C227-E024D

SHIMADZU
Excellence in Science

Dynamic Ultra Micro Hardness Testers

DUH-211/211S

System for Evaluating Thin Films,
Surface-Treated Layers, and Microelectronic Parts

Dynamic Ultra Micro Hardness Testers

DUH-211/211S

Building on our experience with hardness evaluation technology for the micro range, we have taken our quest for greater precision and ease of use to the next level. Our hardness tester can measure the strength properties of material surfaces and microscopic materials using new evaluation methods specified in ISO standards.

Perform evaluation using the hardness and materials parameters specified in ISO 14577-1 (Annex A) *1).

It is possible to evaluate various plastics under the same conditions and on the same scale using ISO/TS 19278 *2).

Evaluates hardness of a wide range of materials

Thin films
Plastics
Rubbers and elastomers
Metallic materials
Fibers
Brittle materials
Microscopic electronic components

Test the surface strength of thin films, surface-treated layers such as ion-implanted layers and nitride layers, as well as nonmetallic materials such as plastics, rubbers, and ceramics.

*1 ISO 14577-1 *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*
Annex A *Materials parameters determined from the force/indentation depth date set*
Standard used for new evaluation methods that continuously measure changes in test force and indentation depth which occur when an indenter is pressed into a material, and for the evaluation of material hardness and strength properties such as Young's modulus and creep deformation

*2 ISO/TS 19278 *Plastics — Instrumented micro-indentation test for hardness measurement*
A standard for evaluating the hardness of various plastics on the same scale using instrumented indentation hardness so that the hardness of various plastics can be compared.

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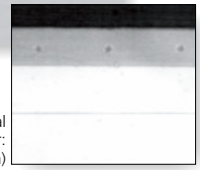
Materials and Applications

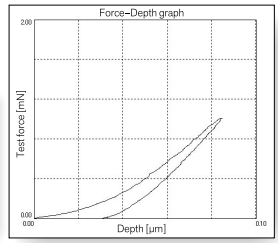
Thin Films Such as Vapor-Deposited Films and Semiconductor Materials, and Surface-Treated Layers Such as Ion-Implanted Layers and Nitride Layers

As film production technology improves and diversifies, it is becoming increasingly important to evaluate the hardness of thin films and coatings on the surface of materials. These include ion-implanted layers, DLC (diamond-like carbon) films, vapor-deposited films produced by CVD (chemical vapor deposition), PVD (physical vapor deposition) and alumite layer. By using an ultra micro test force to measure depths of less than one tenth of a film's thickness, the DUH tester makes it easy to evaluate the hardness of only the film, without influence from underlying materials.

SiO_2 (1 μm)
Test force: 1 mN

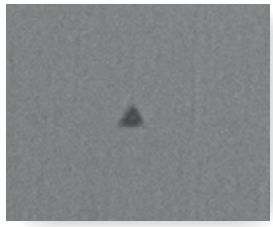


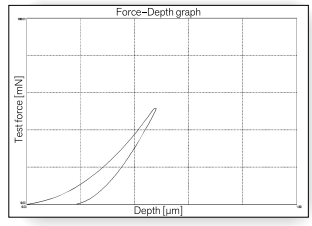
Plating layer on metal plate (Plating layer: Approx. 10 μm)



DLC Films

DLC films offer properties such as high hardness, low friction coefficients, wear resistance, electrical insulation, chemical resistance, and infrared light permeability, and are widely used in tools, automotive parts, semiconductor manufacturing equipment parts, and household goods. Measuring the hardness of such films is necessary for determining the optimal film manufacturing parameters and for monitoring their quality, but creating large indentations is not possible. The DUH is perfect for these types of application, because it can evaluate hardness based on the indentation depth using only a small test force.



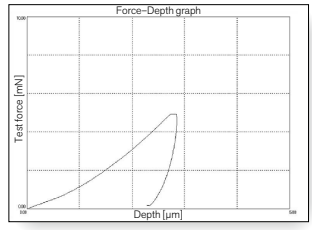


DLC Films Test force: 50 mN

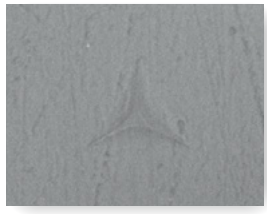
Plastics

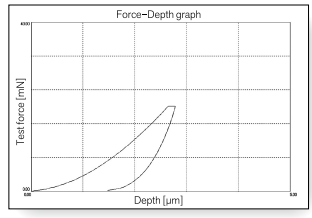
An important feature of engineering plastics is hardness. The DUH can measure the hardness of even highly light-absorbent materials, which are difficult to measure using conventional testers.

Engineering plastics offer high strength, heat resistance, and other properties while still providing the advantages of plastics in general such as superior plasticity and ease of processing. As a result, they are commonly used for internal mechanical parts (such as gears and bearings) in consumer electronic products. They provide higher wear resistance, lighter weight, and lower cost than metal parts, and can be mass produced, where hardness is used to improve performance and control quality. However, the low reflectivity of plastics makes it difficult to measure the size of indentations when using conventional hardness testers. In contrast, the DUH is perfect for these applications, because it evaluates hardness based on the test force applied and the resulting indentation depth.

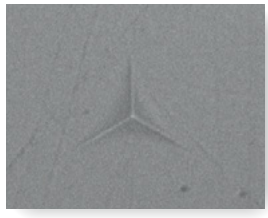


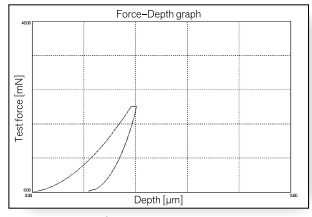
Thin film (30 μm) Test force: 5 mN





Nylon Test force: 20 mN





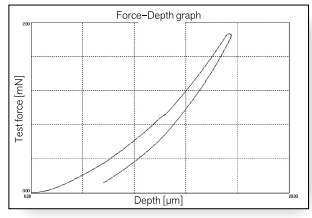
Acrylic Test force: 20 mN

Rubbers and Elastomers

Because indentation depth is used to determine hardness, hardness can be measured using a variety of test forces and the tester can even be used to evaluate deterioration in material surfaces of materials.

Rubber provides a low elastic modulus, high elongation in response to small forces, and high repulsion. Consequently, its raw materials are often mixed with various chemical ingredients to make a variety of products, such as tires, vibration absorbing rubber, and O-rings. Due to the harsh environments where such products are typically used, in addition to evaluating durability, hardness is used to also evaluate surface deterioration. However, conventional hardness testers cannot evaluate the elastic characteristics of rubber because of the indentation after testing. In contrast, the DUH measures both the test force and indentation depth, which makes it the perfect method for evaluating rubber, including its elasticity.

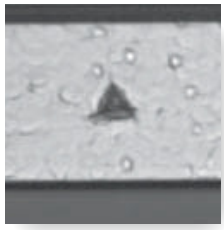
Due to elastic recovery, no indentation remains

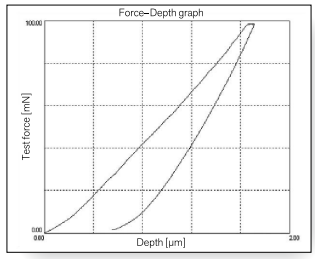


Silicone rubber Test force: 2 mN

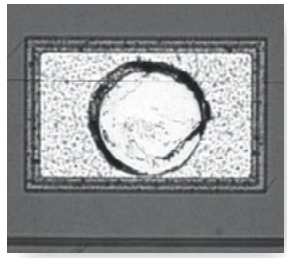
Metallic Materials

Perform micro-region hardness measurement, which has become increasingly difficult as feature sizes have become ever smaller.





Semiconductor pattern Test force: 100 mN

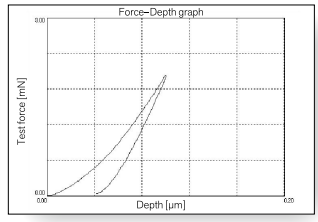


Gold stud bump

Brittle Materials Such as Glass and Ceramics

Use a small test force to evaluate the hardness of brittle materials without generating cracks. Measure the test force required to generate cracks.

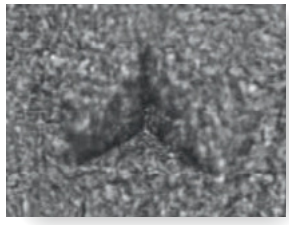
Because glass is clear, hard, highly resistant to thermal deformation, and a good electrical insulator, it is used for a wide range of applications, from window glazing and display screens to various substrate materials, such as for CDs. On the other hand, glass also tends to be brittle and thus requires various material and processing method modifications for it to be used in, for example, large thin display applications. Hardness is used to evaluate glass, but large test forces cause cracks and the indentations are not clearly visible. Therefore, the DUH is ideal for evaluating glass, because it determines hardness based on the indentation depth using a small test force.

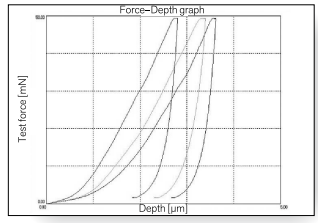


Optical glass Test force: 2 mN

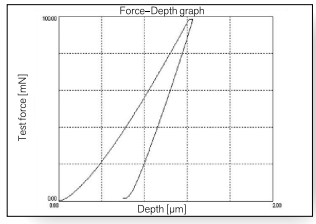
Ultra-Fine Fibers Such as Optical Fibers and Carbon Fibers

Evaluate the strength of specimens taken from composite fiber materials and obtain important information. Measure the hardness of fibers.





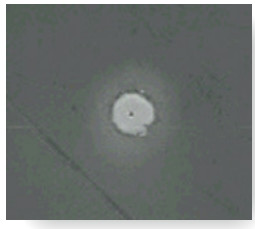
Carbon fiber Test force: 50 mN



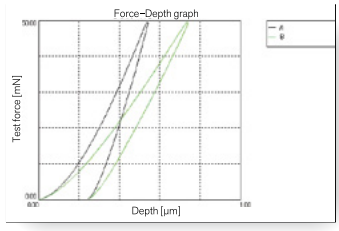
Metal wire Test force: 100 mN

Micro Powders

Advances are being made to create ever finer micro powders in an effort to increase their surface area-to-volume ratios. The strength of powders is evaluated using compression testing, but due to the size of the particles, the DUH is ideal for measuring their hardness in more detail.



Zirconia particles (30 µm)



Example of measuring zirconia particles
Test force: 50 mN

Features

1. Evaluation of Hardness and Material Parameters in Accordance with Standards (ISO 14577-1 Annex A)

Measure the behavior of a specimen as an indenter is pressed into it and evaluate the hardness, elastic modulus, and amount of work done during indentation, in compliance with and ISO 14577-1 (instrumented indentation test for hardness) Annex A.

2. Evaluation of ISO/TS 19278 for Plastics

Measure various plastics under the same measurement conditions in accordance with ISO/TS 19278. (DUH-210/210S)

3. Highly Precise Evaluation of Elastic Modulus

Perform highly precise evaluation of the elastic modulus, using correction based on instrument rigidity and the shape of the intender tip*1.

4. Low Test Force with Measurement Resolution of 0.196 μN

Control the test force using a high resolution of 0.196 μN. This allows measurement of material strength properties in micro regions and in the outermost surfaces of specimens.

5. Ultra-Wide Test Force Range of 0.1 to 1,961 mN

Use a wide test range of 0.1 to 1,961 mN for measurement, and test a variety of industrial materials, including rubber, plastics, and ceramics.

6. High-Precision Measurement of Indentation Depth

No need to measure the actual indentation.
Specimen indentation depth can be measured in units of 0.0001 μm for depths up to 10 μm.

7. Supports a Wide Range of Testing Methods

Record the relationship between the test force and the indentation depth. Test both the unload and load processes. Use the DUH-211S to perform cyclic load-unload tests and step load-unload tests.

8. Supports Vickers Hardness Test

A function to measure the length of diagonals is provided as a standard feature. This function allows you to measure the hardness that corresponds only to plastic deformation, Vickers hardness, and Knoop hardness. (A Vickers indenter and Knoop indenter are available as options.) Maximum microscope magnification is 500× (1000× is available as an option).

*1 Indenter tip shape correction is only available for the 115-degree triangular pyramid indenter. Shape correction is not available for other indenters.

Measurement Principle

Electromagnetic force is used to press an indenter (standard type: 115° triangular pyramid) against a specimen. Pressing force is increased at a constant rate, from 0 to the preset test force. Indentation depth is automatically measured as the indenter is pressed against the specimen. This allows dynamic measurement of changes that occur in the specimen's resistance to deformation during the indentation process, and obtains a wide variety of data. During indentation the DUH-211/211S measures dynamic hardness and evaluates the hardness that corresponds to both plastic and elastic deformation. Also, if the indentation size is large enough to be observed with a microscope, hardness can be calculated using just the plastic deformation, by measuring the diagonal length of the indentation.

Expressions for Dynamic Hardness

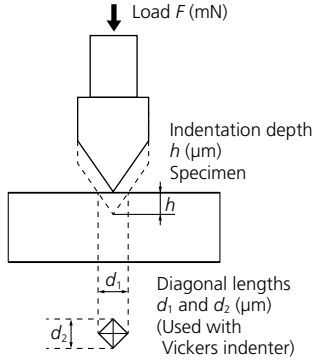
(1) 115° triangular pyramid indenter (standard)
$DHT_{115} = 3.8584 \times F / h^2$

(2) 100° triangular pyramid indenter (option)
$DHT_{100} = 15.018 \times F / h^2$

(3) Vickers indenter (option)
$DHV = 3.8584 \times F / h^2$

(4) Knoop indenter (option)
$DHK = 1.5583 \times F / h^2$

Even though the theoretical unit for these hardness expressions is kgf/mm², it is normally not used.



Expressions for Martens Hardness (ISO 14577-1 Annex A)

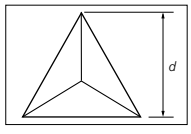
(1) 115° triangular pyramid indenter (standard)
$HM_{115} = 1000\ F / 26.43 \times h^2$ [N/mm²]

(2) Vickers indenter (option)
$HMV = 1000\ F / 26.43 \times h^2$ [N/mm²]

Hardness Expressions Based on Diagonal Length

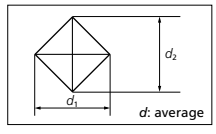
(1) 115° triangular pyramid indenter (standard)
$HT_{115} = 160.07 \times F / d^2$

(2) 100° triangular pyramid indenter (option)
$HT_{100} = 121.53 \times F / d^2$



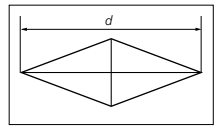
(3) Vickers indenter (option)

$HV = 189.10 \times F / d^2$



(4) Knoop indenter (option)

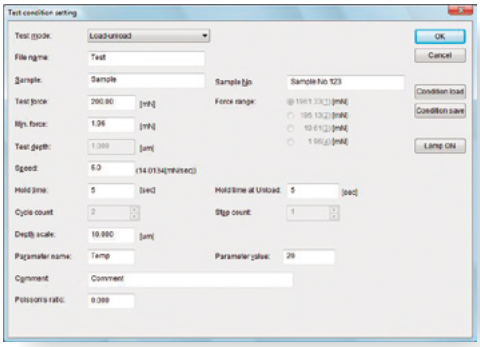
$HK = 1451.1 \times F / d^2$



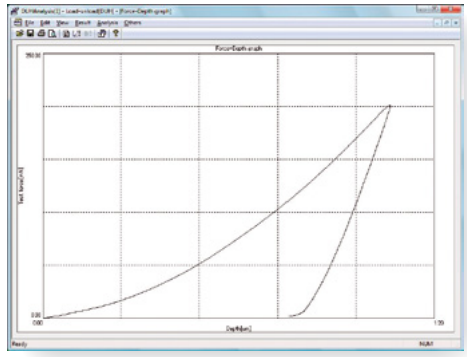
Functions

Combines Easy Operability and High-Level Data Processing Functions

Model used to perform three basic tests: **DUH-211**
Advanced model provides seven test modes: **DUH-211S**



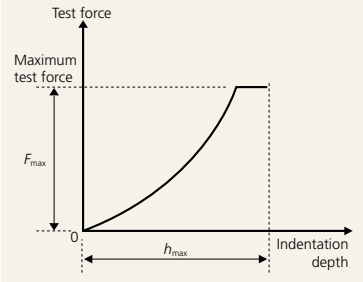
Parameters required for each type of test can be viewed at a glance.



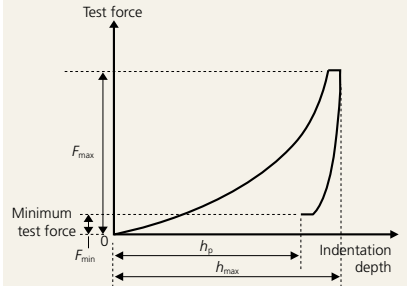
Test Types

Item	DUH-211	DUH-211S
1. Load–Hold Test	✓	✓
2. Load–Unload Test	✓	✓
3. Cyclic Test	✓	✓
4. Depth Setting Test	—	✓
5. Depth Setting Load–Unload Test	—	✓
6. Step Load Test	—	✓
7. Step Load–Unload Test	—	✓

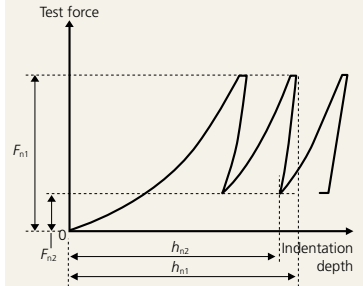
Indentation size can be measured in tests 1, 2, 4, and 5.



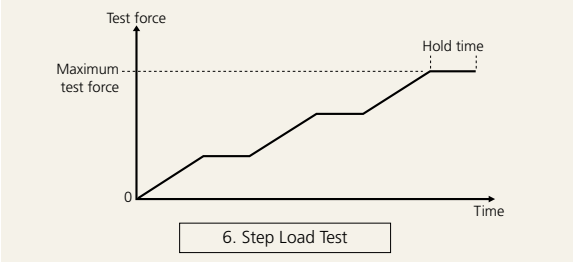
1. Load–Hold Test
4. Depth Setting Test



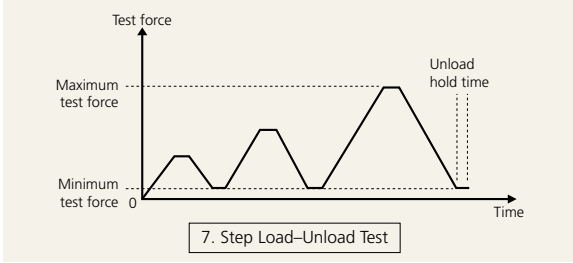
2. Load–Unload Test
5. Depth Setting Load–Unload Test



3. Cyclic Test



6. Step Load Test



7. Step Load–Unload Test

ISO 14577-1 (Annex A) Compliant Evaluation

(Instrumented Indentation Test for Hardness)

Relationship between test force and indentation depth during indentation process can, in accordance with ISO 14577-1 (Annex A), be used to evaluate hardness, elastic modulus, and amount of work done.

HM : Martens hardness
HM_s : Martens hardness obtained from gradient of graph of test force versus depth
H_{it} : Indentation hardness
E_{it} : Indentation elastic modulus
C_{it} : Indentation creep
η_{it} : Indentation work rate
HV* : Vickers hardness obtained by converting H_{it}

1. Indentation Elastic Modulus (E_{it})

Definition of indentation elastic modulus (E_{it}) states that E_{it} is obtained from the inclination of the tangent used to calculate the indentation hardness (H_{it}), and is equivalent to Young's modulus.

$$\frac{1}{E_r} = \frac{1-\nu_s^2}{E_{it}} + \frac{1-\nu_i^2}{E_i}$$

$$S = dP/dh = 2 \cdot E_r \cdot A_p^{0.5}/\pi^{0.5}$$

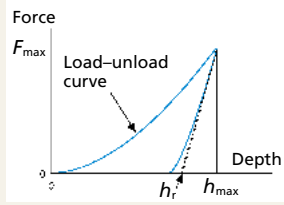
$$A_p = 23.96 \cdot h_c^2$$

$$h_c = h_{max} - 0.75(h_{max} - h_r)$$

If Poisson's ratio for the specimen is set in the test parameters, the DUH-211/211S calculates E_{it}. Otherwise, the DUH-211/211S calculates $(1-\nu_s^2)/E_{it}$.

Here,

E_r: Converted elastic modulus based on indentation contact
E_i: Young's modulus for indenter (1.14×10^{12} N/m^2)
ν_i: Poisson's ratio for indenter (0.07)
E_{it}: ndentation elastic modulus
ν_s: Poisson's ratio for specimen
S: Inclination at start of unloading (inclination of straight-line approximation)
A_p: Projected contact area (23.96 is a constant that applies when using a 115° triangular pyramid indenter.)
h_c: Depth of the contact of the indenter with the test piece at F_{max}
h_r: Point of intersection of the tangent to curve b at F_{max} with the indentation depth-axis

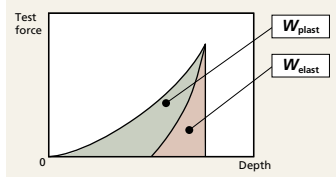


2. Plastic and Elastic Portions of Indentation Work (η_{it})

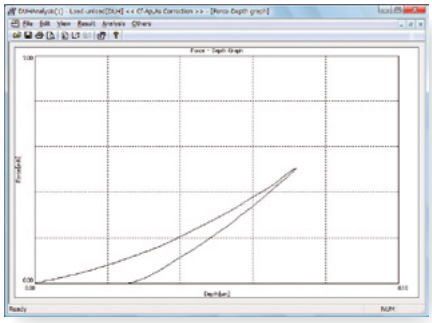
A portion of the total mechanical work performed by indentation, W_{total}, is consumed due to plastic deformation, W_{plast}. The remaining portion of the total mechanical work corresponds to elastic deformation, W_{elast}, which is released when the test force is unloaded. This work is defined by $W = \int F dh$.

$$\eta_{it} = \frac{W_{elast}}{W_{total}} (\%)$$

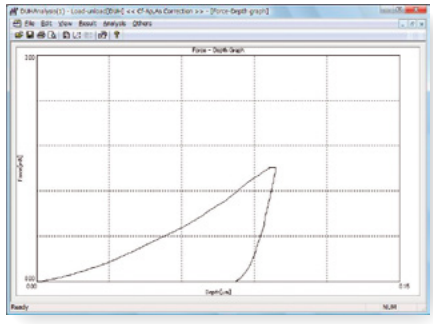
$$W_{total} = W_{elast} + W_{plast}$$



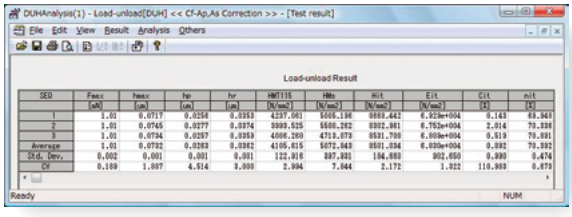
Test Examples

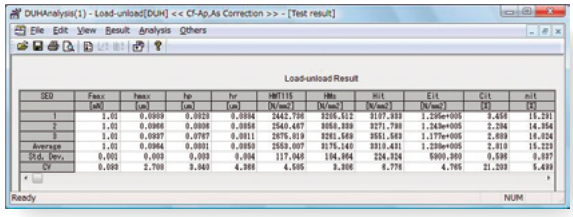


Specimen: Fused silica
Test force: 1 mN



Specimen: Copper alloy
Test force: 1 mN

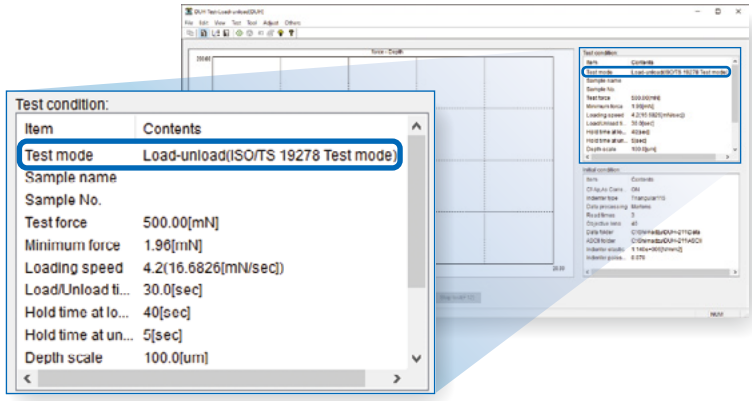




ISO/TS 19278 Compliant Evaluation

Unlike the conventional hardness test standards for plastics, it is possible to compare the hardness of various plastics from rigid plastics to semi-rigid plastics under the same conditions and at the same scale. The software provides a dedicated mode for easy testing.

Test force:	500 mN
Load/Unload time:	30 s each
Holding time:	40 s
Number of tests:	5 or more
Hardness used for evaluation:	H_{IT}

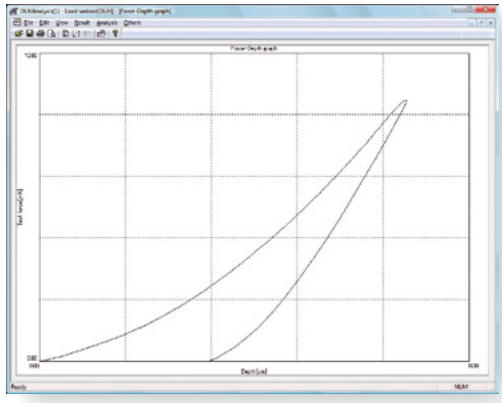


Data Processing

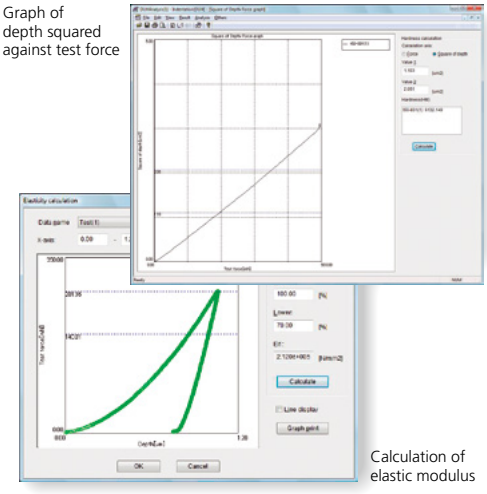
Simply set the required items to obtain the desired information.

Data Processing Items

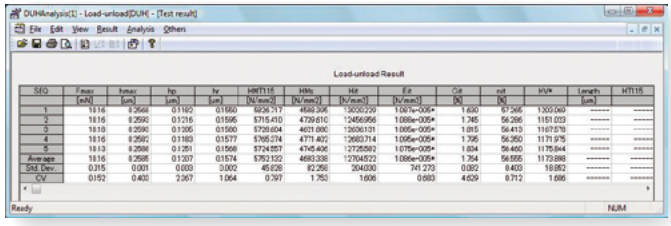
- Results display
- Data output for test force and depth
- Graph output for test force and depth
- Graph output for hardness and depth
- Graph output for hardness between 2 points and depth
- Graph output for depth and time
- Graph output for hardness and test force
- Graph output for depth squared and test force
- Hardness calculation based on preliminary test force
- Graph output for hardness and parameters
- Calculation of converted hardness values
- Repeated changes of surface detection points
- Calculation of elastic modulus
- ASCII file output



Graph of depth squared against test force



Calculation of elastic modulus



Example of test results display (load–unload test)

Specifications

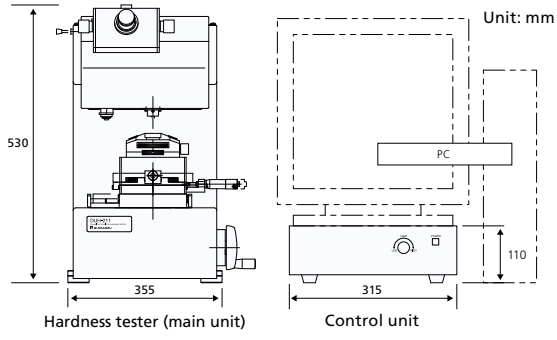
Model		DUH-211	DUH-211S
P/N		344-04250-XX (Windows 10)	344-04251-XX (Windows 10)
Loading Unit	Loading Method	Electromagnetic coil	
	Test Force Range	Full scale of 0.1 to 1,961 mN	
	Test Force Accuracy	±19.6 μN or ±1% of displayed test force, whichever is greater	
	Minimum Measurement Increment	0.196 μN (for a test force not exceeding 1.96 mN)	
Displacement Measurement Unit	Measurement Method	Differential transformer	
	Measurement Range	0 to 10 μm	
	Minimum Measurement Increment	0.0001 μm	
	Linearity	±2% of full scale (20 μm)	
Indenter	Type	Triangular pyramid indenter with tip angle of 115° (Vickers indenter and Knoop indenter are available as options.)	
	Tip Radius	0.1 μm max.	
Optical Monitor	Total Magnification (microscope)	×500	
	Objective Lens	×50 (Up to 2 lenses can be attached.)	
	Eyepiece	×10	
	Lighting Method	Reflected illumination	
	Light Source (lamp)	LED: 3 W, 3 V	
	Light-Path Switching	Observation or photograph (selectable)	
Micrometer	Collimation Method	Direct connection between encoder and control handle; synchronized movement of two indexes	
	Detector	Optical encoder	
	Effective Measurement Range	200 μm (with ×50 objective lens)	
	Minimum Measurement Increment	0.01 μm/pulse	
Specimen Stage	Vertical Distance	Approx. 60 mm	
	Area	Approx. 125 (W) × 125 (L) mm	
	Stage Movement Range	25 mm in both X and Y directions	
	Specimen Holder	Specimen dimensions (i.e., 8 (thickness) × 30 (width) mm) when thin-type attachment (type 3) is used	
Test Modes	Load–Hold Test	✓	✓
	Load–Unload Test	✓	✓
	Cyclic Test	✓	✓
	Depth Setting Test	—	✓
	Depth Setting Load–Unload Test	—	✓
	Step Load Test	—	✓
	Step Load–Unload Test	—	✓
Required PC Specifications	OS	Windows® 10 Pro (64 bit edition)	
	Disk Drives	CD-ROM drive	
	Expansion Bus	PCI Express ×1 (full-length), 2 slots min. more than one ×1 slot is reguired (or slot size ×2 or more)	
Utilities	Power Supply	Single phase, AC 100–115 V ± 10%, AC 230 V ± 10% (Ground resistance 100 Ω max.)	
	Power Consumption	Approx. 100 W (not including power consumption of PC)	
	Grounding	The ground terminal on 3-prong connectors must be properly grounded with grounding resistance at 100 Ω or less.	
	Temperature	Recommended temperature: 23±1°C Allowable range: 10°C to 35°C	
	Vibration	Horizontal vibration: 0.017 Gal max. (at 10 Hz or more), 0.01 μm max. (at less than 10 Hz) Vertical vibration: 0.010 Gal max. (at 10 Hz or more), 0.005 μm max. (at less than 10 Hz)	
	Humidity	80% max. (no condensation)	
Dimensions and Weight	External Dimensions	Tester: Approx. 355 (W) × 405 (D) × 530 (H) mm Control unit: Approx. 315 (W) × 375 (D) × 110 (H) mm	
	Weight	Tester: Approx. 60 kg Control unit: Approx. 5 kg	

Standard Configuration

Name		Quantity
Hardness Tester (main unit)		1
Objective Lens (×50)	P/N 344-89964-40	1
Triangular Pyramid Indenter (tip angle: 115°)	P/N 340-47013	1
Specimen Stage (XY stage)	P/N 347-24205-41	1
Micrometer Head		2
Specimen Holder	P/N 344-17737-40	1
Control Unit		1
Accessories (Cords, AC adapters, tools, instruction manual, installation disk)		1 set

PC and Printer are not included.

External Dimensions

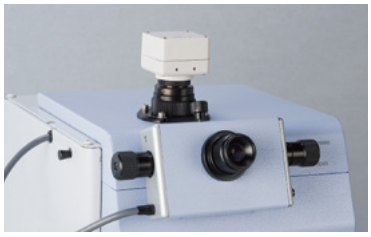


Optional Accessories

Length Measurement Kit (Color or Monochrome)

Length measurement kit, color:
P/N 347-24778-44
Length measurement kit, monochrome:
P/N 347-24778-43

Microscope images of the specimen surface can be displayed on the PC screen. Measure the size of indentations on the screen and save the images. Maximum magnification factor is ×2400 (when using a 17″ monitor and an objective lens with a magnification factor of 50). This accessory can be used with computers designated by Shimadzu.



Objective Lens

×100 objective lens: P/N 344-89977-40
×40 objective lens: P/N 347-25400
×20 objective lens: P/N 344-89924-40
×10 objective lens: P/N 344-89941-40
×40 objective lens with ultra-long operating distance: P/N 344-89300-41

40× objective lens with ultra-long operating distance. Improves contrast in the field-of-view.

Windbreak

P/N 347-24400-01

This case minimizes the influence of air disturbances, such as due to DUH tester exposure to air flow or sound.
W700 × D650 × H750 (mm)

Windbreak (Large type)

P/N 347-24400-02

In the case that Active Vibration-Absorbing Bench is used this is select.
W700 × D650 × H950 (mm)

Vickers Hardness Standard Block

P/N 340-06619-07

Used for measuring hardness with the 700HMV micro Vickers. Used as a rough guide for Vickers hardness measurement.

BK7 (Glass Test Piece)

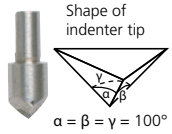
P/N 339-89207-14

Used to obtain the correction factors required for the indenter when measuring the elastic modulus.

Triangular Pyramid Indenter with 100° Tip Angle

P/N 340-47011

This indenter, with a tip angle of 100°, has a smaller tip radius and makes smaller indentations than an indenter with a tip angle of 115°. Used for testing small-size specimens.



Measurement Kit for Vickers Hardness

P/N 347-24449-01

(Contents: Vickers Indenter 1 pc, Inspection Report 1 pc)
The verification in accordance with standard (ISO 6507-2) is done at the factory.
Factory verified for compliance with Vickers hardness test standards.
Please order simultaneously with the DUH.

Measurement Kit for Knoop Hardness

P/N 347-24449-11

(Contents: Knoop Indenter 1 pc, Inspection Report 1 pc)
The verification in accordance with standard (ISO 4545-2) is done at the factory. Factory verified for compliance with Vickers hardness test standards.
Please order simultaneously with the DUH.

Desk-Type Vibration Absorbing Bench

P/N 344-04193-06

This bench with desk-type coil springs is recommended if the DUH-211/211S tester is used in areas that are subject to strong vibrations.

Active Vibration-Absorbing Bench

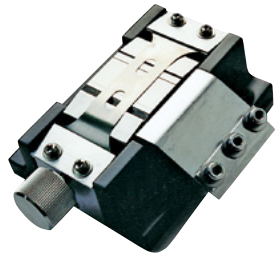
P/N 344-04211-11: AC 120 V
P/N 344-04211-12: AC 230 V

This bench is used together with a special mount and performs active vibration absorption over a wide range, from 0.7 Hz to 100 Hz.

Slender Specimen Holder

P/N 344-82943-40

This attachment is used to firmly hold thin specimens with an outer diameter of 0.15 mm to 1.6 mm, such as sewing machine needles, watch shafts, thin-shaped medical equipment, wires, sintered wires, and nonferrous wires.



Disk-Type Vacuum Suction Unit

P/N 344-86201-42

Used for 5″, 6″, and 8″ wafers. (Air supply for suction must be separately prepared.)

Micrometer Head (Digital Display)

P/N 347-25447-12 (2 unit)

Used to digitally display the amount of stage movement (up to a maximum of 25 mm) in the front/back or left/right directions in 1 µm increments. (Photo shows this head attached to a stage.)

Rotation Stage

P/N 344-82857-01

This stage has a diameter of 125 mm and can rotate in the range ±5°.

Objective Micrometer

P/N 046-60201-02

Used to adjust the microscope's magnification factor. Marked with scale graduations at 10 µm intervals.

Installation Precautions

Consider the following points when deciding on the installation location of the tester.

1. To minimize vibration:

1) Install the tester in a location where floor vibration is minimal. Normally, place the tester on a vibration-absorbing bench.
2) Do not install the tester in a location where people requently walk by.
3) Do not install the tester near equipment that generates vibrations.
4) If possible, install the tester on the first floor of a building.
5) Install the tester as far away as possible from streets, roads and railway tracks.
6) Do not perform testing if vibration-generating equipment (e.g., a crane) is being used nearby.

2. To minimize air drafts and sounds:

1) Do not install the tester in locations that are directly or indirectly subject to streams of air from air-conditioning equipment.
2) Use a windbreak during testing.
3) Do not open or close nearby doors during testing.
4) Do not install the tester near sound-generating equipment (e.g., telephones).

3. To ensure testing accuracy:

Be especially careful when performing the following types of tests:

- Tests involving test forces of 1 mN or less
- Tests involving the measurement of changes for indentation depths of 0.05 µm or less

In these cases, be sure to maintain the following conditions:

- Temperature: No fluctuations greater than ±1°C.
- Vibration: Refer to specification table.

Example of Dynamic Ultra Micro Hardness Tester DUH-211/211S Systems

Electric X–Y Stage System

P/N 347-24625-41

- Stroke: X-axis, Y-axis ±25 mm (There is the function that the slides 50 mm toward the X-axis. All stroke of X-axis are 100 mm)
- Resolution: 0.001 mm
- Drive method: Ball screw actuated by stepping motor

* Compatible to electric Z system.

Related Products

Micro Compression Testing Machine
MCT Series

This machine is used to measure the compressive strength of single particles (of diameter 1 µm or greater). The compressive strength of ceramics, plastics, pigments, food products, and pharmaceuticals can be measured at a particulate matter stage, providing data that is closely related to the final application of these substances.

- Loading Method: Electromagnetic force 9.807 mN to 1.961 N or 9.807 mN to 4.903 N
- Indenter: Diamond, cone-shaped, 50-µmø diameter
- Displacement Measurement: Differential transformer 0 µm to 10 µm or 0 µm to 100 µm
- Optical Monitor: Equipped with ×500 microscope

Micro Hardness Tester
HMV-G31 Series

This type of hardness tester automatically measures length using a built-in CCD camera. The automatic measurement function provides easy and worry-free measurements without human error. The innovative G-frame provides a broad work area, dramatically improves operability, and easily accommodates long samples or other samples with a large area.

The automatic lens switching function automatically switches to the magnification appropriate for the given indentation size, which means anyone can operate the system accurately.

- Test Force Range: 98.07 mN to 19.61 N (Optionally from 9.807 mN)
- Indentation Measurement Time: Approx. 0.3 sec.
- Equipped with revolving electric turret (HMV-G31T)

Fully Automatic Micro Hardness Tester
HMV-G31-FA Series

By including an automatic measurement function, an electric XYZ stage function, and an auto-focus function to a micro Vickers hardness tester used for evaluating the hardness of paint or plating coatings or surface-hardened layers, this automatic hardness tester is able to perform a continuous series of highly precise measurements automatically.

- Test Force Range: 98.07 mN to 19.61 N (Optionally from 9.807 mN)
- Electric XY Stage: Stroke: ±25 mm Resolution: 0.001 mm
- Electric Z Stage: Stroke: 40 mm Resolution: 0.001 mm

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