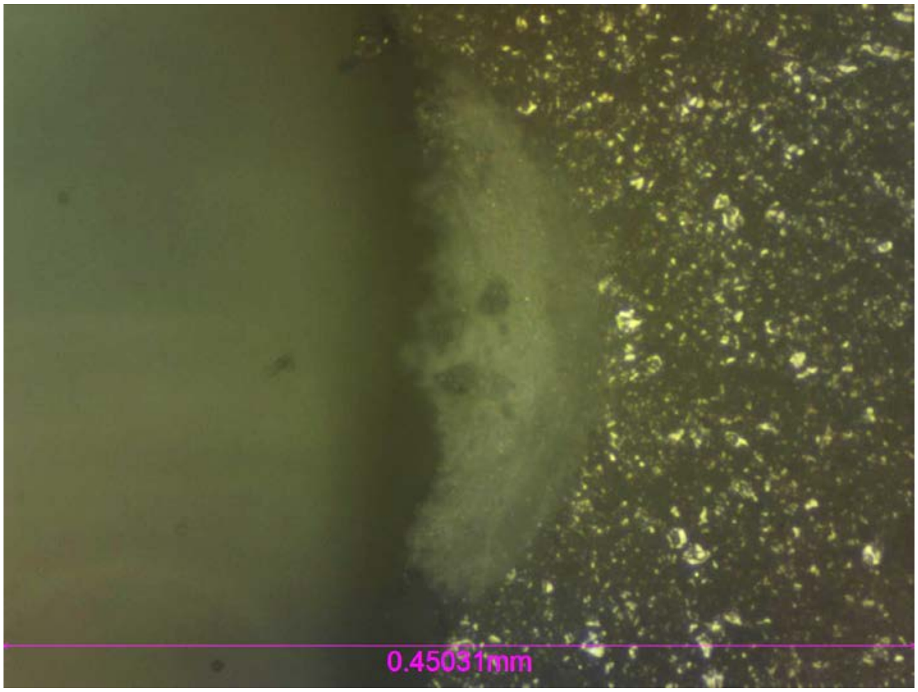
EDGE CHIPPING RESISTANCE USING MACROINDENTATION TESTING



Prepared by

Ali Mansouri

6 Morgan, Ste156, Irvine CA 92618 · P: 949.461.9292 · F: 949.461.9232 · nanovea.com

INTRODUCTION

The resistance of the edges of brittle materials to chipping or flaking from concentrated loads is a critical property for dental restoration ceramics, resin composites, edge-mounted optical devices, ceramic tool bits, thin semiconductor chips, and many other materials. The edge chipping test provides a method to quantify and measure the fracture resistance, toughness, and edge chip strength of these materials. This method uses a conical indenter to chip the rectangular edge of a brittle sample at set distances from the edge. Archeological evidence has revealed that this method is similar to the way early humans selected stones to make tools and weapons. Hundreds of thousands of years later, edge chipping tests remain a critical tool for applications where edge toughness is concerned.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Macroindentation Mode, is used to evaluate the edge chipping resistance of a thin ceramic block. Using a Rockwell C indenter to apply a point load at set distances between 0.05 and 0.40 mm from the edge of the sample, we will identify the flaking load at which the edge will chip.

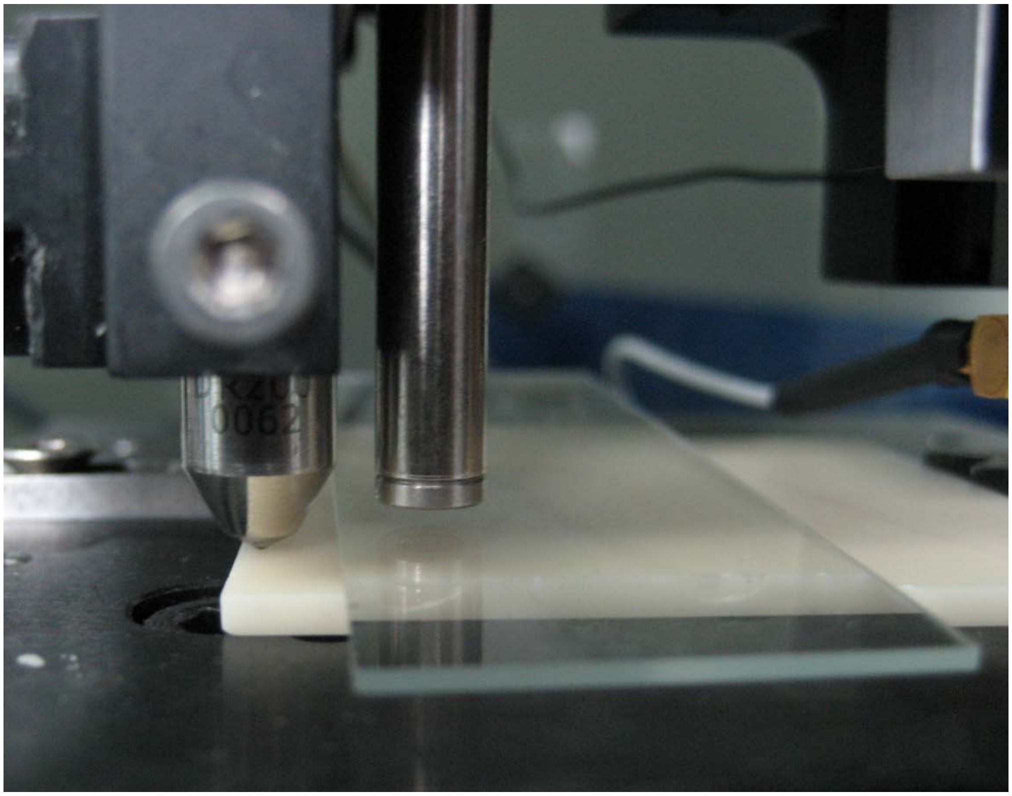


Fig. 1: Indenter and depth sensor on the test sample.

TEST CONDITIONS

The 50x50x2 mm ceramic block was carefully fixed on the sample stage. The indenter tip used was a diamond Rockwell C conical tip of 200 µm diameter. The PID settings were set such that the test stop upon a sudden drop in load. The Nanovea Mechanical Tester was used to perform the Macroindentation tests using the test parameters summarized in Table 1, in order to evaluate the load at which edge chipping occurred on the ceramic sample.

Test parameters	Value
Maximum Force	400 N (but test stops upon failure)
Loading Rate	20 (N/min)
Indenter geometry	Conical
Indenter material (tip)	Diamond
Indenter tip radius	20 µm

Table 1: Test parameters of the Macroindentation measurements.

RESULTS AND DISCUSSION

Several different tests were performed at distances between 0.05 and 0.40 mm from the edge of the sample. The test location was precisely determined using an optical microscope with a previously calibrated microscope-indenter distance. Nanovea's control software ensured the load was immediately removed upon chipping. The acoustic emissions module also helped determine the exact time failure occurred. Table 2 shows the mean load at failure for each distance.

Distance from Edge (µm)	Mean Load at Failure (N)
50	32.12
100	75.87
150	89.73
200	145.50
250	175.85
300	149.30
350	210.40
400	261.10

Table 2: Edge Chipping Test Results

A graph showing the results of 15 different edge chipping tests, along with a superimposed linear regression line is shown in Fig. 2. As can be seen, there was a very strong closeness of fit between the data and the linear regression line.

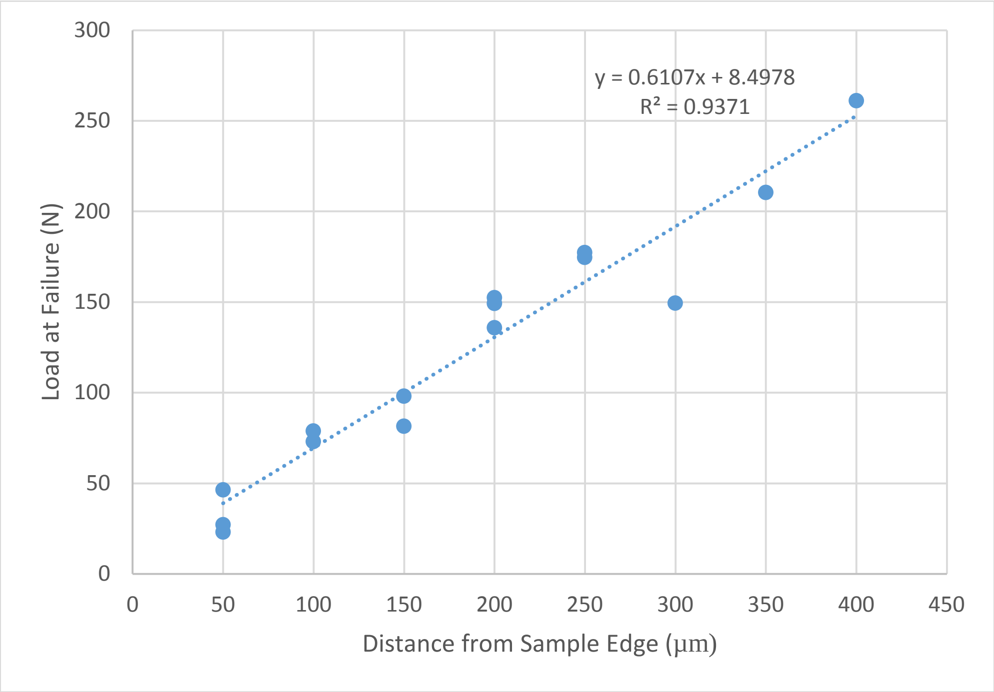


Figure 2: Load at Failure vs Distance From the Sample Edge

The maximum load before chipping occurred can be derived from the Load vs Time Graph of each test, such as the one seen in Fig. 3. Sudden failure creates a clear acoustic emission that can be detected by the Nanovea Mechanical Tester and is shown as a black vertical line. Any continued load application after chipping (overchipping) would show up on the graph as a sudden departure from the linear Load vs Time line. Overchipping can be reduced or prevented using Nanovea' s control software .

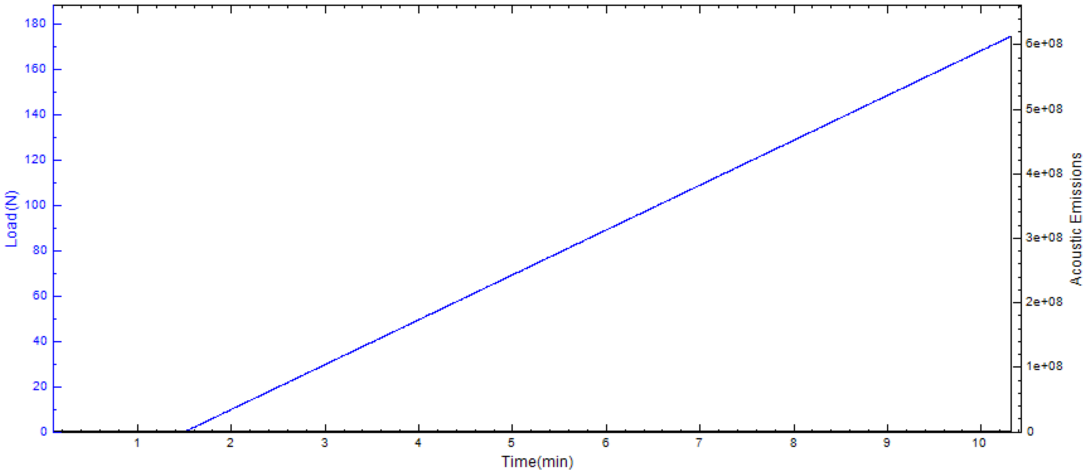
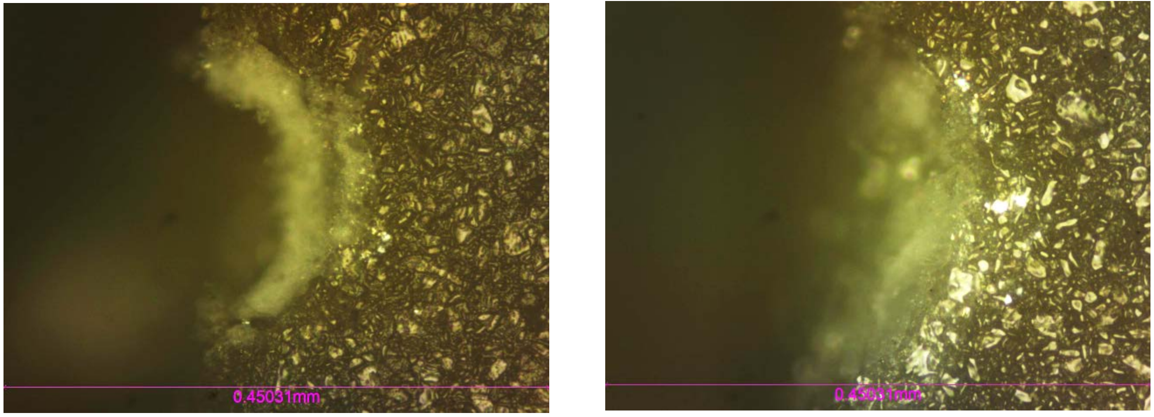


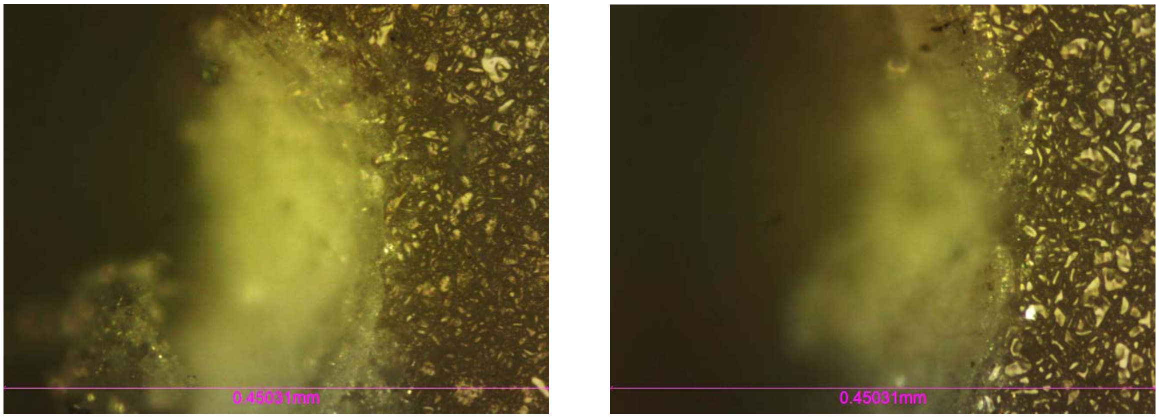
Figure 3: Load vs Time Graph for the Test 250 µm from the edge

The chips themselves can be studied under an optical microscope after the testing is complete. As can be seen in Fig. 4, the chips get progressively wider and deeper the farther away the load is applied from the edge. These can be further characterized to determine the exact surface area and volume lost, as well as get a high resolution 3D topography map using the Nanovea Profilometer



(a) 50 µm from the edge

(b) 100 µm from the edge



(c) 150 µm from the edge

(d) 200 µm from the edge

Figure 4: Micrographs of chipping at different distances– Magnification 100x

CONCLUSION

In this study, we have shown how the Nanovea Mechanical Tester, in Macroindentation mode, can perform an edge chipping test on a brittle material. The distances from the edge were precisely determined using an optical microscope. It was shown that the load feedback loop, along with the acoustic emissions module can be used to identify the exact point before chipping failure occurs. The system can also extend in the micro and nano range for materials needing much lower loads for failure. Edge chipping testing can be done at different environmental conditions using temperature and humidity modules. By allowing the application of a precisely controlled load at a desired position, the Nanovea Mechanical Tester provides a

superior tool to quantitatively evaluate and compare the edge chipping resistance of various brittle materials.
The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young’s modulus, fracture toughness, adhesion, wear resistance and many others.

To learn more about the Nanovea Mechanical Tester or Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

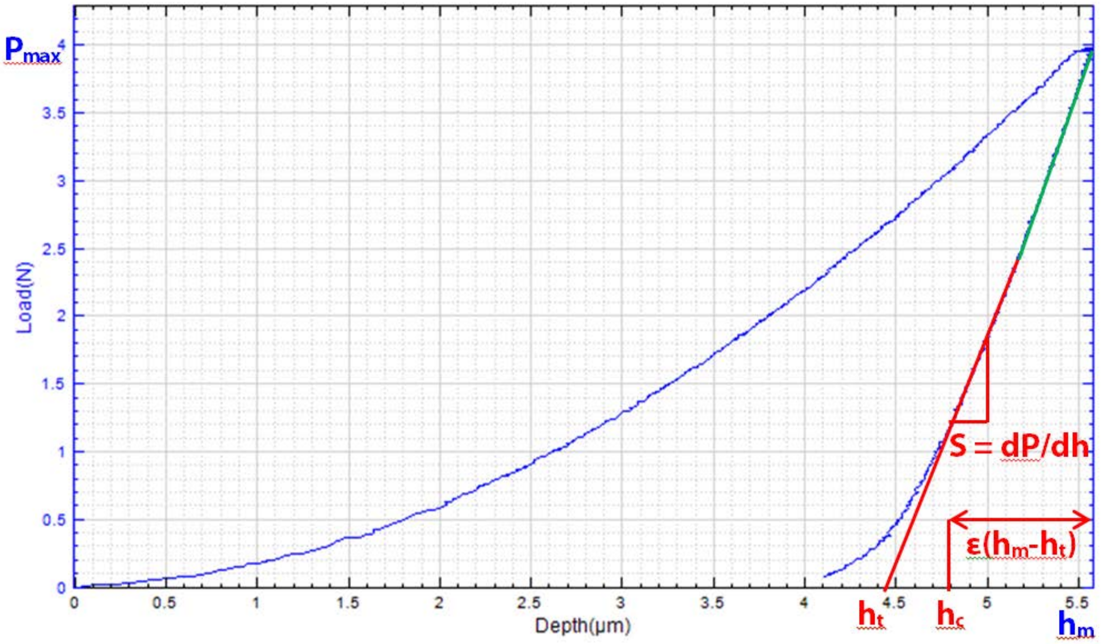
PRINCIPLE OF INSTRUMENTED INDENTATION TEST

The indentation test is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an 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. During the experiment the position of the indenter relative to the sample surface is precisely monitored.

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.

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, $P_{\max}$, divided by the projected contact area, A_c:

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

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 ν_i are the Young's modulus and Poisson's ratio of the indenter and ν the Poisson's ratio of the tested sample.

How are these calculated?

A power-law fit through the upper 1/3 to1/2 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:

$$h_c = h_{\max} - {}^{3P_{\max}}\!\big/_{4S}$$

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: $\sigma = E\varepsilon$ where σ is the stress, E is the elastic modulus of the material, and ε 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 relationship can be given as $\sigma = \eta \frac{d\varepsilon}{dt}$, where σ is the stress, η is the viscosity of the material, and $d\varepsilon/dt$ 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 is a better choice.