

**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  $\mu\text{m}$  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.



## MEASUREMENT PRINCIPAL

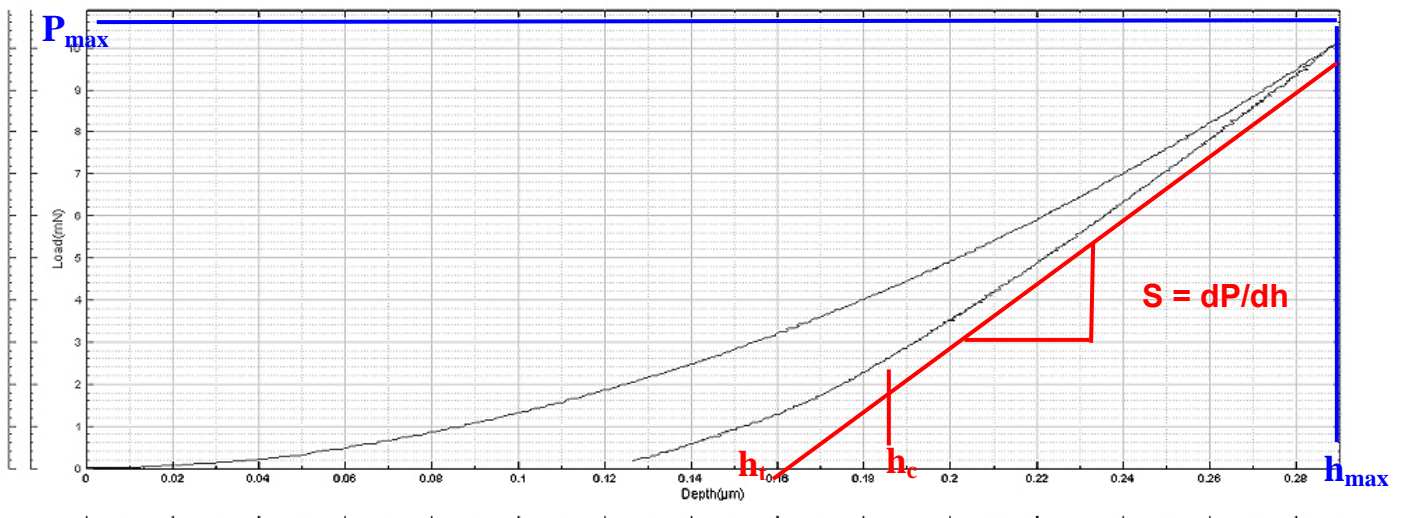
Nanoindentation is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an already established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. The load is applied by a piezo actuator and the load is measured in a controlled loop with a high sensitivity load cell. During the experiment the position of the indenter relative to the sample surface is precisely monitored with high precision capacitive sensor.

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

Maximum displacement (Dual Range)	: 50 $\mu\text{m}$ or 250 $\mu\text{m}$
Depth Resolution (Theoretical)	: 0.003 nm
Depth Resolution (Noise Level)	: 0.05 nm
Maximum force	: 400 mN
Load Resolution (Theoretical)	: 0.03 $\mu\text{N}$
Load Resolution (Noise Floor)	: 1.5 $\mu\text{N}$

### Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.



### Hardness

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

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

## Young's Modulus

The reduced modulus,  $E_r$ , is given by:

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

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  $\nu_i$  are the Young's modulus and Poisson coefficient of the indenter and  $\nu$  the Poisson coefficient of the tested sample.

### How are these calculated?

A power-law fit through the upper 1/3 to 1/2 of the unloading data intersects the depth axis at  $h_i$ . The stiffness,  $S$ , is given by the slope of this line. The contact depth,  $h_c$ , is then calculated as:

$$h_c = h_{\max} - \frac{3P_{\max}}{4S}$$

The contact Area  $A_c$  is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is  $A_c=24.5h_c^2$  For Cube Corner indenter, the area function is  $A_c=2.60h_c^2$  For Spherical indenter, the area function is  $A_c=2\pi Rh_c$  where  $R$  is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant  $E$ , given the formula:  $\sigma = E\epsilon$  where  $\sigma$  is the stress,  $E$  is the elastic modulus of the material, and  $\epsilon$  is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate

$$\sigma = \eta \frac{d\epsilon}{dt}$$

relationship can be given as,

where  $\sigma$  is the stress,  $\eta$  is the viscosity of the material, and  $d\epsilon/dt$  is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen. Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip might be a better choice.

### Other tests possible includes the following:

Work of Indentation, Volume of indent, Plastic & Elastic Work, Visco Elastic , Creep, Compression and others.

## Test conditions and procedure

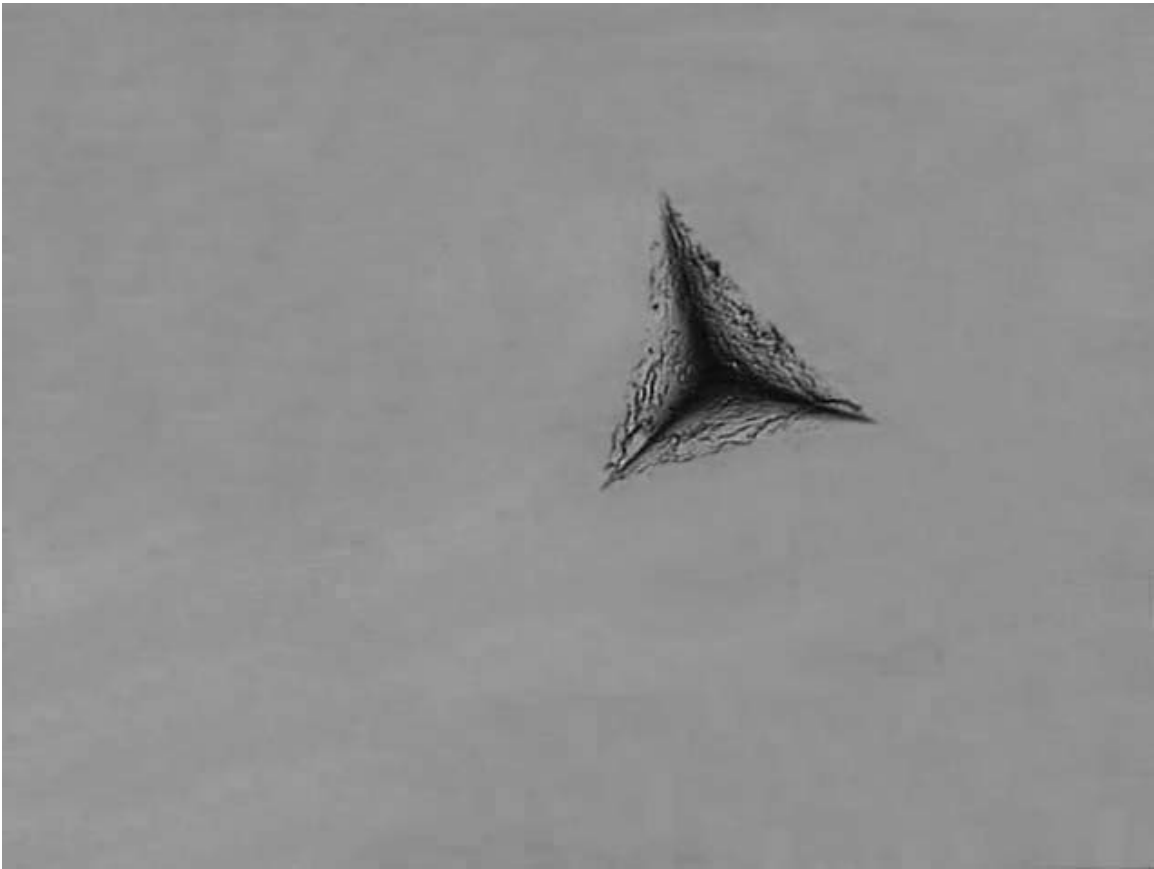
The following indentation parameters were used:

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

## 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.

<b>Wafer</b>				
	<b>Hv [ Vickers ]</b>	<b>H [ GPa ]</b>	<b>E [ GPa ]</b>	<b>Δ d [ μm ]</b>
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	<b>2782.72</b>	<b>29.42</b>	<b>343.70</b>	<b>67.37</b>
Standard deviation	<b>188.14</b>	<b>2.03</b>	<b>19.53</b>	<b>1.52</b>



## 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.