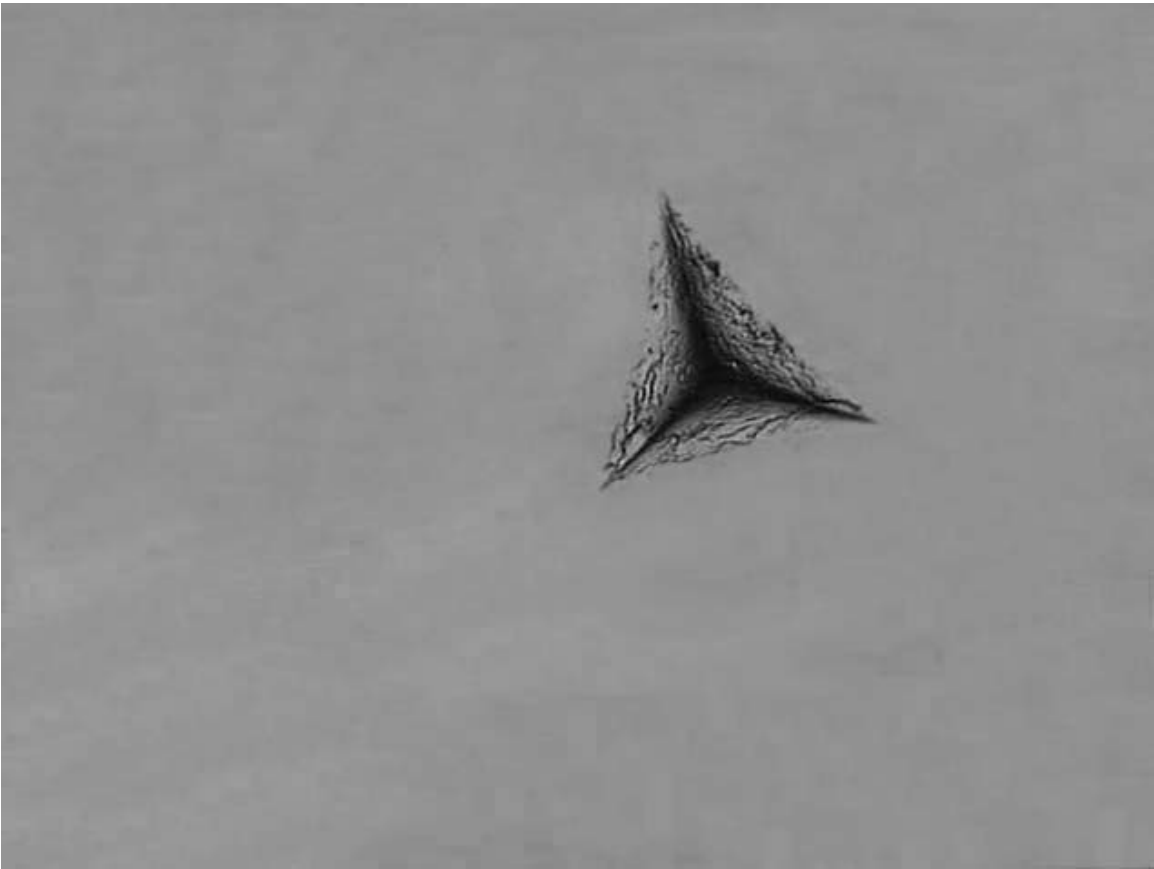
The following indentation parameters were used:

Maximum force (mN)	2
Loading rate (mN/min)	4
Unloading rate (mN/min)	4
Computation Method	ASTM E-2546 & Martens Hardness
Indenter type	Berkovich Diamond KM

Results

These full results present the measured values of Hardness and Young's modulus as the penetration depth (d) with their averages and standard deviations. It should be considered that large variation in the results can occur in the case that the surface roughness is in the same size range as the indentation.

Wafer				
	Hv [Vickers]	H [GPa]	E [GPa]	Δ d [μm]
1	2882.20	30.50	328.95	66.15
2	2540.62	26.77	353.17	68.13
3	2949.56	31.21	347.90	69.67
4	2920.54	30.91	319.74	66.75
5	2620.70	27.73	368.72	66.16
Average	2782.72	29.42	343.70	67.37
Standard deviation	188.14	2.03	19.53	1.52



CONCLUSION:

In conclusion, the Nanovea Nanindentation Tester demonstrates reproducibility and precise indentation at extreme low levels. Additionally, the controlled and closely monitored environment allows the measurement of hardness to use as a quantitative value for comparing a variety of samples. This test also shows that it can measure other characteristics, such as Young's modulus and maximum depth. Here we have shown the ability to control the specific load being applied to test the thin coatings on top of silica carbide wafers and how the wafer affects the hardness of the coating.