Hardness Testing at Elevated Temperatures

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Outline

- What is Hardness?
- How is Hardness Measured?
  - Overview of Hardness Test Methods
- Hardness Testing at Elevated Temperatures
  - Why go to the Trouble?
  - Considerations/Pitfalls
  - A Workable Solution
  - Examples

Scope of this Webinar

1. Not covering Instrumented Indentation
2. Macro or Micro Indentation only

What is Hardness?

- **ISO 14577* Definition:**
  - Resistance of a material to permanent penetration by another harder material.
- **In Mineralogy:**
  - The resistance of a mineral to scratching by another mineral.
- **Practical Definition:**
  - The resistance of a material to Deformation by Indentation or Damage by Scratching.
    - **ASTM Test Methods:** E10 (Brinell), E18 (Rockwell), E92 (Vickers), E384 (Knoop), D2240 (Durometer), G171 (Scratch Hardness), A1038 (Ultrasonic Contact Impedance),…
    - **ASTM Practices:** E448 (Scleroscope), F2496 (Scratch Hardness – Prints and Coatings), …

* Not covering instrumented indentation in this webinar

What are the units of Hardness?

1 – Quantitative, Dimensional Units (stress)

2 – Quantitative, Non-Dimensional, (Comparative) Units

3 – Qualitative or “Arbitrary” Units

1 - Quantitative, dimensional

Units of Hardness

- Stress: Load/Area
  - SI units: kPa, MPa, GPa
  - Imperial units: psi, ksi

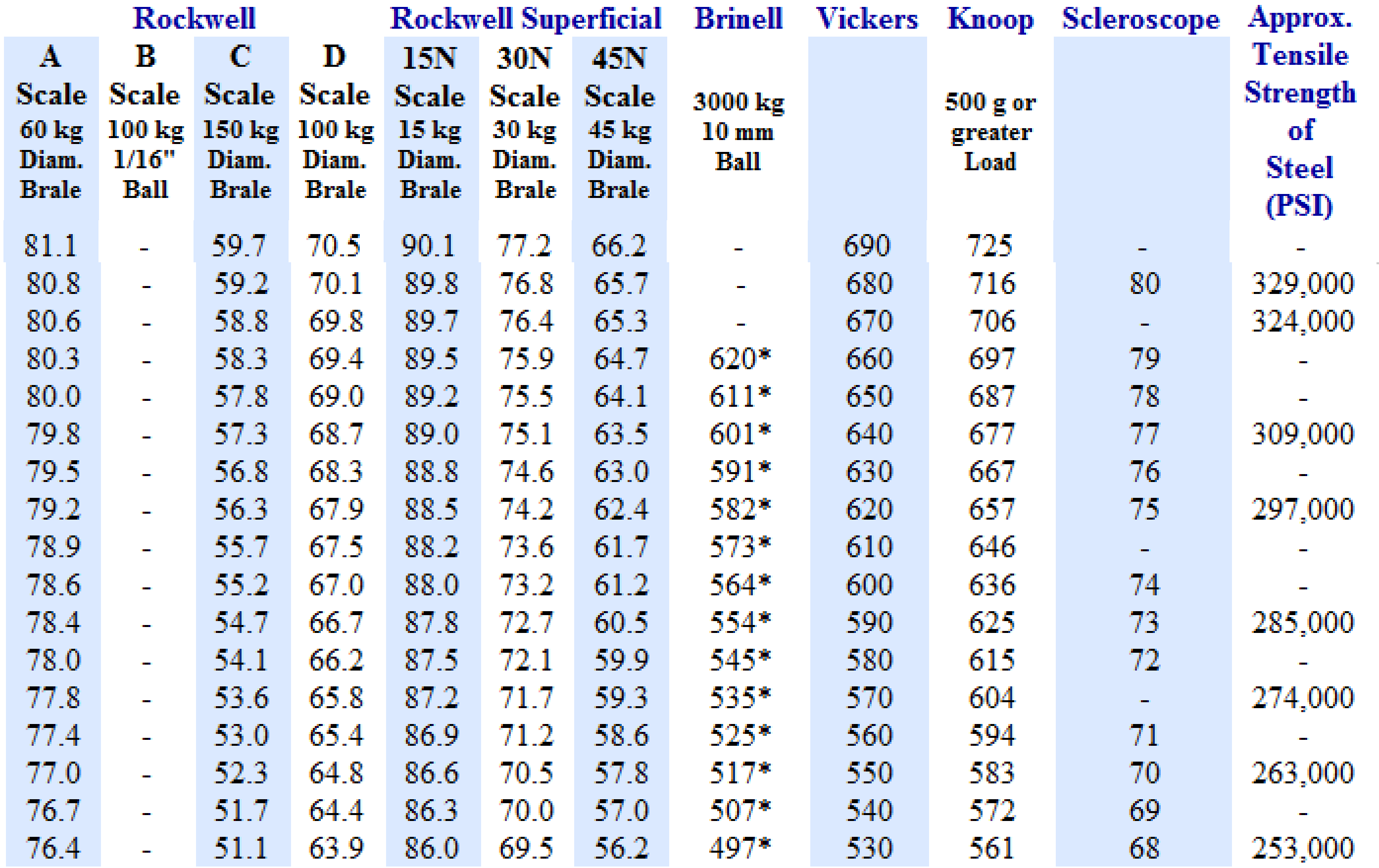
- Hardness Methods which report in units of Stress:
  - Brinell,
  - Vickers,
  - Knoop

Hardness vs. Stress or Strength

- Note that the definitions of hardness on slide 3 did not include stress or strength, yet
  - Hardness is often compared with the Yield Stress or Ultimate Tensile Strength of a material, but…
    - Hardness is generally measured in a compressive state, so "conversion" to tensile test results are primarily empirical
  - The units of some hardness scales are given in stress, but…
    - The stress-reported value of "hardness" of a material will vary a bit, depending on the test (indenter size, shape, load).

Relationship of Hardness to Stress or Strength

“Approximate equivalent hardness numbers for carbon and alloy steels.”



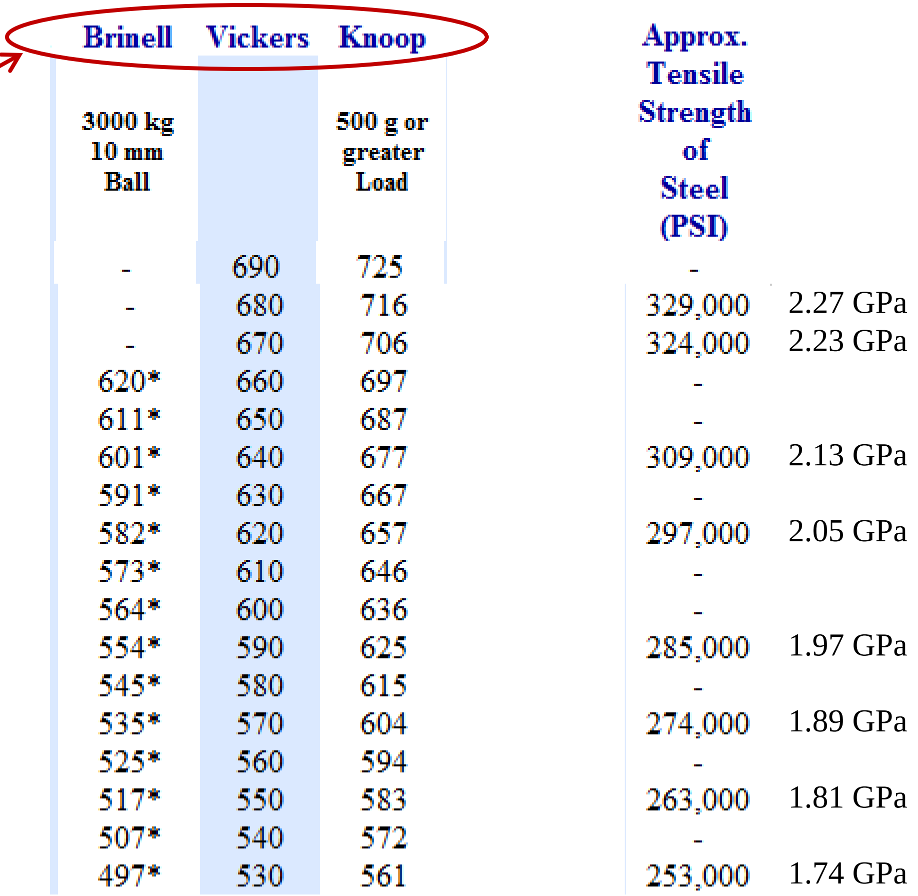
Rockwell				Rockwell Superficial			Brinell	Vickers	Knoop	Scleroscope	Approx. Tensile Strength of Steel (PSI)	
A Scale 60 kg Diam. Brale	B Scale 100 kg 1/16" Ball	C Scale 150 kg Diam. Brale	D Scale 100 kg Diam. Brale	15N Scale 15 kg Diam. Brale	30N Scale 30 kg Diam. Brale	45N Scale 45 kg Diam. Brale	3000 kg 10 mm Ball		500 g or greater Load			
81.1	-	59.7	70.5	90.1	77.2	66.2	-	690	725	-	-	
80.8	-	59.2	70.1	89.8	76.8	65.7	-	680	716	80	329,000	2.27 GPa
80.6	-	58.8	69.8	89.7	76.4	65.3	-	670	706	-	324,000	2.23 GPa
80.3	-	58.3	69.4	89.5	75.9	64.7	620*	660	697	79	-	
80.0	-	57.8	69.0	89.2	75.5	64.1	611*	650	687	78	-	
79.8	-	57.3	68.7	89.0	75.1	63.5	601*	640	677	77	309,000	2.13 GPa
79.5	-	56.8	68.3	88.8	74.6	63.0	591*	630	667	76	-	
79.2	-	56.3	67.9	88.5	74.2	62.4	582*	620	657	75	297,000	2.05 GPa
78.9	-	55.7	67.5	88.2	73.6	61.7	573*	610	646	-	-	
78.6	-	55.2	67.0	88.0	73.2	61.2	564*	600	636	74	-	
78.4	-	54.7	66.7	87.8	72.7	60.5	554*	590	625	73	285,000	1.97 GPa
78.0	-	54.1	66.2	87.5	72.1	59.9	545*	580	615	72	-	
77.8	-	53.6	65.8	87.2	71.7	59.3	535*	570	604	-	274,000	1.89 GPa
77.4	-	53.0	65.4	86.9	71.2	58.6	525*	560	594	71	-	
77.0	-	52.3	64.8	86.6	70.5	57.8	517*	550	583	70	263,000	1.81 GPa
76.7	-	51.7	64.4	86.3	70.0	57.0	507*	540	572	69	-	
76.4	-	51.1	63.9	86.0	69.5	56.2	497*	530	561	68	253,000	1.74 GPa

* http://www.ndt-ed.org/GeneralResources/HardnessConv/HardnessConv.htm.

Relationship of Hardness to Stress or Strength

"Approximate equivalent hardness numbers for carbon and alloy steels."

1) These are all reported in units of kg/mm^2
2) Value depends on test method
3) Poor relation to UTS
4) Good for comparison within the range of the method



Brinell 3000 kg 10 mm Ball	Vickers	Knoop 500 g or greater Load	Approx. Tensile Strength of Steel (PSI)	
-	690	725	-	
-	680	716	329,000	2.27 GPa
-	670	706	324,000	2.23 GPa
620*	660	697	-	
611*	650	687	-	
601*	640	677	309,000	2.13 GPa
591*	630	667	-	
582*	620	657	297,000	2.05 GPa
573*	610	646	-	
564*	600	636	-	
554*	590	625	285,000	1.97 GPa
545*	580	615	-	
535*	570	604	274,000	1.89 GPa
525*	560	594	-	
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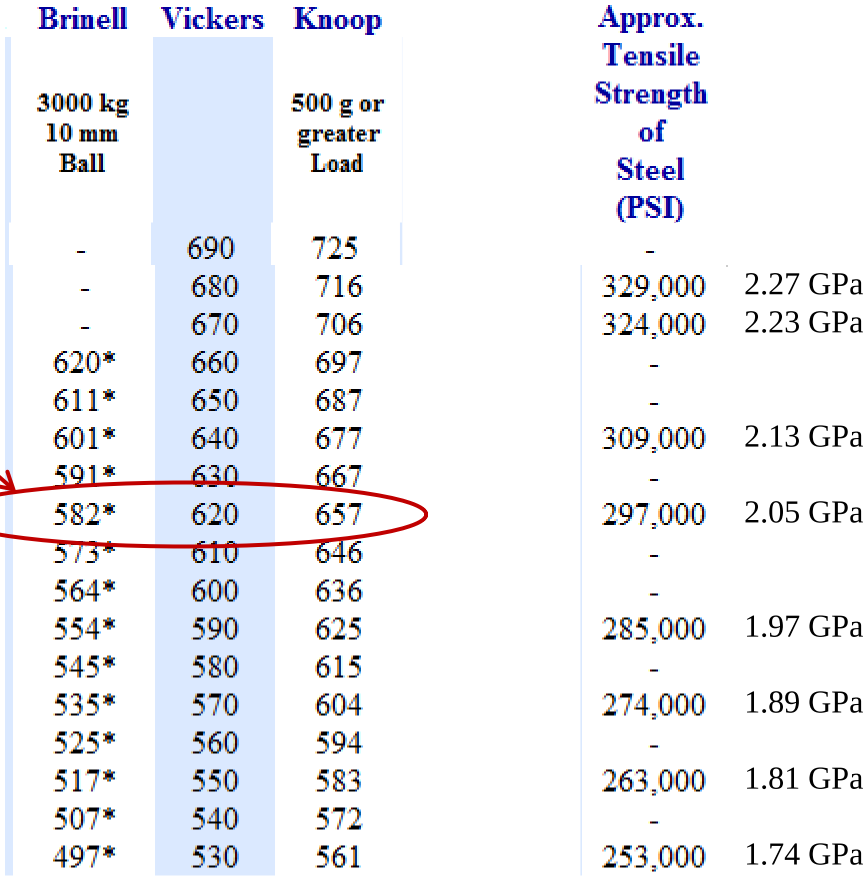

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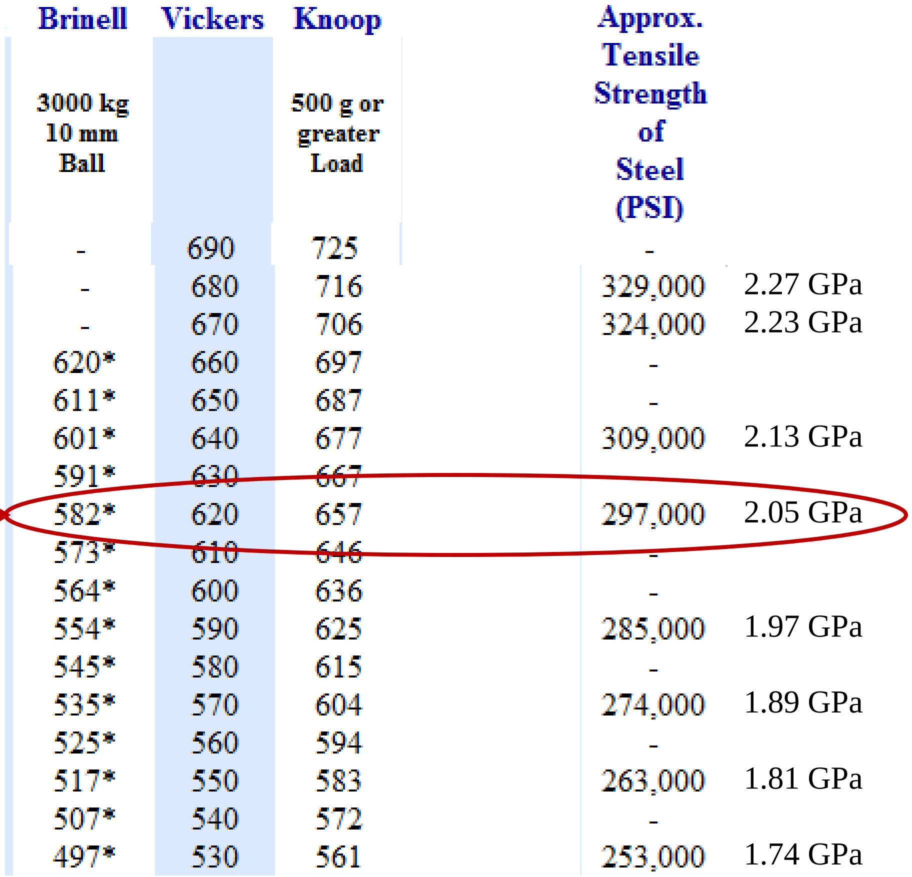

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2 - Quantitative (Comparative)

Units of Hardness

- Comparative Units
  - Rockwell Scales (Less than 100) ASTM E18
    - Formula: (100 - ”permanent deformation” in units of 2 µm*)
  - Shore Scales (Less than 100) ASTM D2240
    - Formula: (100 – depth [depending on spring force])

* - 2 µm is used for “macro” Rockwell tests. For superficial Rockwell tests, the unit for the calculation is 1 µm

3 - Qualitative or Arbitrary

Units of Hardness

- Arbitrary Units
  - Mohs’ Scale (1-10)
    - “A scratches B”

How is Hardness Measured?

1. Scratching

- Mohs’ - A material scratches or is scratched by another.
- ASTM G171 – Scratch Hardness: The width of scratch made using a diamond under a known load is measured.

(To be covered as separate topic)

How is Hardness Measured?

2. Indentation

An indenter of a given shape is pressed into a material at a known load for a given amount of time.

1. The area of the indent caused by permanent plastic deformation after complete removal of the load can be used (Vickers, Knoop, Brinell)

2. The depth of only the permanent plastic deformation after release of the maximum load can be used (Rockwell)

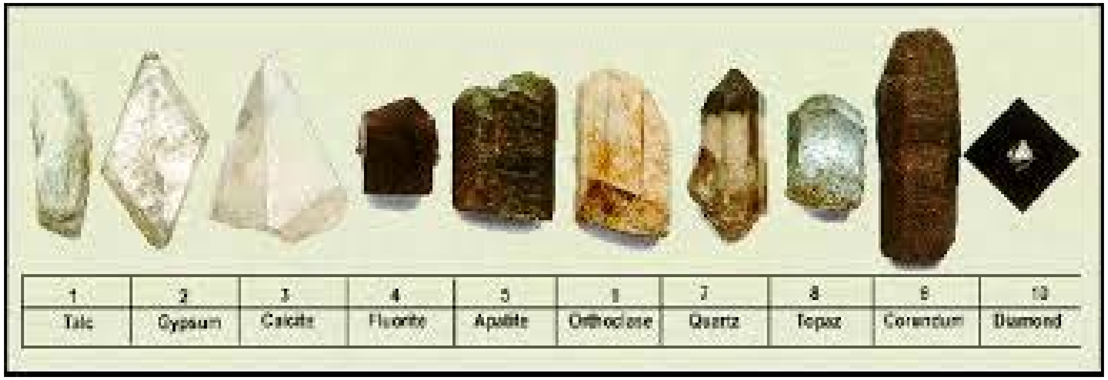
3. The total depth to which the indenter penetrates, including elastic deformation, can be used (Shore)

Measuring Hardness

1. Mohs’
2. Brinell
3. Rockwell
4. Vickers/Knoop
5. Shore

Mohs' Scratch hardness
A Relative Scale

- Friedrich Mohs (29 January 1773 – 29 September 1839)
  - Attempt to scratch a mineral (or material) with a "reference" mineral.
    - No scratch: harder than the reference
    - Scratch: softer than (or equal to) the reference



http://geology.com/minerals/mohs-hardness-scale.shtml

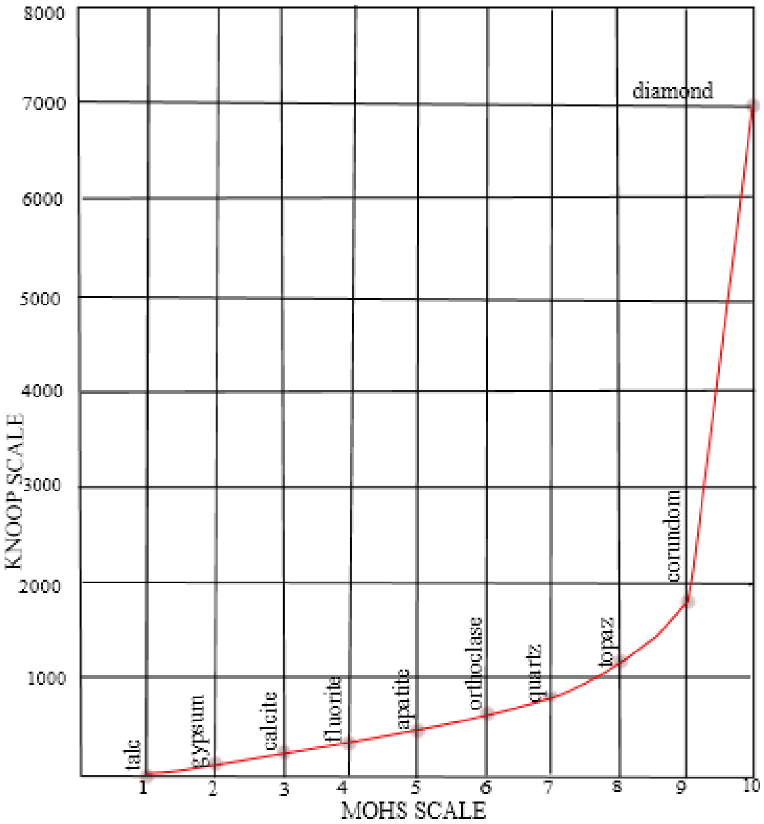
Mohs' Scratch hardness:
A Non-Linear Scale

Mohs' and Vickers

Mohs' Hardness Number	Qualitative Rating	Mineral Example	~ Vickers Equivalent
1	Very Soft	Talc	7
2	Soft	Gypsum	17
3	Soft	Calcite	90
4	Semi-Hard	Fluorite	250
5	Hard	Feldspar	600
6	Hard	Apatite	750
7	Very Hard	Quartz	1,000
8	Very Hard	Topaz	1,500
9	Extremely Hard	Corundum (Al_2O_3)	2,500
10	The Hardest	Diamond	10,000

http://www.rocksforkids.com/RFK/identification.html#Hardness

Mohs' vs. Knoop



From: http://upload.wikimedia.org/wikipedia/commons/1/1f/Knoop-and_Mohs-_scale.svg

Mohs' at Elevated Temperature?

- Advantages:
  - Standardized pick set is available, ranging from Mohs' 2 through 9
  - Tips can be easily held in a fixture for automated testing

http://geology.com/minerals/mohs-hardness-scale.shtml

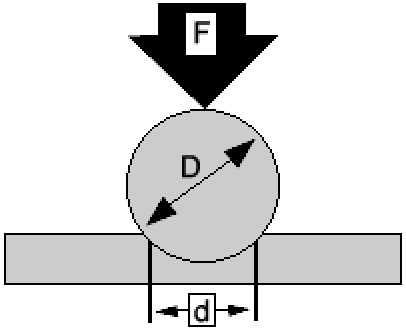
- Considerations:
  - Heat treated alloys can change their hardness with temperature

Brinell Hardness

(ASTM E10, ISO 6506)

Developed in Sweden in 1900 – J.A. Brinell

- A Carbide ball is pressed into a surface under a known load (F), for 10-15 seconds.
- After removal, the diameter of the indentation (d) is measured using a microscope.
- The Brinell Hardness is calculated from the load and the area of the indent, assuming a spherical geometry (D).
- The units of Brinell Hardness (HB) are stress (MPa)



$$HB = \frac{2F}{\pi D\left(D - \sqrt{D^2 - d^2}\right)}$$

(in kg/mm^2)

$$HBW = 0.102 \times \frac{2F}{\pi D\left(D - \sqrt{D^2 - d^2}\right)}$$

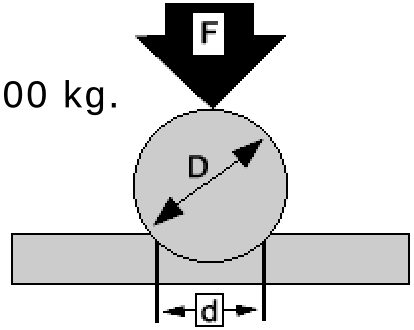
(in MPa)

http://www.instron.us/wa/applications/test_types/hardness/brinell.aspx

Brinell at Elevated Temperatures?

Advantages:

- Tungsten Carbide balls can be used at HT
  - WC itself can withstand temperatures upwards **of 2000° C.**
  - Cobalt (used as binder) melts at **1470° C**.
- Good for large or small parts
  - A variety of ball sizes and loads can be used. (1-10 mm and 1 to 3000 kg.
- Indentations are large (2.5-5 mm) so easy to measure
- Standard Reference Blocks (SRBs) are available



$$HB = \frac{2F}{\pi D\left(D - \sqrt{D^2 - d^2}\right)}$$

Considerations:

- A reasonably “polished” surface is needed, so that the diameter can be accurately determined. Using a microscope
- Very high loads dictate special furnace integrity/support requirements
- No Brinell SRBs for high temperature

http://www.instron.us/wa/applications/test_types/hardness/brinell.aspx

Rockwell Hardness

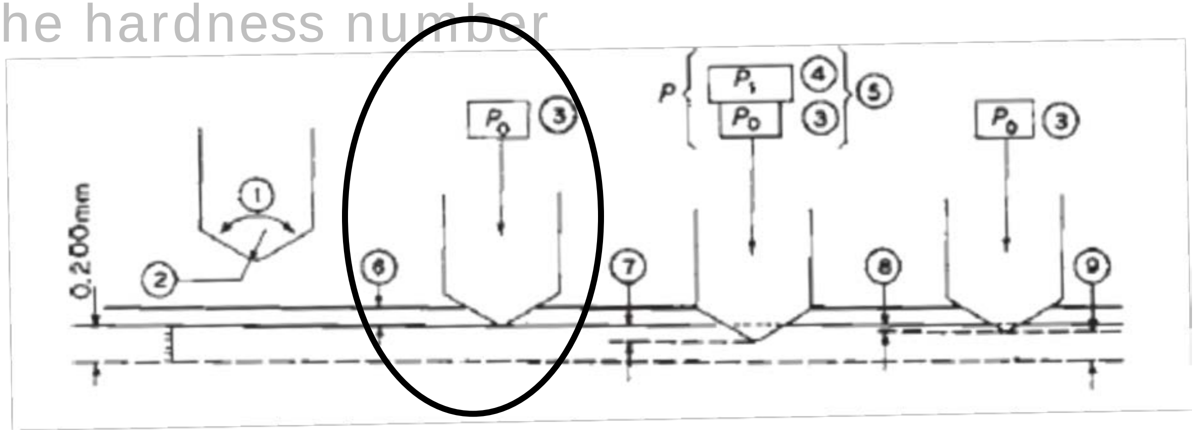
(ASTM E18, ISO 6508, ASTM D785)

1. A “minor load” is applied through a conical diamond (“Brale”) or a carbide ball, and is held for a prescribed time.

   The depth of penetration is noted.

2. A major load is applied and is held for a prescribed time.

3. The force is returned to the minor load, and the depth of penetration at this load, considered “permanent plastic deformation” caused by the major load, is used to calculate the hardness number



* ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Rockwell Hardness

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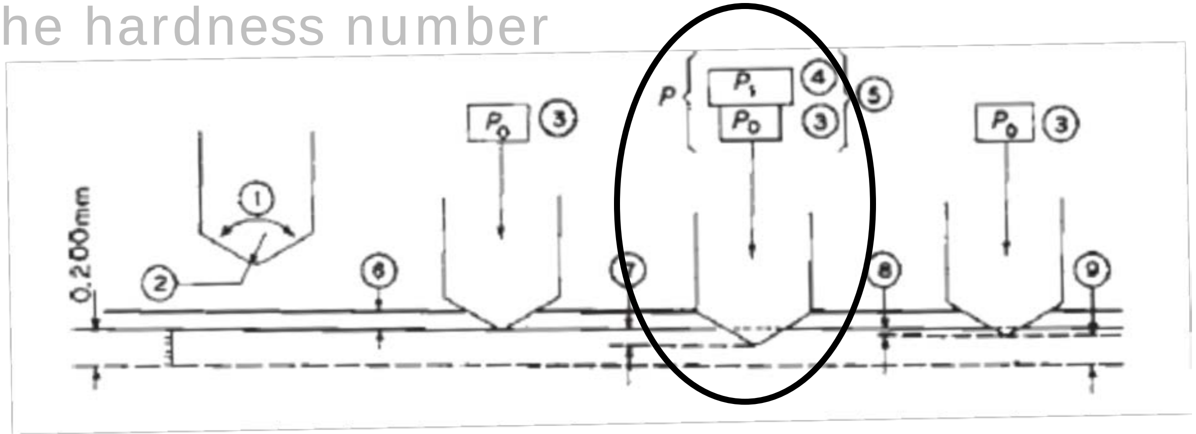

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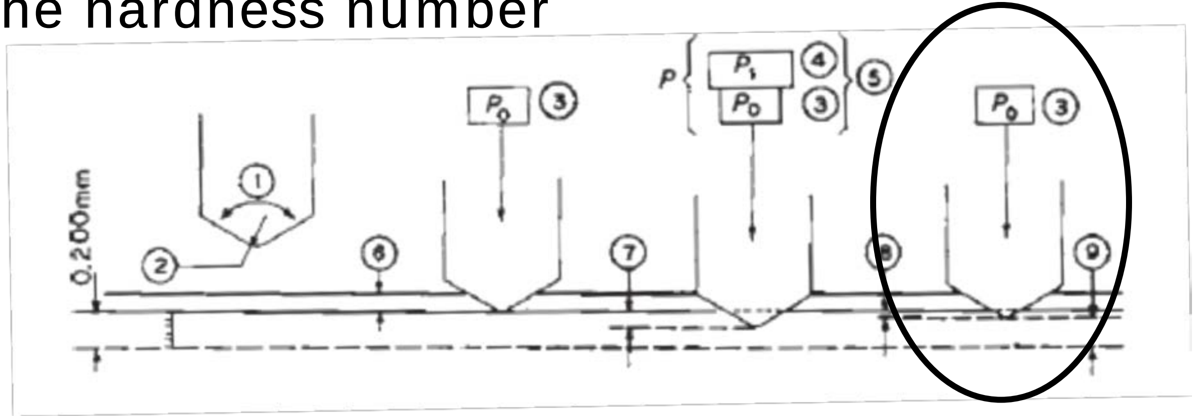

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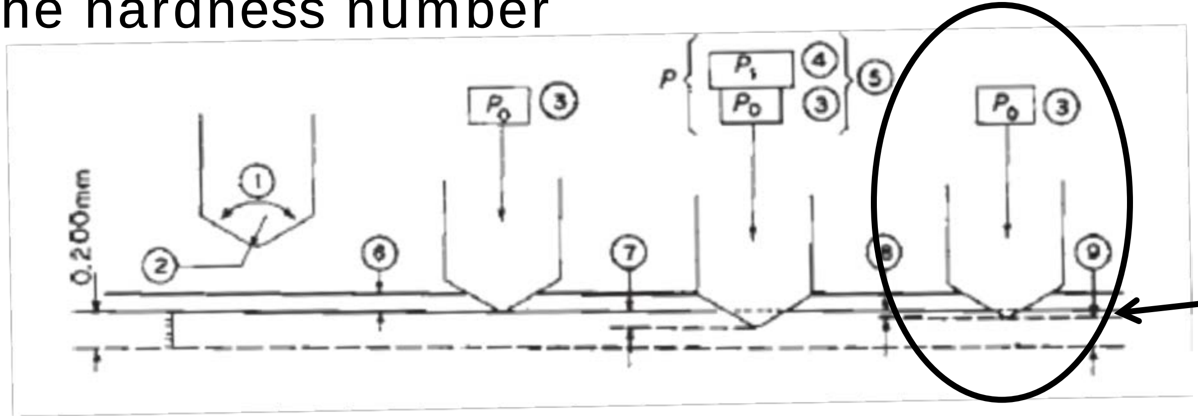
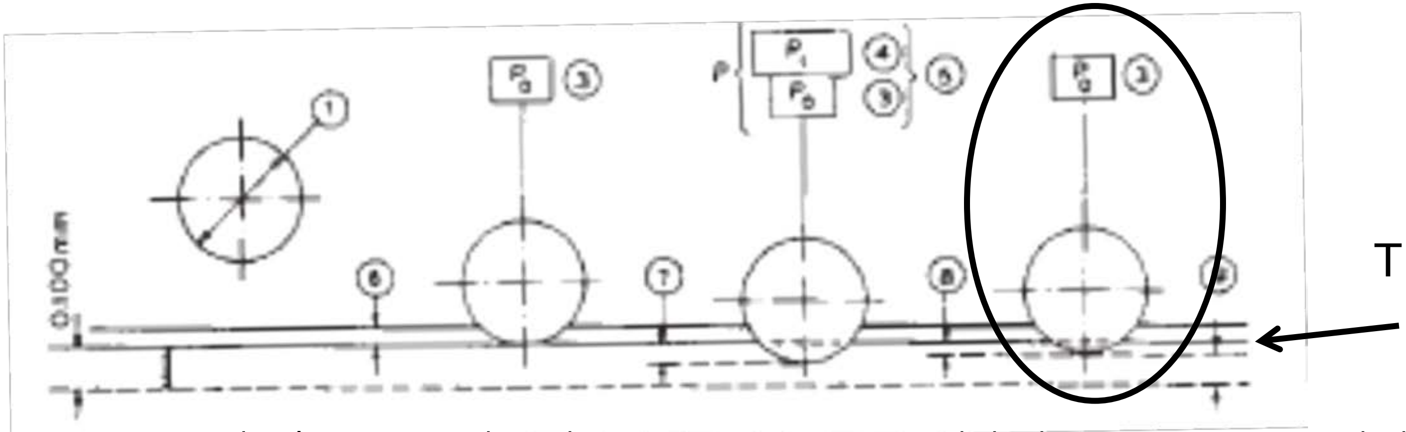


Height difference at minor load.

* ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

Rockwell Hardness Units

- There are **no dimensional units** for Rockwell Hardness measurements
- Rockwell provides quantitative, but relative scales
- The calculation is simply:
  - HRX = 100 – (height difference at minor load, in units of 2 µm*)
  - Irrespective of intenter type or load



This difference is the dimension used.

ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

* - For superficial Rockwell tests, the unit for the calculation is 1 µm

Rockwell Hardness Units

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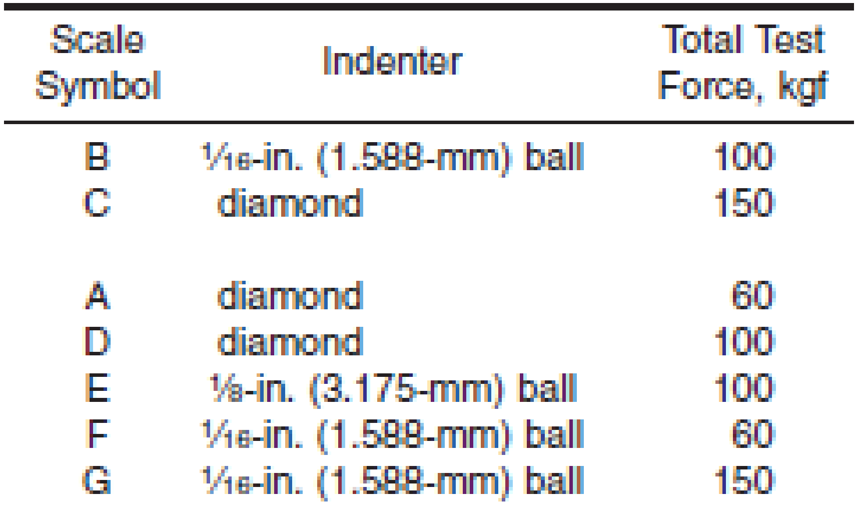
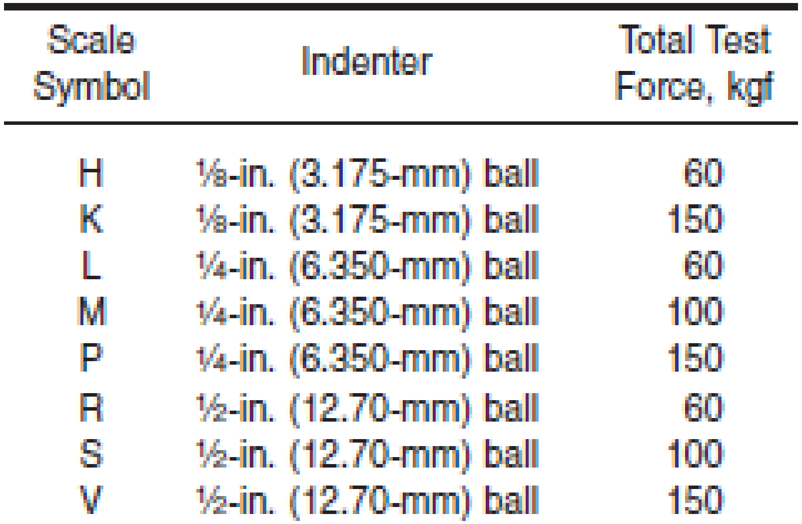
Rockwell “Macro” Tests



Scale Symbol	Indenter	Total Test Force, kgf
B	¹⁄₁₆-in. (1.588-mm) ball	100
C	diamond	150
A	diamond	60
D	diamond	100
E	⅛-in. (3.175-mm) ball	100
F	¹⁄₁₆-in. (1.588-mm) ball	60
G	¹⁄₁₆-in. (1.588-mm) ball	150



Scale Symbol	Indenter	Total Test Force, kgf
H	⅛-in. (3.175-mm) ball	60
K	⅛-in. (3.175-mm) ball	150
L	¼-in. (6.350-mm) ball	60
M	¼-in. (6.350-mm) ball	100
P	¼-in. (6.350-mm) ball	150
R	½-in. (12.70-mm) ball	60
S	½-in. (12.70-mm) ball	100
V	½-in. (12.70-mm) ball	150

* - For superficial Rockwell tests, the unit for the calculation is 1 µm

Rockwell Hardness
“Superficial”

- Used for softer or thinner samples
- The calculation is nearly identical:
    - HRX = 100 – (height difference at minor load*, in units of 1 µm)

Rockwell “Superficial” Tests

Scale	Indenter	Test Forces (kg)
15N, 30N, 45N	Diamond Brale	15, 30, 45
15T, 30T, 45T	1.59 mm ball	
15W, 30W, 45W	3.18 mm ball	
15X, 30X, 45X	6.35 mm ball	
15Y, 30Y, 45Y	12.7 mm ball	

* - For superficial Rockwell tests, the minor load is 3 kg,

Rockwell at Elevated Temperatures?

Advantages:

- A very wide range of materials can be covered
  - A variety of indenter shapes and loads can be used.
  - Well defined indenter geometries, alternate indenter materials can be used
- Microscope is not required for measurement of indent dimensions
- Indentation depth change is measured in-situ, at constant load
  - Not influenced by frame stiffness/compliance
  - Measurement is of many 10s of microns, so precision demand is easily met
- Rockwell Standard Reference Blocks (SRBs) are available

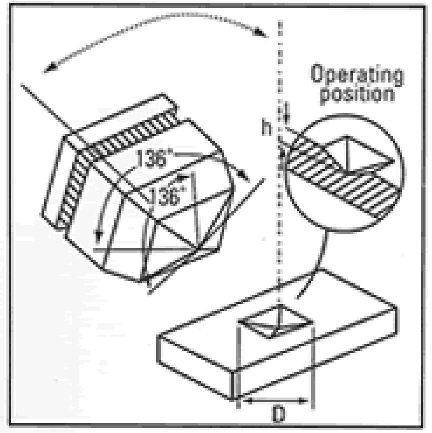
Considerations:

- Diamond indenters are susceptible to oxidation and thermal degradation above ~450° C.
- No Rockwell SRBs for high temperature

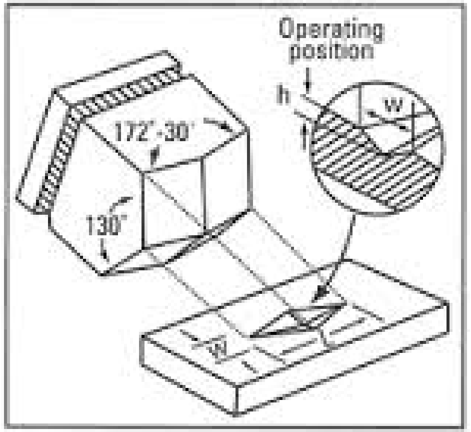
Vickers/Knoop Hardness

(ASTM E92/E384, ISO 6507/4545)

- Diamond with specific tip geometry
  - Vickers – 136° angle between opposite faces
  - Knoop – two angles, 172.5° and 130°



http://www.instron.us/wa/applications/test_types/hardness/knoop.aspx



http://www.instron.us/wa/applications/test_types/hardness/knoop.aspx

- Load is applied for specified time (10-15 sec)
- After load is removed, length of diagonals are measured using a microscope.
- Contact area is calculated from geometry of indenter

Vickers/Knoop Hardness

Method

Vickers and Knoop calculations give quantitative hardness in units of Stress: load/area

$$HV = F_{(kg)}/A_{(mm^2)} \approx 0.1891\ F/d^2$$

d is the average length of the 2 diagonals

$$HK = F_{(kg)}/A_{(mm^2)} \approx 14.229\ F/L^2$$

L is the length of the long diagonal

Vickers/Knoop at Elevated Temperatures?

Advantages

- Indenter angle provides great sensitivity for low to moderate range of depth penetration
  - 7:1 for Vickers (diagonal to depth)
  - 30:1 for Knoop
- By choosing an appropriate load, Vickers and Knoop scales can cover the complete range of metals and other materials hardness
- Knoop can be used for relatively thin layers, small samples, and cross-sectional near surface profile measurements.
- Vickers and Knoop Standard Reference Blocks (SRBs) are available

Considerations

- Same diamond thermal considerations as other methods
- Samples must have a polished surface
- Have to find the indentation afterwards
- No Vickers or Knoop SRBs for high temperature

Shore Hardness Test Method

ASTM D2240

Albert Shore (1920s) – Instrument called the “Durometer”

- Primarily for softer materials: polymers, elastomers, rubber
- A metal pin, with defined tip geometry, is pressed into the sample under a known force
  - Measurements are made under fixed, specific load
  - Includes elastic and permanent deformation
  - The Shore Hardness number is inversely proportional to the amount of penetration (0 to 2.5 mm)
    - 12 scales for different ranges of materials
    - Dimensionless, quantitative scales, (0-100)
    - Most common are Type A and Type D

Durometer at Elevated Temperatures?

Advantages

- Simple test
- Designed for soft materials, so conducive to decreased mechanical properties at high temperature

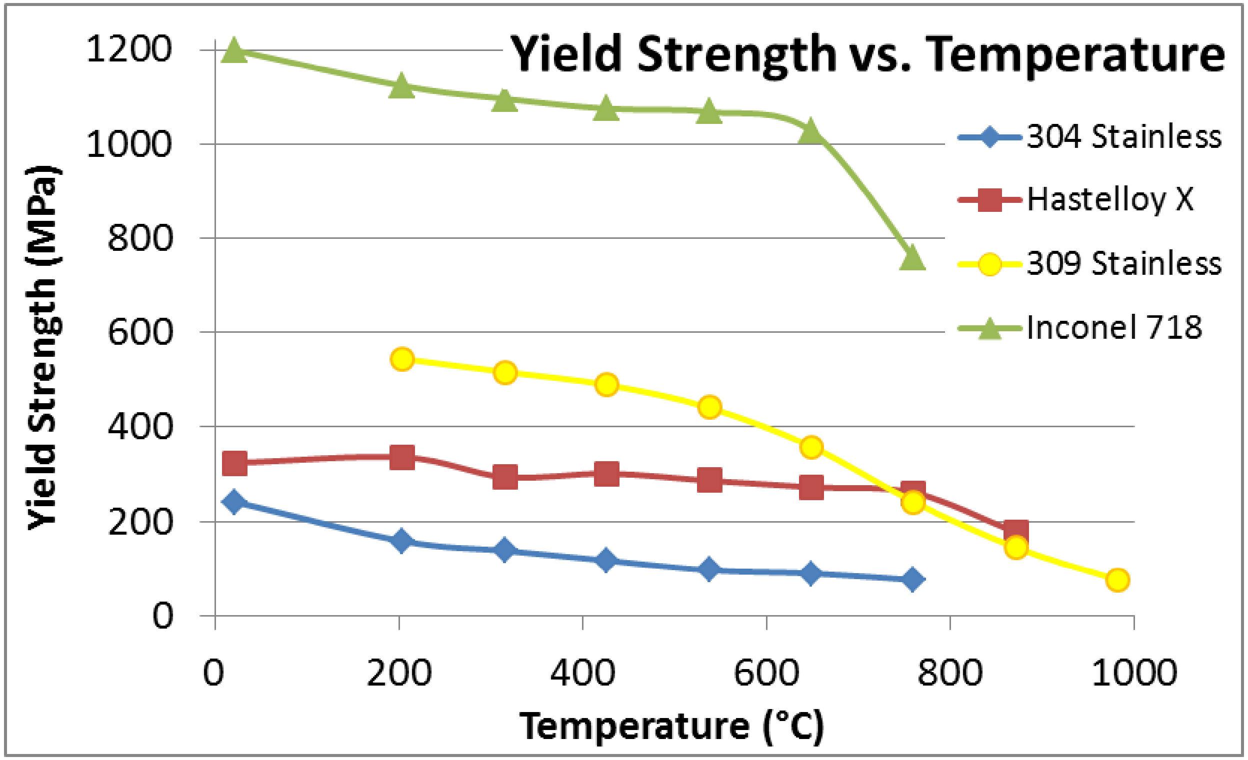
Considerations

- Cannot measure high hardnesses of materials at lower temperatures
- Steel tips not conducive to very high temperatures
- System compliance influences measurements
- Standard Reference Materials for HT are not available

Hardness Testing at Elevated Temperatures

Why Hot Hardness?

- Material Properties typically decrease with Temperature



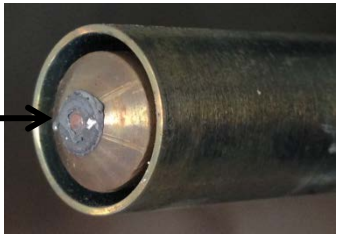
Why Hot Hardness?

- Because Hardness is one measure of strength, testing at elevated temperatures provides:
    - A means to rank materials' strength at elevated temperatures
    - A means to assess abrasion resistance at elevated temperatures
    - A means to assess creep behavior
    - Sample preparation is simplified and facilitated over tensile testing
    - A simple means to identify critical temperature for properties degradation

Hot Hardness Technical Considerations

Hot Hardness Technical Considerations

1. Degradation of diamond, other system components and some samples at HT in the presence of oxygen
    - Indenter tip must withstand same temperature as samples (including bond material)
    - Indenter tip and holder material must withstand loads and furnace temperature



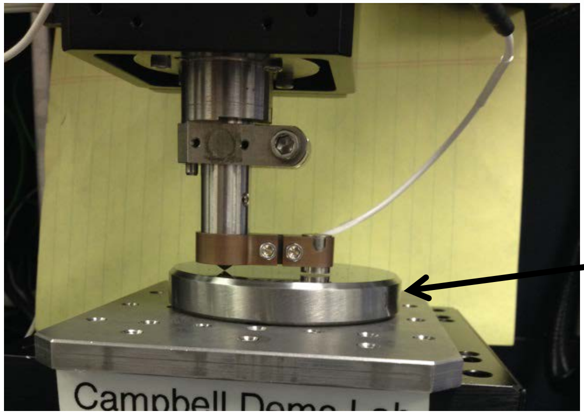
Extruded Braze

Buckled Neck

2. Test rig requirements: Accurate load, temperature, indentation depth measurement at high temperature
    - Thermal expansion of loading train may influence result accuracy, depending on method of measurement
    - System compliance/stiffness under load

Hot Hardness Technical Considerations

3. No available Standard Reference Blocks for HT Hardness



Standard Reference Block
(RT only)

4. Selecting the right combination of load and indenter

   Depth of indentation of soft materials (at HT) at major load may exceed range of measurement for test method

5. Indentations from Vickers/Knoop diamond may be challenging to find/measure

6. Thermal isolation of sensitive components

Discussion/Resolution of Technical Considerations

1. Degradation of components at HT
   - Use inert cover gas or vacuum for diamond (HRA HRC, Vickers/Knoop)
   - WC ball can be used for other scales (HRB, HRE, Brinell)
   - Sapphire tip of same geometry (already an oxide)
   - Use high temperature materials (e.g. Hastelloys, Inconels) for holders
2. Measurement accuracy and system compliance/stiffness
   - Thermal Expansion:

     Sufficient soak time for steady state conditions at each temperature

     Monitor sample temperature with separate thermocouple

     For Rockwell-style measurements, measurements are taken within a few second of each other, so very small change in temperature occurs, and does not significantly compromise accuracy
   - System Compliance:

     Obtain and incorporate system stiffness curve in necessary calculations

     Rockwell-style method uses depth difference at same minor load

       Depth at major load is <u>not</u> used in calculation

Discussion/Resolution of Technical Considerations

3. High Temperature Reference Standard availability

- No immediate solution
  - Can compare relative change for known materials from published data
  - Can compare ranking of materials, also with known data
  - Send request to NIST for consideration

4. Selecting the right combination of load and indenter

- Choose combination of load and indenter tip to minimize penetration depth, but allow discrimination between materials
- Use consistent combination for materials within range of hardnesses expected
- Some experimentation will be necessary

Discussion/Resolution of Technical Considerations

5. Indentations from Vickers/Knoop diamond may be challenging to find/measure
    - For vacuum systems, use fixed external microscope and indexing turret with calibrated offset for internal indenter
    - Use insertable fiber optic imaging system
    - Measure indents after removal of sample from furnace, indexed from reference surface of stage or sample holder
    - Use depth of indent and tip geometry to calculate diagonal length

6. Thermal isolation of sensitive components
    - Use sufficient insulation and fans to keep force sensors below 100° C

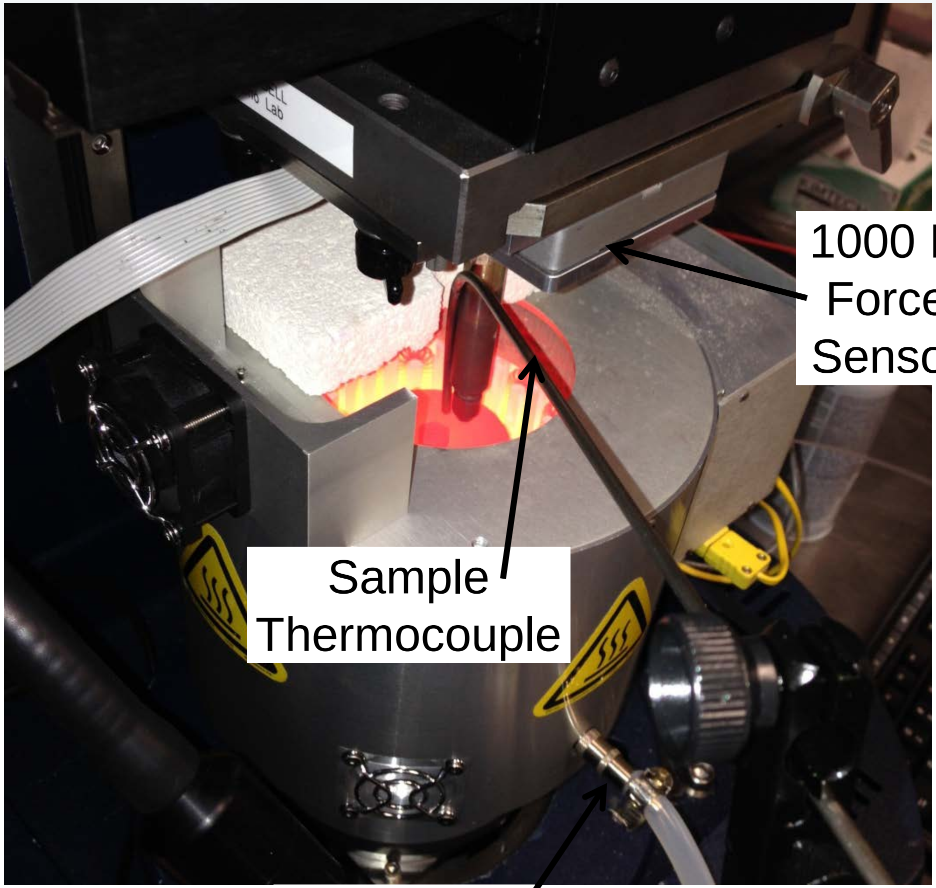
A Viable Method for High Temperature Hardness:

- Use Rockwell-style method of change in depth at minor load
- Choose Appropriate Indenter and load
  - Small WC ball (3.2 mm diam), 50 kg major load
- Provide descriptive designation
  - $\mathrm{HBB}^{3.2}_{50}$

Ball Diam in mm

Major Load in kg

Example of HT Hardness Solution



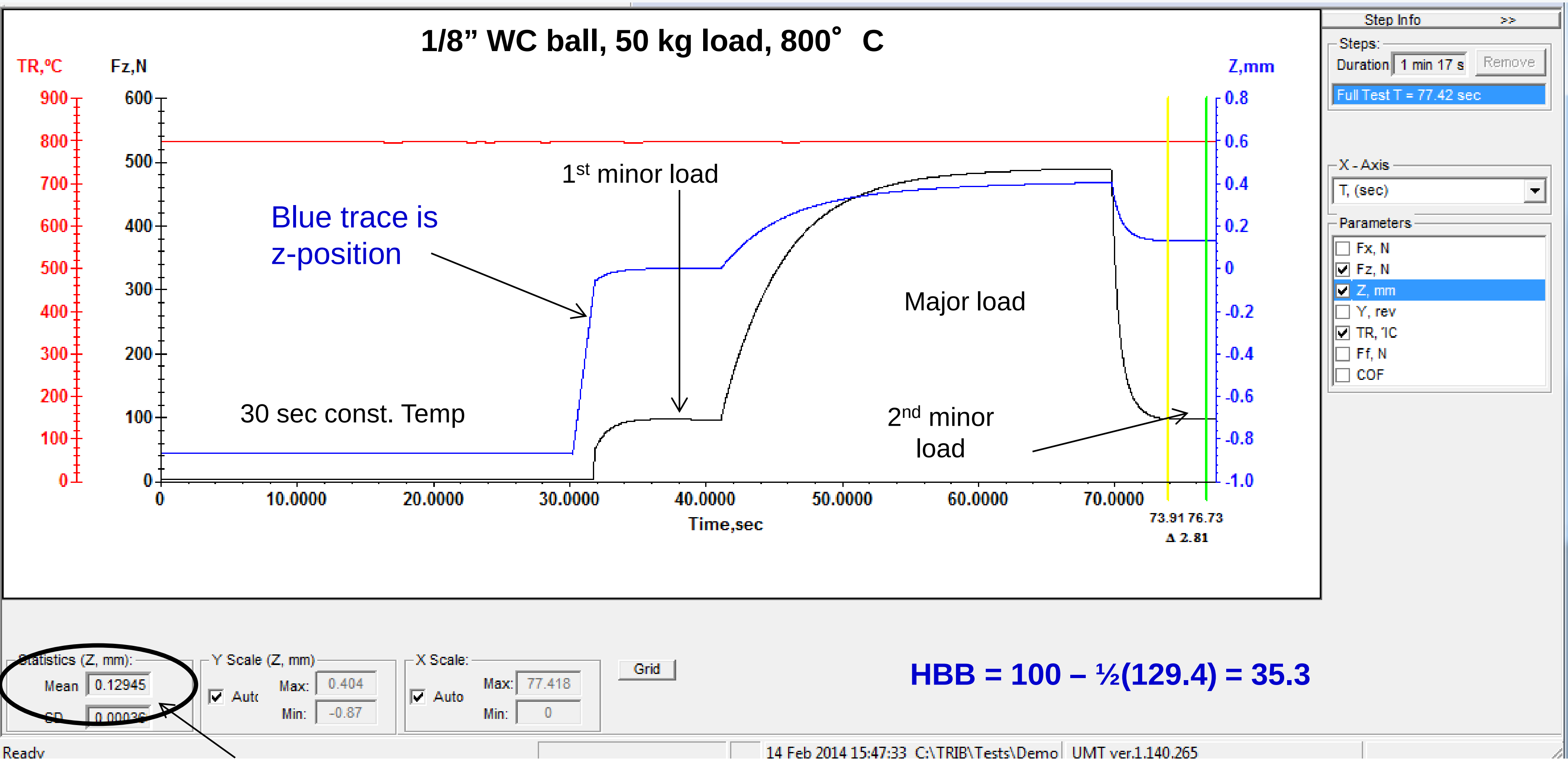
1000 N Force Sensor

Sample Thermocouple

Cover Gas Inlet Port

1/8” Diam. WC Ball

Data Example:



1/8” WC ball, 50 kg load, 800° C

Blue trace is z-position

1st minor load

Major load

30 sec const. Temp

2nd minor load

Steps:
Duration: 1 min 17 s
Full Test T = 77.42 sec

X - Axis: T, (sec)

Parameters:
- ☐ Fx, N
- ☑ Fz, N
- ☑ Z, mm
- ☐ Y, rev
- ☑ TR, 'IC
- ☐ Ff, N
- ☐ COF

Statistics (Z, mm):
Mean: 0.12945
SD: 0.00036

Y Scale (Z, mm):
☑ Auto
Max: 0.404
Min: -0.87

X Scale:
☑ Auto
Max: 77.418
Min: 0

HBB = 100 – ½(129.4) = 35.3

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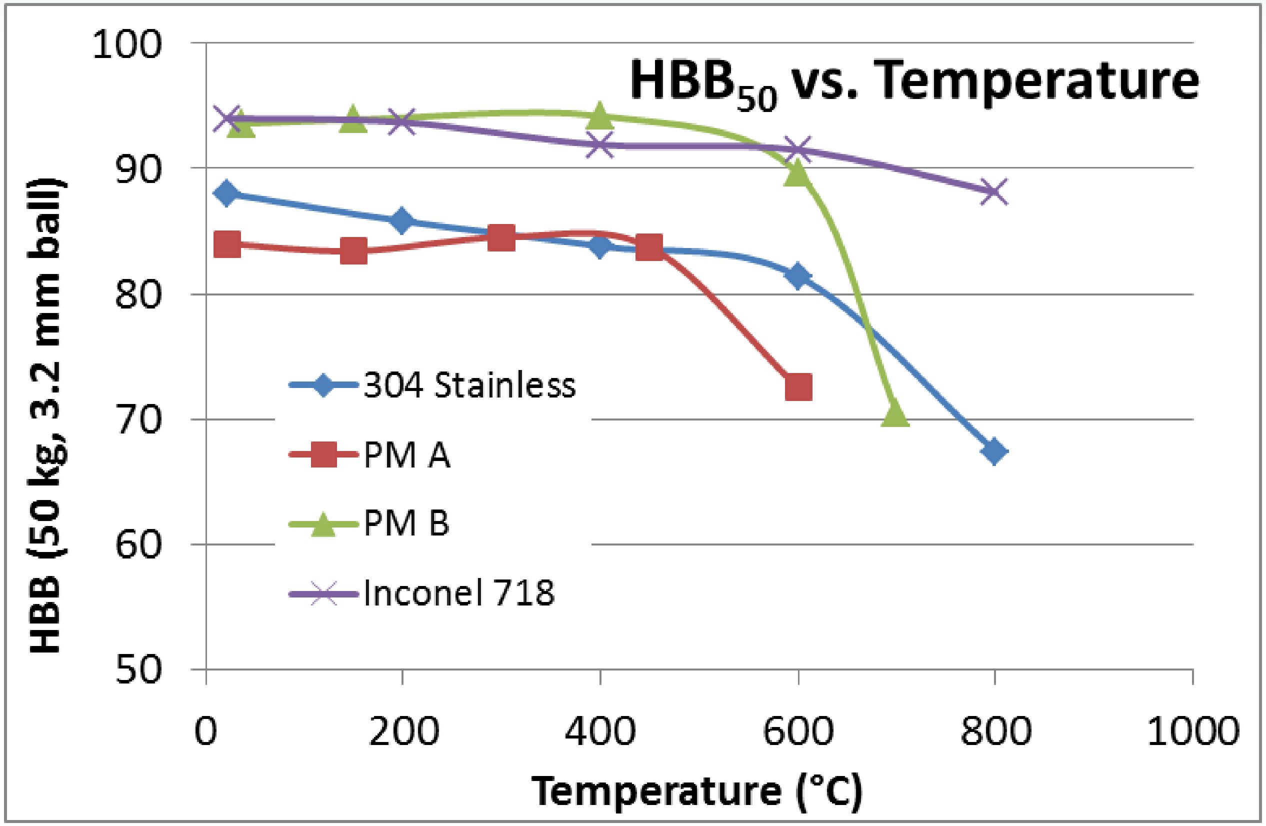
Shows average value of selected value between cursors

Data Example:

HBB$^{3.2}_{50}$



PM-A would be a suitable substitute for 304 SS at or below 400° C

PM-B would be better than 304 SS up to 700° C, and equal to Inconel 718 up to 600° C

Inconel 718 is best choice at 800° C

Concluding Remarks
for High Temperature Hardness

- With appropriate considerations for test equipment and technique, HT hardness can be readily measured
- HT hardness measurements can be obtained using existing Bruker UMT platform
- Conversion tables to high temperature yield and HT tensile data must be developed over time, just as they have been for RT methods
  - Collaborative work between different labs is needed to develop and grow such a database
  - High Temperature Reference Standards would be valuable to aid such collaborations

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