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Document History

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Review and Approval

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1. Purpose

This procedure describes a standard method to realize an indentation hardness test on a material sample using the Mach-1™ mechanical tester.

2. Scope

This procedure can be applied on any type of material with shape and dimension compatible with the specifications presented in the material section while the expected hardness results remain within tester specifications. It is highly recommended to use standard protocol for sample preparation. Some parameters must be specified in an associated protocol/report: test type, magnitude of force, velocity rate of indenter, sample geometry, sample preparation, vertical stage resolution, filename, test parameters, ambient conditions.

3. Background

An indentation hardness test is a conventional method of measuring a solid material's resistance to permanent damage after undergoing a compressive force. Using a very hard pointed indenter with known geometry, a sample may be compressed and the resulting impression measured. The resulting indentation geometry can be directly related to the hardness of the sample through simple calculations.

This hardness value can be measured on samples which underwent varying conditions to gain insight into the influence of such conditions. The test can also be used to validate the properties of a material of known origin and composition. This value can be correlated to tensile strength and is an indicator of wear resistance, toughness and ductility. Different types of hardness tests, however, are not comparable because of the different geometries of the indenter tips. Thus, one test should be chosen for a set of samples to gain a conclusion.

In the case of the Mach-1, the available load cells do not reach a range high enough to perform Rockwell or Brinell hardness tests. This operating procedure will focus on the methods of Vickers, modified-Berkovich and Knoop hardness testing. Some particular indenter geometries are more appropriate for certain materials. It is thus recommended to search the literature to select the best indenter for particular application. Low load testing (under 10N) is known as "microindentation hardness" testing, while higher load testing (over 10N) is refer to as "macroindentation hardness" testing.

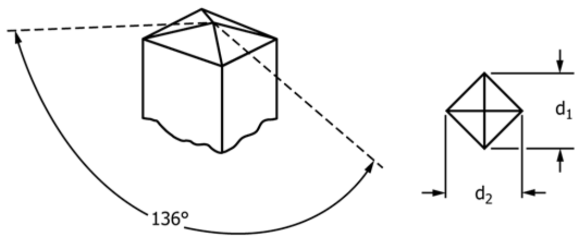

Figure 1. Vickers Indenter (ASTM C1327-15, 2015)

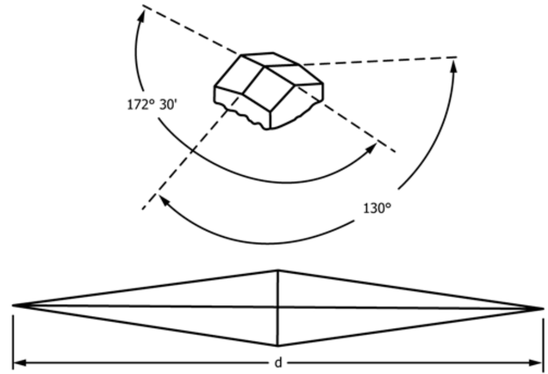

Figure 2. Knoop Indenter (ASTM E384-16, 2016)

In the case of a Vickers hardness test, a square-based pyramidal diamond tip indenter is used to transfer a constant force for a specified dwell time. The resulting indentation diagonals are measured to obtain the Vickers hardness number (HV). The modified-Berkovich is a 3-sided triangular based pyramid with the same projected area as Vickers. For a Knoop hardness test, a rhombus-based pyramidal diamond tip is used in the same manner. The resulting long indentation diagonal is measured to obtain the Knoop hardness number (HK).

General equation for these methods:

$$Hardness\ value = \frac{Test\ Load}{Area\ of\ impression}$$

Testing Considerations: It is important that the resulting indentation is symmetric, measurable and repeatable. The perpendicularity of the sample surface to the indenter as well as the surface finish of the sample are important factors in obtaining valid hardness results. The presence of large cracks, asymmetry and coincidence with pores are factors that could invalidate a result. Thus, multiple tests must be evaluated to obtain a statistically relevant result.

Large enough indent diagonals are desired for high measurement precision (>20 µm). Since the applied force is directly related to the size of the indent, a large enough force is also desired (≥25 gf).

When producing a set of results for comparison, the methods (load, dwell, rate) and test type (Vickers, Knoop, etc.) should remain constant.

Table 1. Some literature' parameters and reference values.

Reference	Sample Material	Indenter	Force (gf [N])	Dwell time (sec)	Hardness Result
Baldassarri et al., 2008	Dental enamel in rats	Vickers	100 [0.98]	5	337 ± 44 HV (wet and dry)
Bartlett et al., 2004	Dental enamel in mice	Knoop	10 [0.098]	10	327 ± 70 HK (control)
Beniash et al., 2010	Oyster shells	Vickers	50 [0.49]	5	249 ± 8 HV (control)
Farina et al., 2012	Acrylic resins for denture bases	Vickers	25 [0.25]	30	17.5 ± 2.5 HV

Note: HV (Vickers hardness number), HK (Knoop hardness number), gf (gram-force).

4. References & Applicable Standards

ASTM E384-16	ASTM E384-16, Standard Test Method for Microindentation Hardness of Materials, ASTM International, West Conshohocken, PA, 2016, www.astm.org
ASTM C1327-15	ASTM C1327-15, Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics, ASTM International, West Conshohocken, PA, 2015, www.astm.org

5. Definitions

SOP	Standard Operating Procedure

6. Responsibilities

Technician performing a test on a material using Mach-1™ mechanical tester shall follow the directives of this SOP.
Technician performing this SOP shall have documented training records.

7. Material

- Testing sample. *Note that it is highly recommended to use standard protocol for leveling and surface finish preparation.* Sample specifications:
  - **Thickness**: A minimum thickness of 1.5x the diagonal measurement should be used. Samples of 0.50mm or thicker would conservatively avoid affecting results for microindentation tests.
  - **Leveling**: To obtain valid results, it is essential that the produced indentations are symmetrical. The angle between the indenter and specimen surface should be perpendicular to within 2°.
  - **Surface Finish**: Prepared and polished surface free of damage and roughness that could affect indentation and subsequent measurement. A common guideline is that smaller indentation measurements (smaller forces) require a higher quality of surface preparation.

- Mach-1™ mechanical tester (minimal configuration: model **MA056-v500c**):
  - *Optional* - Resolution Improved to 0.1 µm on a Stage (**MA175**) *(to be identified in associated protocol/report*)
  - Multiple-Axis or Single-Axis Load Cell (*to be identified in associated protocol/report*), either:
    - Calibration = 17N (**MA233**), multiple axis
    - Calibration = 35N (**MA234**), multiple axis
    - Calibration = 70N (**MA235**), multiple axis
    - Calibration = 100N (**MA296**), single axis
    - Calibration = 10N (**MA410**), single axis
    - Calibration = 1.5N (**MA999**), single axis
  - Standard Hardness Test Indenter, either:
    - Indenter Kit, Vickers (Indentation Depth = 40um) (**MA684**)
    - Indenter Kit, Knoop
    - Indenter Kit, Modified-Berkovich
  - *Optional* – Sample holder:
    - 6-Screw (D = 63.5 mm) (**MA646**)

- *Optional* - Automated Indentation Mapping (2 or 3-Axis)
  - Multiaxial Mach-1™ mechanical tester (**MA056-v500cs** or **v500css**)
  - *Optional* – Mapping Toolbox (includes software, camera, lens and fixture) (**MT337**)

8. Methods

8.1 Setup the Mach-1 as per user manual using appropriate accessories.

8.2 Installation of Indenter

8.2.1 Remove indenter from its protective container while keeping its protective sleeve.
8.2.2 Place bottom of indenter into adaptor and tighten using fingers.
8.2.3 Remove protective sleeve from indenter while making sure to avoid touching the diamond tip with fingers.
8.2.4 Clean the indenter tip using a cotton swab and alcohol.
8.2.5 Lightly install the adaptor and indenter into the load cell using the threads. **Be careful to avoid causing excessive torsional or shear forces to the load cell that could damage it.**

Figure 3. Adaptor (top) and indenter (bottom)

Figure 4. Completed installation

8.3 Secure sample in center of testing platform alone or with optional sample holder (**MA646**).

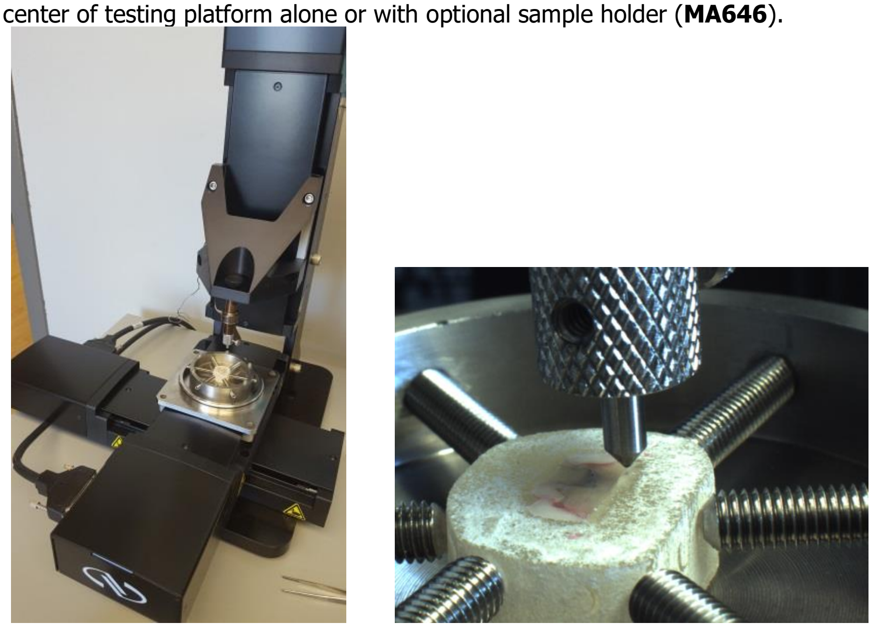

Figure 5. (a) Photograph of Mach-1 test setup (b) Close-up of indenter and sample

8.4 Using the Mach-1 Motion software, (low velocity PID setting recommended – contact Biomomentum for details) manually lower the top fin (Z-axis) about 2 mm over the sample **making sure not to contact the sample as this could overload and damage the load cell**.

8.5 Avoid vibrations through contact with the test table or Mach-1 while performing indentations. Small movements of the indenter head could affect results. *Use of an antivibration table could be useful.*

8.6 Execute the following test sequence to find and zero the surface of the sample:

Functions	Recommended Parameters
Zero load	No parameter
Find contact	Stage Axis: Position (z) Load Cell Axis: Fz Direction: Positive Stop criteria: ~1gf (or ~5X Load Cell Resolution) Velocity: 0.02 mm/s Stage repositioning: 2X Load Resolution
Zero position	Stage Axis: Position (z)

8.7 Execute the follow test sequence to perform the indentation hardness test:

Functions	Recommended Parameters
Zero load	No parameter
Find Contact	Stage Axis: Position (z) Load Cell Axis: Fz Direction: Positive Stop criteria: Desired Load (refer to **Table 1** for some examples) Velocity: 0.0005 – 0.0150 mm/s Data File Path: User Specified
Move Absolute	Stage Axis: Position (z) Position: 0.0000 mm Velocity: 0.0005 – 0.0150 mm/s (same as Find Contact) Data File Path: User Specified

Note 1: Velocity and desired load need to be adjusted based on the estimated mechanical properties of the sample. Successful parameters will produce valid, measurable, and reproducible indentations. A good benchmark is to produce diagonals of over 20µm.

Note 2: Force readings should be reproducible for a set of testing. Modifying the indenter velocity to very low values may allow for a more gradual force buildup (and extend testing time). At the end of preliminary tests, a force vs time graph should be visualized to confirm this accuracy using the Mach-1 Analysis software per its user manual (**SW186-ART01-D**).

8.8 After each test, the sample must be moved for the indenter to compress a new location. This may be done manually while keeping track of indentation locations. If a multi-axis system is used, the "Move Relative" function or the manual axis controls may be used. **Be sure to safely raise the indenter head before performing sample displacements, as hooking the load cell could damage it.**

8.8.1 *Optional* - For automated indentation sequencing, the Mapping Toolbox and its respective user manual can be used to automate the indentation procedure using the "Scan" function.

8.9 **Spacing of indents**: The indents should be far enough from each other so that consecutive tests do not influence results. For microindentation tests (1-1000 gf), the distance between indent centers should be at least 2.5x the diagonal lengths. If cracks are present, the distance between centers should be at least 3x the length of the crack radius. In case of brittle materials such as ceramics, the distance should be raised to 4x the diagonal lengths.

8.10 Make sure to clean the indenter tip after the test. This can be done using a cotton swab and alcohol. Never touch the indenter tip with your fingers.

9. Data Analysis

9.1 Appropriately measure the length the diagonals on the imprinted indent. A minimum accuracy of ± 0.5 µm should be used.

9.1.1 Common instruments for this purpose: Optical Microscope (with high enough resolution), Scanning Electron Microscope (SEM), extremely high resolution video equipment.

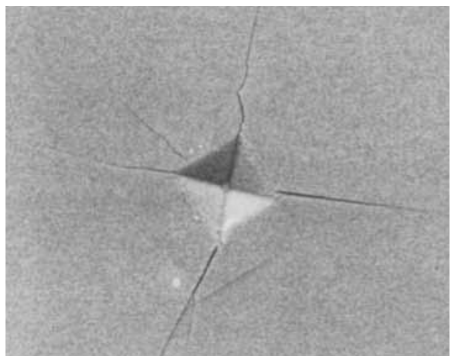

Figure 6. SEM of Vickers indentation (Anstis et al., 1981)

9.2 **Vickers Measurement**: Vickers hardness numbers are commonly written alone without units. Therefore, the final units of $(gf/\mu m^2)$ or (kgf/mm^2) are not applicable. If one half of a diagonal is 5% longer than its other half, the result is normally considered invalid.

9.2.1 Vickers hardness numbers in $gf/\mu m^2$ or (kgf/mm^2) are commonly calculated as follows:

$$HV = 10^3 \times (P/A_{surface}) = 1854.4 \times P/d^2$$

where:
P = force (gf)
d = mean length of indentation diagonals (µm)

9.2.2 Vickers hardness numbers are also less commonly reported in GPa and can be calculated as:

$$HV = 0.0018544 \times P_1/d_1^2$$

where:
P_1 = force (N)
d_1 = mean length of indentation diagonals (mm)

9.3 **Knoop Measurement**: Knoop hardness numbers are commonly written alone without units. If one half of the measured long diagonal is 10% longer than its other half, the result may be invalid.

9.3.1 Knoop hardness numbers in $gf/\mu m^2$ or (kgf/mm^2) are commonly calculated as follows:

$$HK = 10^3 \times (P/A_{projected}) = 14229 \times P/d^2$$

where:
P = force (gf)
d = measured length of long diagonal (µm)

9.3.2 Knoop hardness numbers are also less commonly reported in GPa and can be calculated as:

$$HK = 0.0014229 \times P_1/d_1^2$$

where:

P_1 = force (N)
d_1 = measured length of long diagonals (mm)

9.4 Reporting Results: Hardness numbers are commonly reported using the following style: XXX HV FF, (e.g. 300 HV 0.1) where:
300 = the hardness number
HV = the indenter of choice
0.1 = the magnitude of the force applied (kgf)

9.5 Common Invalid indentations: Asymmetric, large cracks protruding from indentation, coinciding with a large pore in the material.

10. Further Analysis: Fracture toughness (K_C) is another common material characteristic found using this type of testing (Anstis *et al.*, 1981).

11. Referenced Documentation

MA056-ART01-D	Mach-1 - User Manual
SW186-ART01-D	Mach-1 Analysis - User Manual
Anstis et al., 1981	Anstis GR, Chantikul P, Lawn BR, Marshall DB (1981) A critical evaluation of indentation techniques for measuring fracture toughness: I. Direct crack measurements. J Am Ceram Soc 64:533–538
Baldassarri et al., 2008	Baldassarri M, Margolis HC, Beniash E (2008) Compositional determinants of mechanical properties of enamel. J DentRes 87:645–649
Bartlett et al., 2004	Bartlett JD, Beniash E, Lee DH, Smith CE (2004) Decreased mineral content in MMP-20 null mouse enamel is prominent during the maturation stage. J Dent Res 83:909–913
Beniash et al., 2010	Beniash E, Ivanina A, Lieb NS, Kurochkin I, Sokolova IM (2010) Elevated level of carbon dioxide affects metabolism and shell formation in oysters Crassostrea virginica. Mar Ecol Prog Ser 419:95-108
Farina et al., 2012	Farina, A. P., Cecchin, D., Soares, R. G., Botelho, A. L., Takahashi, J. M. F. K., Mazzetto, M. O. and Mesquita, M. F. (2012), Evaluation of Vickers hardness of different types of acrylic denture base resins with and without glass fibre reinforcement. Gerodontology, 29:e155–e160

12. Record Disposition

Records are maintained in accordance with ***BMMT QOP4.02 – Control of Documents and Records***.