NANO INDENTERS

FROM MICRO STAR TECHNOLOGIES

Micro Star makes a variety of nano indenters following defined standards or custom requested geometries and dimensions. Micro Star calibration laboratory complies with the requirements of the International Standard ISO/IEC 17025.[1]

Standard nano indenters and custom indenters with applicable geometry are accepted only if the pertinent dimensions and angles are within the ranges specified by the ISO 14577-2[2] which defines internationally accepted micro and nano indenter tolerances.

Figure 1 shows an example of a nano indenter with its three parts, the diamond, the holder and the bond.

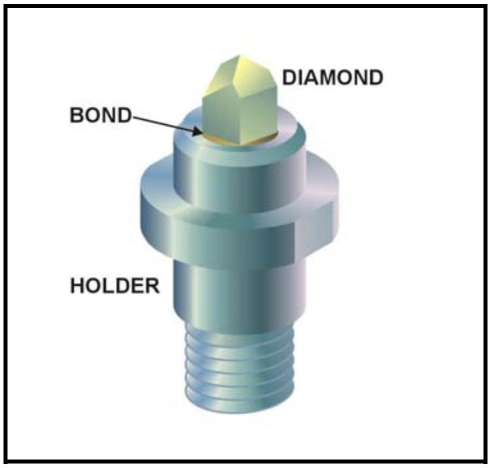


Figure 1 Nano Indenter Example

There are numerous **geometries** available for the indenter shape such as three sided pyramids, four sided pyramids, wedges, cones, cylinders or spheres. The tip end of the indenter can be made sharp, flat, or rounded to a cylindrical or spherical shape.

Diamond and sapphire are the primary **materials** of Micro Star nano indenters but other hard materials can also be used such as quartz, silicone, tungsten, steel, tungsten carbide and almost any other hard metal or ceramic. Micro Star also has **conductive diamond** available.

Nano indenters are mounted on **holders** which could be the standard design from a manufacturer of nano indenting equipment, or a custom design. Micro Star can design and make the holders or use the ones supplied by the customer.

The **holder material** can be steel, titanium, machinable ceramic or other suitable material. In most cases Micro Star attaches the indenter to the holder using a rigid metal bonding process.

Micro Star can provide **Standard Traceable Nano Indenters**. They are inspected and measured with equipment and standards traceable to the NIST[3] or PTB[4] and supplied with a **calibration certificate** containing pertinent measurement records and references.

TIP GEOMETRIES

The tabulated illustrations show most of the indenter geometries available from Micro Star. Each type of indenter is designated by two letters in blue. There are examples of TEM [5] micrographs of critical measured dimensions, which are included with traceable indenters or per request.

Figure 2 shows sharp point 3-sided pyramid geometry. The defining angle **a** is the angle between the axis and any of the faces. The 3 faces are symmetrically placed around the axis 120° apart. The Berkovich[6] indenter is designed to have the same area as the Vickers indenter at any given indentation depth. The modified Berkovich [7] indenter is designed to have the same projected area as the Vickers indenter at any given indentation depth.

Micro Star makes a wide range of 3-sided indenters with custom angles and flat or round ends of any compatible size.

Figure 3 shows sharp 4-sided geometries. Most 4-sided indenters have their faces symmetrically placed around the axis 90° apart and the defining angle **a** is measured between the axis and each face. The standard Knoop indenter has a special geometry as shown.

A sharp 4-sided pyramid tip always ends in a small line (shown on the TEM micrograph) called the **line of conjunction**.

Figure 4 shows 3 and 4-sided indenters with flat and rounded tips. **Figure 5** shows wedge indenter configurations.

Figure 6 shows the general cone geometry with sharp, flat or spherical end. There are no recognized standard angles or sizes for conical indenters.

The “taper cone” configuration makes possible a very small cylindrical rod at the end of a large body as shown on **Figure 7**. Many Micro Star nano indenters are made at the end of a taper cone. The small volume of material to be shaped to a particular geometry provides higher precision and minimizes anisotropic[8] effects.

Figure 8 shows a spherical indenter. Spheres made of various materials are attached to the indenter holder. Many available spheres are rough at microscopic scale. If required, the end surface of the sphere is polished up to a few nanometers and measured with the AFM[9].

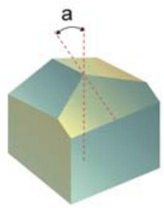
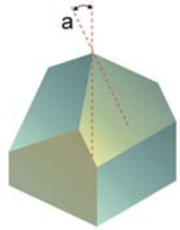
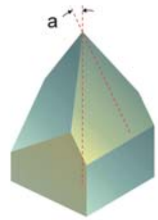
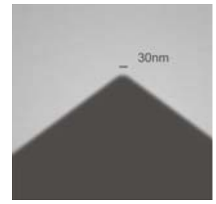
BERKOVICH **TB** 	**CUBE CORNER** **TC** 	**3-SIDED CUSTOM** **TD** 	**SHARPNESS** TEM micrograph 
Berkovich: a = 65.03° Mod. Berkovich: a = 65.27° Available as **Traceable Standard**	Cube corner: a = 35.26° Available as **Traceable Standard**	Custom 3-sided indenters: 80° > a > 20°	Micro Star 3-sided sharp indenters tip radius < 50nm.

Figure 2 SHARP 3-SIDED INDENTERS

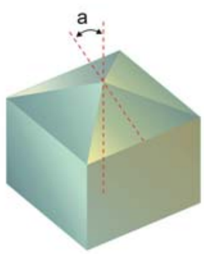
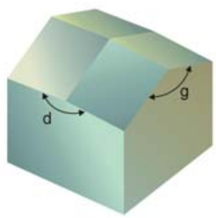
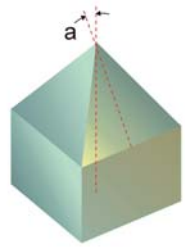
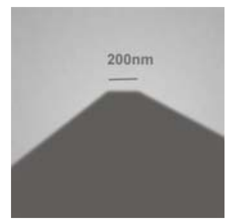
VICKERS **FV** 	**KNOOP INDENTER** **FK** 	**4-SIDED CUSTOM** **FD** 	**END LINE** TEM micrograph 
Standard Vickers indenter: a = 68.00° Available as **Traceable Standard**	Standard Knoop indenter defined by 2 angles: d = 172.50°, g = 130.00°	Custom 4-sided indenters: 80° > a > 20°	Micro Star indenters maximum line of conjunction: 400nm.

Figure 3 SHARP 4-SIDED INDENTERS

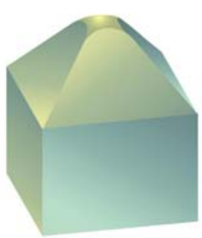
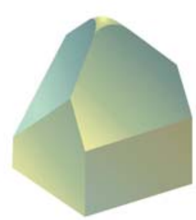
4-SIDE FLAT END **FP** 	**4-SIDE ROUND END** **FR** 	**3-SIDE FLAT END** **TP** 	**3-SIDE ROUND END** **TR** 
Flat square side from 500nm to any compatible size.	End radius from 100nm to any compatible value.	Flat triangle from 300nm side to any compatible size.	End radius from 100nm to any compatible value.

Figure 4 FLAT AND ROUND ED INDENTERS

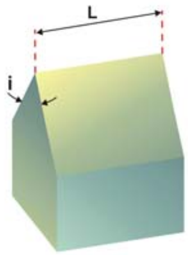
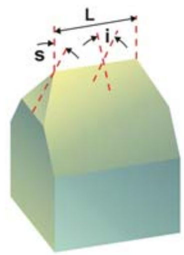
WEDGE INDENTER **WS** 	**TAPERED WEDGE** **WT** 	**CYLINDRICAL WEDGE** **WC** 	**ROUNDED WEDGE** **WR** 
Edge length from 0.3mm to 3mm. Sharp edge radius less than 10nm. Included angle: 30°< i < 90°	Wedge with tapered corners. Edge length from 0.5µ to 2mm. Angles: 30°< i < 90°, 15°< s < 45°	Wedge indenter with cylindrical edge angles same as WT. Radius from 200nm to 5µ.	Wedge indenter with cylindrical edge and rounded corners. Radius from 200nm to 5µ.

Figure 5 WEDGE INDENTERS

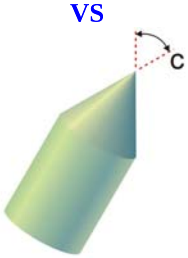
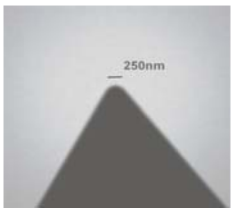
CONE TIP **VS** 	**POINT SHARPNESS** TEM micrograph 	**FLAT END CONE** **VP** 	**ROUND END CONE** **VR** 
Included conical angle: 20° > c > 140°	Micro Star sharp cone radius less than 300nm.	Flat from 500nm diameter to larger compatible sizes.	Spherical end radius <50nm to larger compatible sizes.

Figure 6 CONE INDENTERS

FILAMENT ROD **YS** 	**CONE END ROD** **VS** 	**FLAT END ROD** **YP** 	**ROUND END ROD** **YR** 
Thin cylindrical probe 0.4µ < diameter < 20µ Straight section up to 50µ long. End not defined.	Straight cone at end of cylindrical section. Most Micro Star cone indenters (Type V) are made this way.	Diameter from 20µ to larger sizes compatible with indenter. Straight section up to 0.3mm long.	Spherical end rod. Diameter from 20µ to larger compatible sizes. Straight section up to 0.3mm long.

Figure 7 TAPER CONE INDENTERS

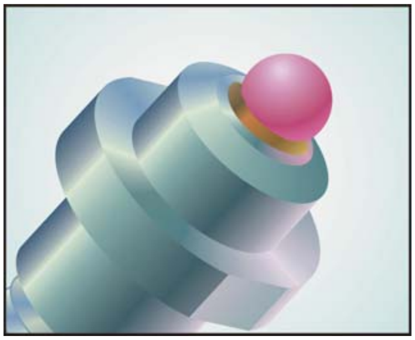


Figure 8 SPHERICAL INDENTER BR
Spheres of sapphire and other materials metal bonded to the holder. Sphere diameters between 5μ and 2mm. Not all intermediate sizes available. Surface polished to < 20 nm roughness[10] on request.

DIAMOND AND OTHER MATERIALS

Diamond is the primary material for nano indentation. Only **single crystal diamond**, free of impurities or inclusions is used. Most Micro Star diamond nano indenters are shaped at one end of a large square section prism, as seen on some of the illustrations.

Diamond indenters are accurately aligned with the **atomic crystal orientation** such that the axis is in line with the 100 direction. One face of 3-sided and all four faces of 4-sided indenters follow the 100 crystal direction. This is illustrated on **Figure 9**. Other crystal orientations are available at customer request.

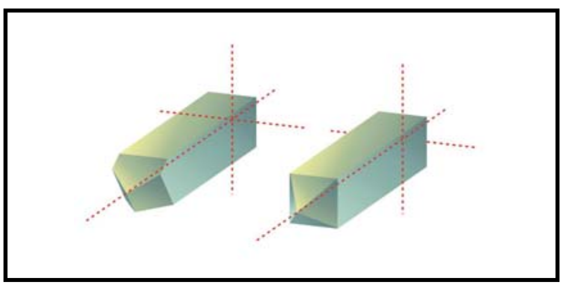


Figure 9 Diamond Atomic 100 Crystal Directions (dotted lines) that Coincide with Micro Star Indenter's Axis

The advantages of diamond for nano indentation come from its exceptional properties of hardness, thermal conductivity and chemical inertness, which surpass any other known material.

The strong **diamond anisotropy** is a disadvantage for circular geometry nano indenters. This applies to conical, cylindrical and spherical indenters. Figure 10 shows the flat "circular" end of a diamond cone indenter (types VP or YP), compared with the same indenter made of sapphire. If a non circular perimeter shape is acceptable, the diamond indenter can be made to a precise area specification in square microns. With diamond, it is possible to approach circular or spherical geometries but at higher cost.

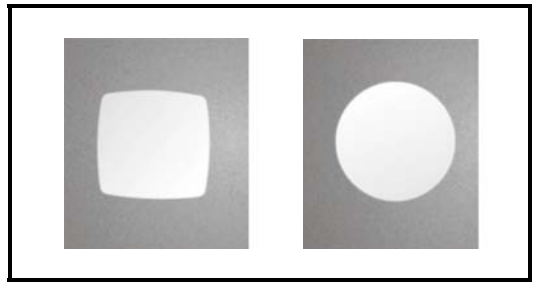


Figure 10 Diamond 4-sided Anisotropy - A diamond "circular" flat end becomes almost a square (left). Sapphire can approach a perfect circle.

Micro Star has **electrically conductive diamond** available. The conductivity comes from Boron ions dispersed through the bulk of the material not just from surface coating. Conductivity is required in some special applications and to prevent static charge distortion of measurements. The resistivity is 0.04 Ohm•m, similar to graphite.

Sapphire is the second material available for Micro Star nano indenters. Although not as hard as diamond, sapphire can be shaped to similarly sharp points and edges. Micro Star sapphire indenters are made with the crystal C axis aligned with the indenter axis. The anisotropy of sapphire is much weaker than diamond permitting nearly perfect spherical and circular shapes. **Figures 10 and 11** compare some diamond and sapphire properties.

Property	Diamond	Sapphire	Units
Hardness	10	9	Mohs-scale
Density	3.51	3.96	g/cc
Young Modulus E	1050	345	GPa
Thermal conductivity	2050	46.0	W/(m • K)
Thermal expansion	0.8	5.8	10^{-6}/°C
Electric resistivity	Insulator 0.04[11]	Insulator	Ohm • m

Figure 11 Some Diamond and Sapphire Properties

Most **other hard materials** can be used for making nano indenters, such are quartz, silicone, tungsten, steel, tungsten carbide and almost any other hard metal or ceramic.

INDENTER HOLDERS

Micro Star has indenter holders available made to the specifications of some current nano indenting instrument manufacturers. Micro Star can also make holders for existing instruments, or design and make new ones based on customer requirements.

Figure 12 shows 3 general holder design examples. The reference cylindrical and flat surfaces define the central axis and are the means of aligning the indenter with the axis of the instrument. Holders A and B are fastened with threads while C relies on a set screw in the instrument. Holder C has an orienting tab which aligns the indenter rotational angle with the instrument. Rotational orientation is not applicable for the majority of indentation measurements.

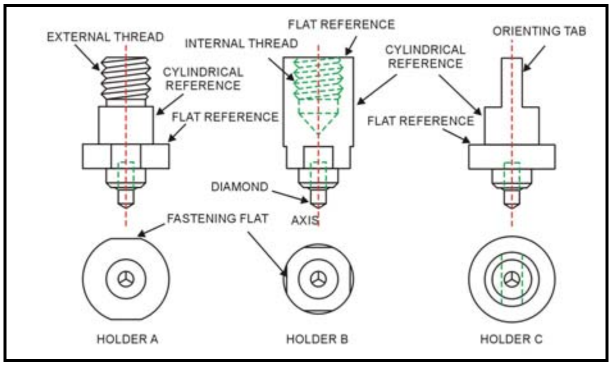


Figure 12 Indenter Holders Design Features

Holders are made of steel, titanium, machinable ceramic, other metals or rigid materials. Micro Star attaches the indenters to the holder using a proprietary **metal bonding** process. The metal forms a molecular bond with both materials be it diamond-steel, diamond-ceramic, sapphire-steel, sapphire-diamond, etc. The bonding metal is rigid up to 500°C and has a tensile strength of 400 MPa. In some cases diamond can be pressed into a metal holder if, for instance, is to be used at higher temperatures.

HANDLING AND CLEANING

Micro Star nano indenters are made to probe materials properties at nanometer scale. It follows that a basic requirement is the total absence of debris and particle contamination which would affect the indenter's performance in two ways.

a) Distorting the measurements because contaminating material adds an unknown factor negating the precise geometry of the clean indenter.

b) Damaging the indenter as debris particles are crushed between the indenter and the sample, producing lateral forces which the nanometer size tip is not designed to withstand.

Before shipment, Micro Star indenters are cleaned in vacuum and inspected at high magnification. The user needs to maintain the probe clean and undamaged in order to acquire accurate test results.

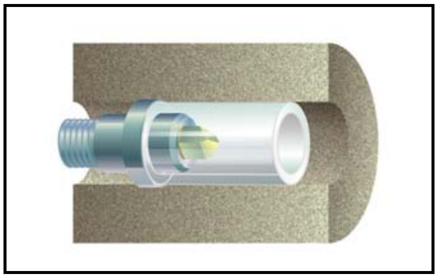


Figure 13 Indenter Protective Packaging

The indenters are supplied in special protective packaging, **Figure 13**. The tip is protected from contact with other objects by the tube as shown. This tube fits lightly on the cylindrical part of the holder. The tube may be used as a handle to attach the indenter to the instrument and then remove it with a straight motion avoiding to touch the tip.

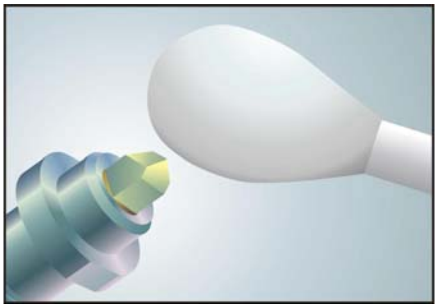


Figure 14 Indenter Cleaning with a Cotton Swab

If the diamond nano indenter is properly handled, it should not require cleaning. If it nevertheless becomes contaminated, clean as follows.

Looking through a stereo microscope, rub the diamond with a cotton swab soaked with isopropyl alcohol, **Figure 14**. Before the alcohol dries, blow the tip with pressurized air or other clean gas.

Inspect the diamond with an optical microscope at 400X. Repeat cleaning if necessary. Do not use the indenter unless the tip is totally clean and free of debris.

SPECIFICATIONS AND TOLERANCES

Micro Star indenters are measured with instruments calibrated with traceable standards. The angles are measured with a specially designed goniometer. Linear dimensions are measured with calibrated optical microscopes or electron microscopes. Micro Star has a specially modified TEM for sub micron measurements, as well as a standard SEM[12] and field emission SEM. An AFM are used for surface roughness measurements which are available on request for some indenters.

Each Micro Star indenter is measured individually for all pertinent dimensions. **Figure 16** shows the angles and nomenclature used on a 3-sided indenter. **Figure 17** is the specification sheet issued with each indenter. Sample data from a cube corner indenter is shown.

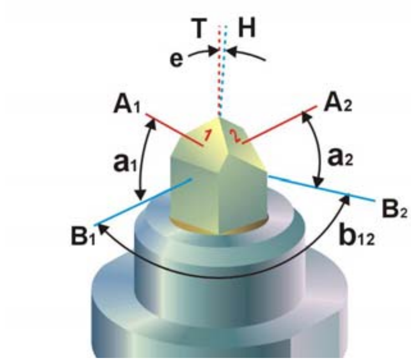


Figure 16 Angle Measurements on a 3-sided Indenter (for clarity, not all the angles are shown).

Figure 18 is a table of the symbols used with Micro Star indenter measurements and specifications with the ISO required and Micro Star standard tolerances. Smaller tolerances are available for some indenters at extra cost.

The tolerances of all specified angular or linear dimensions meet or exceed ISO 14577-2 which defines internationally accepted micro and nano indenter tolerances.

STANDARD TRACEABLE NANO INDENTERS

Micro Star can provide the **Berkovich, Modified Berkovich, Cube Corner, and Vickers** as standard traceable nano indenters following the definitions of ISO 14577-2[2].

These indenters are inspected and measured with equipment and standards traceable to the NIST or PTB. A **Calibration Certificate** accompanying these indenters covers the following items pertaining to the particular indenter:

a) Table of measurements.
b) Uncertainty of measurements.
c) Confidence level of measurements.
d) Measuring methods.
e) Measuring instruments.
f) Instrument calibration information.
g) Standards used.
h) Standards traceability.
i) Inspector’s signature and date.

NANO INDENTER

SPECIFICATIONS

SERIAL NUMBER	11035
INDENTER TYPE	TC
DESCRIPTION	CUBE CORNER
HOLDER TYPE	MS1

DIAMOND ✓ CONDUCTIVE DIAMOND
SAPPHIRE OTHER

Dimension		Nominal Value	Measured Value	Uncertainty	Units
Angle	a_1	35.26	35.28	±0.025	deg
Angle	a_2	35.26	35.26	±0.025	deg
Angle	a_3	35.26	35.30	±0.025	deg
Angle	a_4				deg
Angle	b_{12}	120.00	119.85	±0.025	deg
Angle	b_{13}	240.00	240.04	±0.025	deg
Angle	b_{14}				deg
Angle	c				deg
Angle	e*		0.15	±0.025	deg
Line	L				
Radius	R		< 50		nm
Diameter	D				
Area	A				
Indentation depth	h		>2		µ
Surface roughness	r*		<20		nm

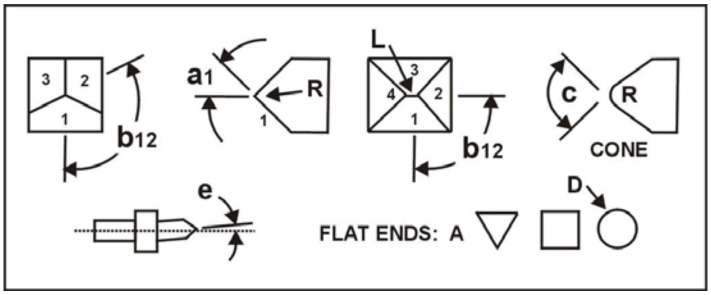


Figure 17 Indenter Specification Sheet with Example Data in Blue *Measured on Request at an Extra Cost

DIMENSIONAL NOMENCLATURE AND TOLERANCES			
SYMBOL	**DESCRIPTION**	**ISO REQUIRED TOLERANCE**	**MST STANDARD TOLERANCE**
1 (2, 3, 4)	Face number 1. Face 1 can be marked on request.		
A_1 (A_2, A_3, A_4)	Line perpendicular to face 1.		
B_1 (B_2, B_3, B_4)	Projection of line A_1 on a plane perpendicular to the Indenter axis H.		
H	Indenter holder axis. Final angle measurements and user results refer to this axis.		
T	Indenter crystal axis. Ideally should be the same as H but in practice may differ by angle e.		
e	Angle between T and H.		± 0.25°
a_1 (a_2, a_3, a_4)	Face 1 tilt angle with respect to H, equal to angle between A_1 and B_1.	± 0.3°	± 0.25°
b_{12} (b_{13}, b_{14})	Rotation angle between face 1 and face 2, equal to angle between B_1 and B_2.	± 0.3°	± 0.25°
c	Included angle on a cone.	± 5°	± 5°
L	Line of conjunction on 4-sided indenters.	< 500 nm	< 400 nm
L (wedge indenter)	Wedge length.		± 20% of L
R (sharp tip)	Tip radius.	< 200 nm	< 50 nm
R (spherical tip)	Radius.	± 25% of R	± 10% of R
D	Diameter of a circular flat tip or cylinder.		± 10% of D
A	Area of a flat tip.		± 20% of A
h (sharp tip)	Valid indentation depth.	> 200 nm	> 1000 nm
h (cone with spherical tip)	Valid indentation depth.	>15 % of R (c = 90°) >35 % of R (c = 40°)	>15 % of R (c = 90°) >35 % of R (c = 40°)
h (spherical indenter or cylinder)	Valid indentation depth.		>35 % of R
r	Surface roughness.		<30nm

Figure 18 Symbols in Figures 16 and 17 (ISO required tolerances and Micro Star standard tolerances are shown where applicable)

[1] ISO/IEC 17025 = International standard comprising general requirements for the competence of testing and calibration laboratories.

[2] ISO 14577-2 = Instrumented indentation test for hardness and materials parameters. Part 2: Verification and calibration of testing machines. In particular section 4: Direct verification and calibration.

[3] NIST = National Institute of Standard and Technology.

[4] PTB = Physikalisch-Technische Bundesanstalt (the counterpart of NIST in Germany).

[5] TEM = Transmission Electron Microscope.

[6] Berkovich, E. S., *Three faceted diamond pyramid for micro hardness testing* Industrial Diamond Review, 11, #127, June 1951.

[7] Oliver, W.C. and Pharr, G.M., An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. J Mater. Res.,7, #6, June 1992, pp 1564-1583.

[8] Anisotropy = The change of crystal properties with atomic directions. Anisotropy is especially pronounced in diamond with different abrasion resistance depending on the crystal orientation.

[9] AFM = Atomic Force Microscope

[10] Measured with a calibrated AFM.

[11] Conductive diamond available from Micro Star.

[12] SEM = Scanning Electron Microscope