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MICROPARTICLE COMPRESSION STRENGTH WITH MICROINDENTATION

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INTRO

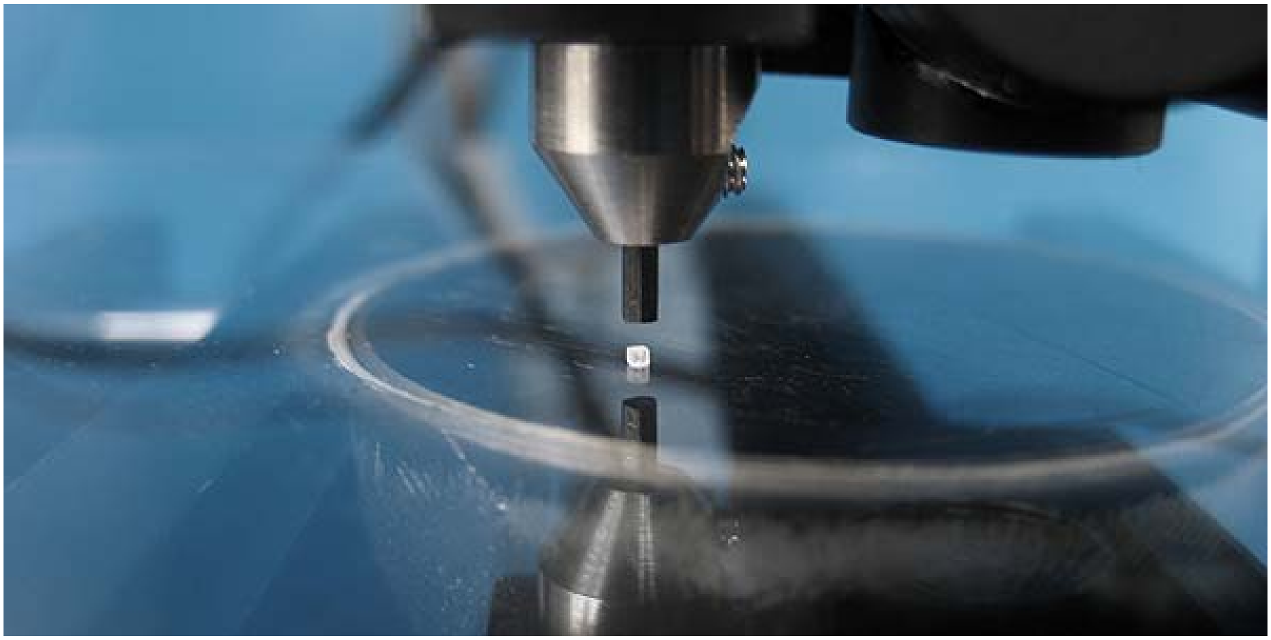
Compression strength has become a vital control measure in developing and improving new and existing microparticles and or micro features seen today. Microparticles have various shapes, sizes and can be developed from ceramics, glass, polymers, and metals. Uses include drug delivery, food flavor enhancement, concrete formulations among many others. Micro features such as micropillers, microspheres and others are also similarly in need of this type of measurement. Controlling the mechanical properties of microparticles and or micro features will be critical to their success and requires the ability to quantitatively characterize their mechanical integrity.

IMPORTANCE OF DEPTH VERSUS LOAD COMPRESSION STRENGTH

Standard compressive measurement instruments are not capable of low loads and fail to provide adequate depth data for microparticles. By using Nano or Microindentation the compression strength of nano or microparticles, soft or hard, can be precisely measured.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Microindentation mode (seen below), is used to measure the compression strength of salt microparticles with a flat tip.

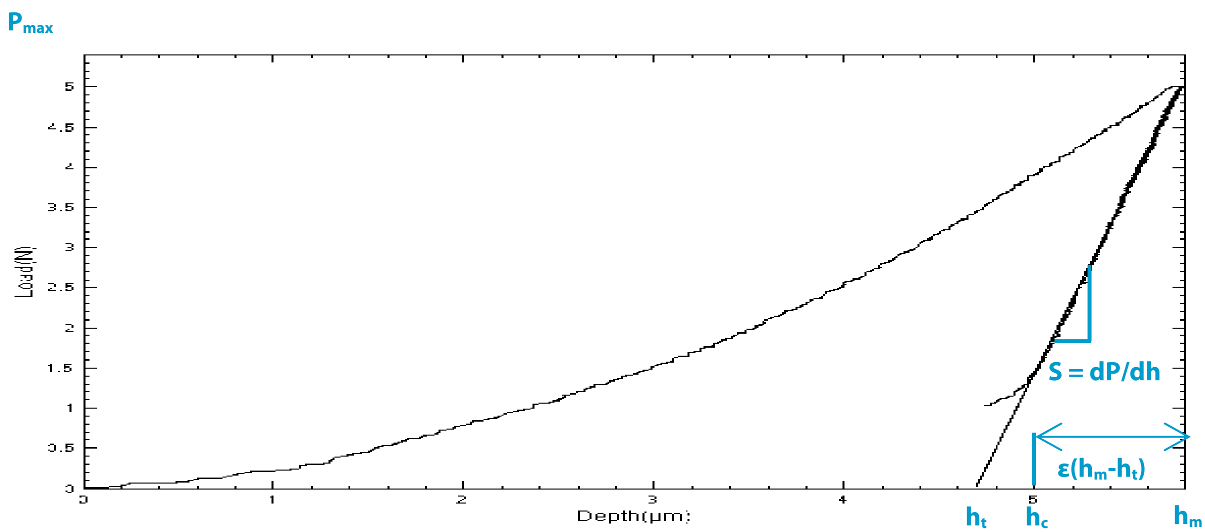


MEASUREMENT PRINCIPLE

Microindentation 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 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 an optical non-contact depth sensor. 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 MHT is especially suited to perform tests of penetration depths in the micrometer scale and has the following specifications: • Displacement measurement: Non-contact optical sensor • Displacement resolution: 10 nm • Maximum Indenter range: 300µm • Load application: Z motor controlled with force feedback loop • Load range: 0–30N • Normal load noise floor resolution: 1.5mN • Minimum load: 10mN • Maximum load: 30N • Contact force hold time: Unlimited.

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 hc are determined after correction for thermal drift.



Calculation of Young's Modulus and Hardness

Young's Modulus: The reduced modulus, Er, 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 Ac from the indentation curve using the area function, Ac 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 Ei and ν i are the Young's modulus and Poisson coefficient of the indenter and E and ν the Young Modulus and Poisson coefficient of the tested sample. Hardness: The hardness is determined from the maximum load, Pmax, divided by the projected contact area, Ac: $H = \frac{P_{max}}{A_c}$

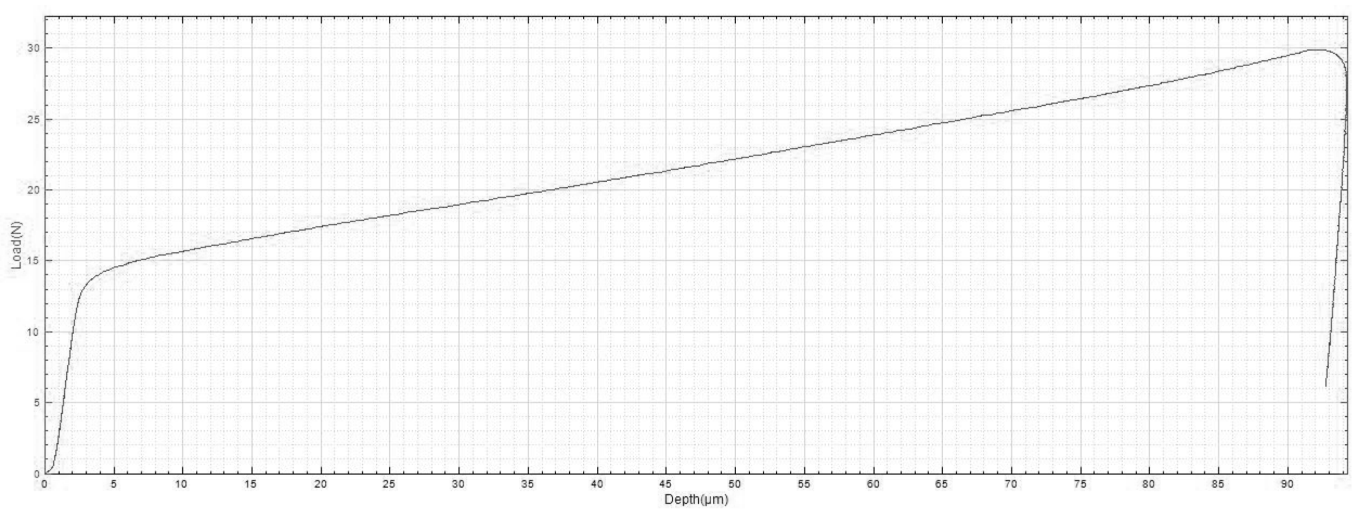
TEST CONDITIONS AND PROCEDURE

The following indentation parameters were used:

Maximum force (N)	30
Loading rate (N/min)	60
Unloading rate (N/min)	60
Creep (s)	0
Indenter type	Flat Steel | 1 mm Diameter

RESULTS

Sample 1 | Photo from left to right: Prior to test, after test and zoom in after test



	Size(µm)	Failure Load (N)	Strength At Failure (MPa)
Sample 1	**250**	**3.4**	**54**

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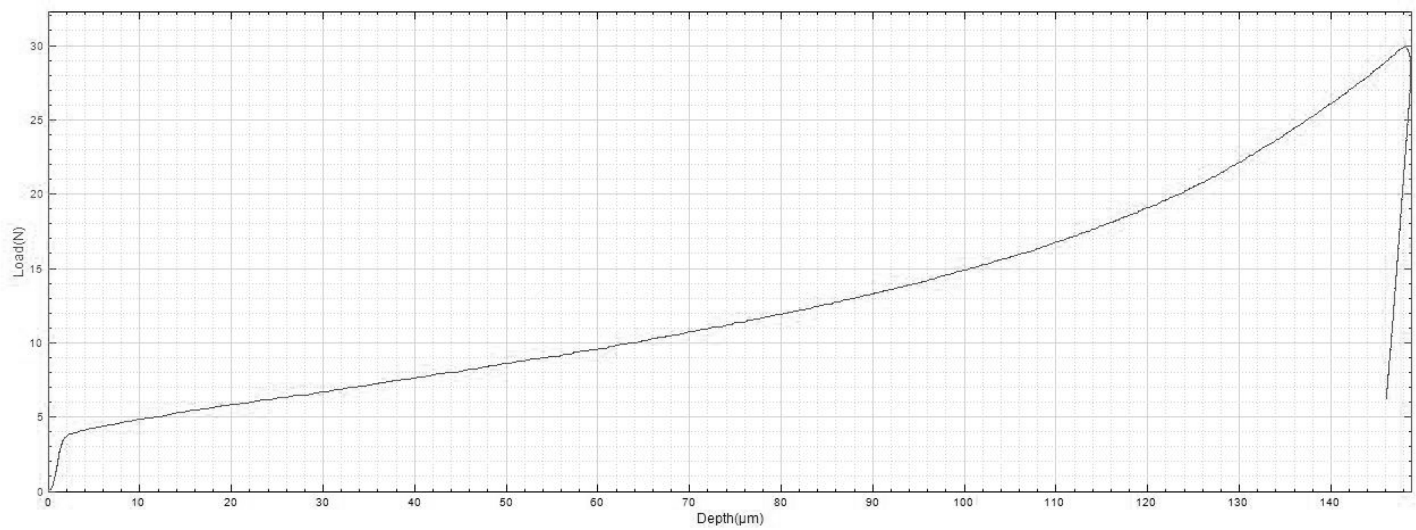
RESULTS

Sample 2 | Photo from left to right: Prior to test, after test and zoom in after test



	Size(µm)	**Failure Load** (N)	**Strength At Failure** (MPa)
Sample 2	**377**	**12.5**	**88**

DISCUSSION & CONCLUSION:

In conclusion, we have shown how the Nanovea Mechanical Tester, in Microindentation mode, can provide compression strength of microparticles. We suspect that the difference in the strength at failure varies based on micro cracks present in the particles that will affect the failure point. Acoustic emission could be used to measure the cracks propagation during the test. The use of the large Z range allows test with displacement up to 50mm at load from the 0.1N to 200N. Lower loads down to 0.1mN, is possible for extremely sensitive compression tests using the nano module. Displacement resolution down to sub nanometer allows study of very small particles or features. Multi-cycling of the load can also be used to study the fatigue properties. It would also be possible to test the hardness and elastic modulus of on the surface of the particle using a sharper Berkovich tip. AFM or optical profiler integration is an option to measure the residual surface deformation or damages.