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2.002 Mechanics and Materials II
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Laboratory Module No. 5

Heat Treatment of
Plain Carbon and Low-Alloy Steels:
Effects on Macroscopic Mechanical Properties.

1 Background and Objectives

Iron is one of the oldest known metals, and carbon is the cheapest and most effective alloying element for hardening iron. Iron-Carbon alloys are known as "**carbon steels**" and account for more than 70% of the tonnage of metallic materials used in the United States for engineering applications. Carbon is added to iron in quantities ranging from 0.04 to 2 wt% to make low, medium, and high carbon steels. The microstructure and resulting mechanical properties of these steels are amenable to modification via **heat treatment**, and a wide range of mechanical properties can be obtained by proper variations of heating and cooling cycles. Modest amounts (up to a few wt% percent each) of costlier alloying elements such as nickel, chromium, manganese, and molybdynum can be added to the composition, resulting in "**low alloy**" [content] steels that possess additional desirable properties, including achievability of high strength and good ductility in larger sections.

In this laboratory module we will demonstrate the essential steps involved in the heat treatment of a medium carbon steel, AISI 1045 (Fe + 0.45 wt % C + 0.75 wt % Mn + 0.2 wt % Si), and a low alloy steel, AISI 4140 (Fe + 0.40 wt % C + 0.75 wt % Mn + 0.2 wt % Si + 1.0 wt % Cr + 0.2 wt % Mo), and measure macroscopic mechanical properties of the materials after different heat treatments.

2 Lab Tasks

In this laboratory module we will perform the following tasks:

- Introduce, in elementary terms, essential steps involved in the heat treatment of carbon and low alloy steels, and briefly discuss aspects of the underlying materials science.

- Demonstrate **austenitizing**, **annealing**, **quenching**, and **tempering** heat-treating procedures for AISI 1045 and/or AISI 4140 steels.

- Conduct mechanical tests on specimens of 1045 and/or 4140 steels which have been subjected to four different heat treatment procedures:

    1. As-received (normalized);
    2. Austenitized and slow-cooled (annealed);
    3. Austenitized and water-quenched; and
    4. Austenitized, water-quenched, and tempered.

    For each material condition and associated microstructure we will perform:

    1. A tensile test,
    2. A Rockwell hardness test.
       Also, in order to get a tangible perception of the relative properties of the three material conditions, we will manually bend rods and compare the relative loads required to initiate plastic deformation.
    3. A Charpy U-notch impact test.

- We will discuss the correlations between the measured mechanical properties (strength, hardness, ductility, impact energy) in the various heat-treated conditions.

3 Lab Assignment: Specific Questions to Answer

1. Describe the heat treatment processes of 1045 (or 4140) steel introduced during the laboratory session.

2. Typical tensile stress-strain behavior for 1045 steel, in the four conditions considered, will be generated. From these curves for each of the four heated-treated material states, obtain the following tensile properties:

   (a) Young's modulus, E;

   (b) Tensile yield strength (0.2 percent offset), σ_y;

   (c) Ultimate tensile strength, σ_{UTS};

   (d) The reduction in area at fracture, $q \equiv (A_0 - A_f)/A_0$ where $A_0 \equiv$ original cross-sectional area, and $A_f \equiv$ final cross-sectional area of the tensile test specimens. Note that q is a measure of ductility, or plastic strain to failure.

   (e) Based on the Rockwell hardness data collected during the lab session, estimate the ultimate tensile strength $\sigma_{\text{UTS}}^{(\text{HRC})}$ of the alloy in the three conditions considered. To complete this task use the conversion charts attached to this handout.

   (f) Tabulate, for each microstructural state, the tensile properties, $(E, \sigma_y, \sigma_{\text{UTS}}, q)$, the Rockwell hardness and corresponding estimated tensile strength $\sigma_{\text{UTS}}^{(\text{HRC})}$, and the Charpy U-notch impact energy $(\mathcal{E}_U^{(\text{Charpy})})$ data.

3. Identify trends in the data. In particular, how do the moduli, strength, ductility, hardness and impact energy change with heat treatment?

4. How well does the hardness-estimated tensile strength, $\sigma_{\text{UTS}}^{(\text{HRC})}$, compare with the actually-measured tensile strength, σ_{UTS}? In particular, what is the source of discrepancy for the data from the austenitized and quenched condition? (Hint: Does the compressive yield strength correlate better with the hardness-estimated "tensile strength" in those cases where there is a major discrepancy between the estimate and the maximum measured stress in the tensile test?)

5. Over the set of lab sections, data was obtained for the quenched-and-tempered condition using a range of tempering temperatures (see the web page for a summary of the results). Identify and discuss trends in the resulting properties with respect to the tempering temperature.

4 Introduction to Heat Treatment of Plain Carbon and Low-Alloy Steels

Additional reference material: Dowling, Section 3.3

It is perhaps presumptuous to attempt even a cursory synopsis of the vast literature surrounding the heat-treatment of carbon and low alloy steels. There are at least two levels at which the subject can be approached. One level constitutes a phenomenological description of what thermal histories are applied, along with details such as temperature levels, times of holding at temperature, maximum allowable time limits for executing "rapid" temperature changes, and technical nomenclature associated with the processes. A second, and more ambitious level focuses on fundamental physical processes, equilibrium states of matter under different temperatures, and kinetics of the processes by which one state transforms to another under changes in temperature. The second level is based on fundamentals of thermodynamics, kinetics, and materials science which can not be easily (or effectively) introduced in a brief format such as the present laboratory module. Nonetheless, we will attempt to highlight "major aspects" of the heat-treating carbon and low alloy steels from both the phenomenological/technological and the scientific/academic points of view.

The first notion of importance is that of *equilibrium state* of a collection of matter. We are familiar with the changes of state of a pure substance like water, as a function of temperature: at sufficiently low temperatures, (below $0°C$), the stable, equilibrium state is a solid (ice), while at ambient pressure and higher temperatures, water is liquid. What may not be as well-appreciated is that the equilibrium crystal microstructure of ice is not constant, but depends on temperature and pressure.

And so it is with the solid states of the substance pure iron (Fe):

1. At ambient pressure and temperatures between 1394C and 1534C (the latter of which is the melting temperature of pure Fe), its equilibrium phase is a body-centered cubic (BCC) crystal called δ-iron;

2. At temperatures in the range 912C to 1394C, the stable phase is a face-centered cubic (FCC) phase called γ-iron;

3. While at temperature from absolute zero to 912C, the equilibrium phase is a BCC crystal structure called α-iron.

For our purposes, only the temperature ranges corresponding to stable α and γ phases are of interest.

When iron is alloyed with carbon (C), in concentrations corresponding to $0.04\,wt\%$ to $2.0\,wt\%$ one obtains "carbon steels". In this case, an essential question is: "*What happens to the carbon atoms in the iron crystal structures?*" Broadly speaking, there are two places for the carbon atoms to go:

1. They can dissolve in **solid solution** into the Fe crystal structure (either BCC γ or FCC α), leading to a single phase (crystal with dissolved C):

    (a) When the C is dissolved into the γ crystal phase of Fe, it is termed **austenite**: solid solution C in FCC iron;

    (b) When the C is dissolved into the α crystal phase of Fe, it is termed **ferrite**: solid solution C in BCC iron.

2. Alternatively, carbon atoms can form a chemical reaction with iron atoms, forming a crystalline second-phase iron carbide compound, Fe_3C, called **cementite**, which precipitates out into spatial regions separate from the solid-solution phase. In general, this carbide is more resistant to plastic deformation than the iron/carbon solid solution.

The "equilibrium" structure of polycrystalline low carbon steels at low temperature is two-phase: there are spatial regions of the cementite compound (Fe_3C) distributed within ferrite crystals (α); the resulting two-phase microstructure is sometimes denoted by "α + Fe_3C". A map of the relevant portion of the **equilibrium phase diagram** for the Fe-C system is shown in Figure 1, showing the combinations of temperature (vertical axis) and carbon weight percent (horizontal axis) leading to the various equilibrium microstructures.

Image removed due to copyright considerations. See Figure 11.1 in Ashby, M. F, and D.R.H. Jones. *Engineering Materials 2: An Introduction to Microstructures, Processing and Design*. Elmsford NY: Pergamon, 1986.

The most important single fact about the iron-carbon system is that the solubility of C is γ-iron is **very much larger than** its solubility in α-iron. (The maximum possible equilibrium C content in α iron is only 0.0218 wt %, occurring at 723 C, as shown in Fig. 1.) This fact is exploited in constructing heat-treatment thermal histories that produce special microstructures in carbon steels; in turn, these distinct microstructures endow the heat-treated steel with distinct material properties.

Some common heat-treatments for low and medium carbon steels:

1. **Austenitize and Air-Cool**:

   This is the typical heat-treatment given to the steel by the manufacturer, and is accordingly termed the **as-received** condition. The thermal history leading to this state is also called **normalizing**. Normalizing of 1045 steel typically consists of the following steps:

   - **Austenitize**: put in furnace at 850C in the austenite range, and hold for 1

hour until equilibrium temperature and corresponding solid solution structure have been reached.

- **Air-cool**: remove from furnace and allow to air-cool to room temperature.

2. **Austenitize and Furnace-Cool**:

   This heat-treatment is sometimes also called **annealing**. Here the steel is subjected to the following temperature histories:

   - **Austenitize**: put in furnace at 850C in the austenite range, and hold for 1 hour until equilibrium temperature and corresponding solid solution structure have been reached.

   - **Furnace-Cool**: slowly cool in the furnace, from 850C to 700C, over a period of 10 hours.

   - **Air-cool**: remove from furnace and allow to air-cool to room temperature.

3. **Austenitize and Quench**:

   - **Austenitize**: put in furnace at 850C in the austenite range, and hold for 1 hour until equilibrium temperature and corresponding solid solution structure have been reached.

   - **Quench**: **Rapidly** remove material from furnace, plunge it into a large reservoir of water at ambient temperature, and **stir vigorously**.

     For 1045 steel, the quenching medium is water at ambient temperature (for other steels, other quenching media such as oil or brine are used).

4. **Austenitize, Quench, and Temper**:

   - **Austenitize**: put in furnace at 850C in the austenite range, and hold for 1 hour until equilibrium temperature has been reached.

   - **Quench**: **Rapidly** remove material from furnace, plunge it into a large reservoir of water at ambient temperature, and **stir vigorously**.

   - **Temper**: Re-heat the steel to to the **tempering temperature** (example: 250C), and hold for approximately 2 hours. Note: there is a range of possible tempering temperatures; for 1045 steel, this range is approximately from 200C to 500C. Different tempering temperatures lead to differences in the resulting mechanical properties; in general, 'lower' tempering temperatures lead to high yield strength, but lower toughness and ductility, while 'higher' tempering temperatures lower strength, but increase toughness and ductility.

   - **Air-cool**: remove from furnace and allow to air-cool to room temperature.

Each of these thermal histories produces unique microstructural conditions in the steel, and in turn, each microstructural state exhibits a unique combination of mechanical properties. We will perform the heat treatments, and measure the resulting mechanical properties.

Notes on successful quenching

We described the quenching process exclusively from the phenomenological/technological perspective, noting only that the process should be performed "rapidly" and that the part should be "stirred vigorously" in the quenching liquid. Left unaddressed were quantitative details of the resulting rapid temperature change: "what temperature[s] must the steel reach, and within what amount[s] of time, in order for the quench to be successful?" In order to provide a satisfactory answer to such a question, it is necessary to look briefly at the quenching process from the materials science perspective.

Referring again to Fig. 1, suppose that our 0.45 wt % carbon steel is cooled moderately fast through both the so-called A_3 temperature separating the γ and $\gamma + \alpha$ domains. (ca 870C for this carbon content) and the A_1 temperature (723 C) below which a microstructural 'composite' of $\alpha + Fe_3C$ becomes the equilibrium phase. At temperatures above A_3, the C was homogeneously distributed in solid solution γ, but Fe_3C is carbon-rich (6.67 wt %), while the α will hold increasingly less C as temperature decreases below A_1. Thus, there must be **diffusive mass transport** of C from surrounding regions toward the sites which will be occupied by the Fe_3C compound. This takes time; for slow cooling, there is ample time for transport, and equilibrium is achieved. But diffusion is also thermally-activated: it proceeds exponentially faster with increasing absolute temperature (rate $\propto e^{-Q/kT}$, where T is absolute temperature, k is Boltzmann's constant, and Q is activation energy for diffusion). If the temperature could be brought low enough, fast enough, so that negligible C transport had taken place, we would reach a meta-stable state in which BCC α crystals were "supersaturated" with 0.45 wt % C, more than 20 times its equilibrium solubility for C! Moreover, because the absolute temperature has been so dramatically reduced, the process of diffusive transport of C has also been effectively "shut down," so there is really no mechanism to spatially redistribute the C. The excess C would be kinetically 'trapped" in place. *What happens under such extreme deviations from equilibrium?*

Answer: the supersaturated BCC crystals undergo a spontaneous, diffusionless crystalllographic transformation to a less dense (i.e., the new lattice occupies more space than the BCC lattice did) body-centered tetragonal (BCT) crystal phase called **martensite**, retaining full carbon content locally. Martensite is **very hard** (and the higher its carbon content, the harder the martensite), and because it has rapidly formed *in situ*, it contains large internal stress; together, these features render "as-quenched" martensite very hard, but quite brittle. Subsequent tempering provides enough thermal driving force for limited diffusive atomic rearrangements that relieve internal stress and modify the

martensite. **Tempered martensites** are generally somewhat less hard, but much more tough, than as-quenched martensite.

Returning to the question motivating this section, now re-phrased in light of the just-discussed martensite reaction: "what temperature[s] must the steel reach, and within what amount[s] of time, in order for the quench to successfully produce martensite?" The answer lies in a so-called time-temperature-transformation (or "TTT" curve), a space of temperature versus time elapsed in a thermal transient. (see Fig. 2). Depending on the composition of the steel, various curves can be drawn, indicating a locus of the 'start' (subscript s) and 'finish' (subscript f) of various transformations in the cooling steel. One possible reaction product is called "**bainite**", which is a particular spatial arrangement of α and Fe_3C; another possible morphology, obtained from slower cooling histories, consists of alternating layers of α and Fe_3C collectively called **pearlite** (initially so-named because of its resemblance to mother-of-pearl). The left-pointing U-shaped curve labeled "B_s" ("bainite start") occupies the upper-left-most (high temperature; short time) position in the TTT curve. The lower left part of the TTT curve (low temperature; short time) has 2 constant-temperature (horizontal) lines labeled M_s (martensite start) and M_f (martensite finish).

> **In order for a quench to successfully produce martensite at a given location in the steel, it is necessary that the time versus temperature trajectory experienced at that location reach and cross the M_s and M_f lines without first intersecting the "nose" of the B_s curve.**

Otherwise, bainite production will commence before the hard martensite can be formed.

Finally, we have a basis for answering the question of what temperature, and within what time. In carbon steels, the nose of the B_s curve extends down to $\sim 540° C$ and a time of **only 1 second**! (Note log scale on time axis of Fig. 2.) Unless we can get the temperature below $\sim 540 C$ in less than one second, carbon steels such as 1045 will not produce all-martensite upon quenching. This constraint has important consequences:

- the maximum rate of heat transfer (and hence of cooling rate) achievable at the surface of the steel depends on the quenching medium (brine > water > oil), and increases with velocity of the fluid moving past the surface (aggressive stirring > no agitation).

- for large parts, the cooling rate in the interior is governed by heat conduction within the steel; no matter how aggressively the surface is quenched, the cooling rate in the center will be limited, and large carbon steel parts simply can not be successfully quenched to produce martensite in their interiors
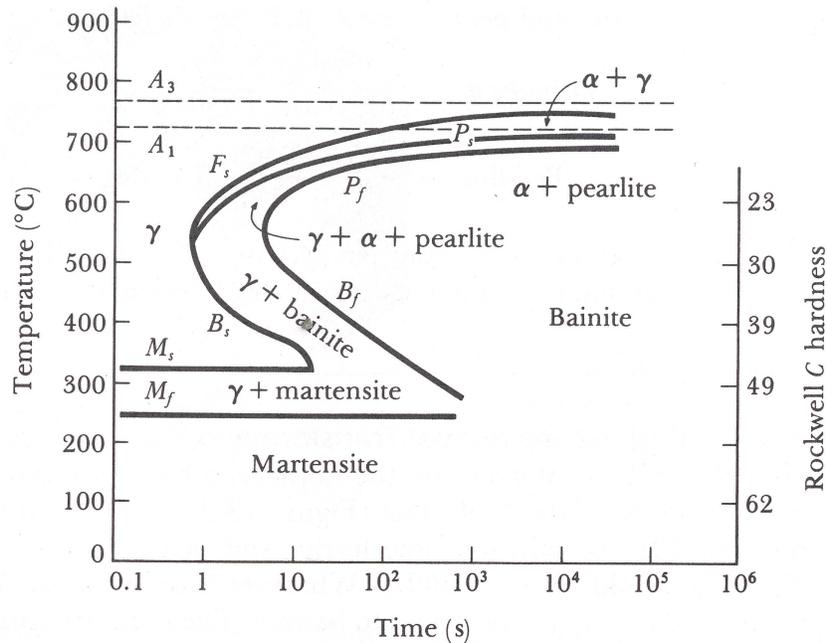
Figure 2: Time-Temperature-Transformation (TTT) curve for carbon steel AISI 1050.

This constraint on requisite cooling rate in 1045 has, at times, lead some student lab groups to obtain puzzling, or mixed, results from quenched or quenched-and-tempered specimens. The likely reason: insufficiently rapid cooling during the quench, leading to mixed microstructures. Such eventualities can be frustrating for everyone. For that reason, as a precaution, this term's lab is also looking at a low alloy steel, AISI 4140. As noted in section 1, this alloy has 0.4 wt % C, and alloying elements Cr, Mo, Si, and Mn.

These chemistry changes move the nose of the B_s curve to <u>much longer times</u>, of order tens of seconds, and to only slightly lower temperatures (say, 450 C).

Thus, even novice lab groups have no difficulty in quenching 4140 sufficiently fast to produce "all martensite"!

5 Hardness Testing

See also: Dowling, Section 4.7

This common testing mode consists of pressing a pointed diamond or hardened steel ball indenter into the surface of the material to be examined. The further into the material that the "indenter" sinks, the *softer* is the material, and the lower is its plastic flow strength.

The **hardness**, H, is the applied load, F, divided by the projected area of the indent into the surface, A:

$$H = \frac{F}{A}$$
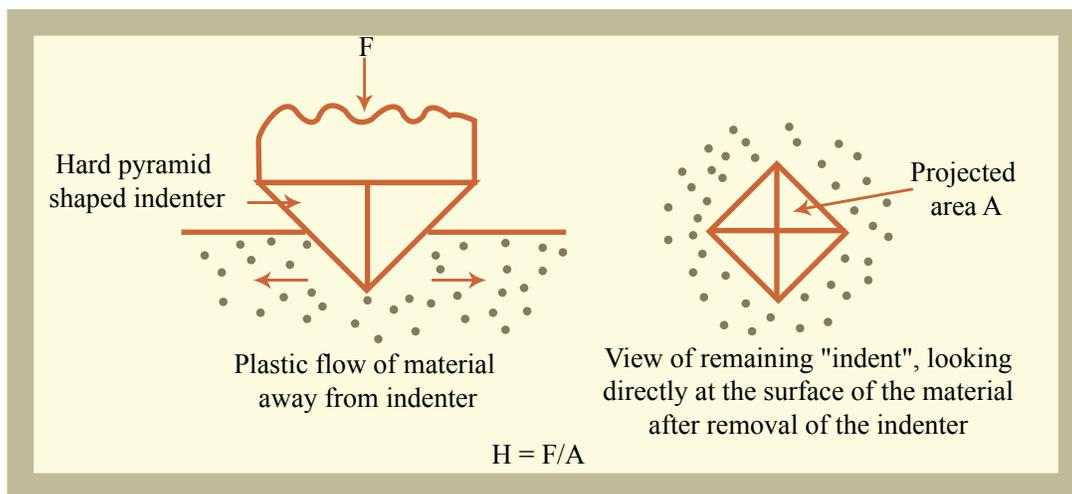<div align="right">5.1</div>

Figure 3: Schematic of a hardness test

For materials which do not appreciably strain-harden, it may be shown that the hardness H is approximately related to the tensile yield strength, σ_y, by

$$\boxed{H \approx 3\,\sigma_y}.$$
<div align="right">5.2</div>

However, suitable correction factors need to be added for materials which strain harden appreciably. Thus, the hardness test is a very quick and cheap **non-destructive test** for estimating σ_y. There is no need to go to the trouble and expense of making and testing tensile specimens, and the hardness indenter is so small that it scarcely damages the (surface of the) material. Hardness testing can be used as a quality assurance test to check if materials meet specifications on σ_y without damaging them.

The **Rockwell Hardness Test** is one commonly-used measure of hardness. Instead of separately measuring the force of indentation and the projected area of indent, this test simply measures the depth of indentation under different loads.

The Rockwell Hardness test employs several scales (see Table 1), each associated with a specific indentation load and indenter type. Depending on the expected hardness of the material being measured, the appropriate Rockwell Hardness scale is selected. Here the empirical measure of hardness on appropriate "scales" is correlated with the ultimate tensile strength, σ_{UTS}, of metals by performing an exhaustive series of experiments, Table 2. These correlations have been established mainly for martensitic and ferritic steels, and should not be applied to estimate tensile strengths of austenitic steels.

TABLE 1

COMMONLY-USED ROCKWELL HARDNESS SCALES

Symbol, HR'X' 'X' =	Penetrator Diameter if Ball, mm (in)	Load kg	Typical Application
A	Diamond point	60	Tool materials
D	Diamond point	100	Cast irons, sheet steels
C	Diamond point	150	Steels, hard cast irons, Ti alloys
B	1.588 (0.0625)	100	Soft steels, Cu and Al alloys
E	3.175 (0.125)	100	Al and Mg alloys, other soft metals; reinforced polymers
M	6.35 (0.250)	100	Very soft metals; high modulus polymers
R	12.70 (0.500)	60	Very soft metals; low modulus polymers

TABLE 2.

HARDNESS CONVERSION CHART
(HIGHER HARDNESS)

Brinell Hardness Tungsten Carbide Ball 3000 kg	Rockwell Hardness			Approximate Tensile Strength	
	A Scale 60kg	B Scale 100kg	C Scale 150kg	(ksi)	(MPa)
-	85.6	-	68.0	-	
-	85.3	-	67.5	-	
-	85.0	-	67.0	-	
767	84.7	-	66.4	-	
757	84.4	-	65.9	-	
745	84.1	-	65.3	-	
733	83.8	-	64.7	-	
722	83.4	-	64.0	-	
710	83.0	-	63.3	-	
698	82.6	-	62.5	-	
684	82.2	-	61.8	-	
682	82.2	-	61.7	-	
670	81.8	-	61.0	-	
656	81.3	-	60.1	-	
653	81.2	-	60.0	-	
647	81.1	-	59.7	-	
638	80.8	-	59.2	329	2267
630	80.6	-	58.8	324	2232
627	80.5	-	58.7	323	2225
601	79.8	-	57.3	309	2129
578	79.1	-	56.0	297	2046
555	78.4	-	54.7	285	1964
534	77.8	-	53.5	274	1888
514	76.9	-	52.1	263	1812
495	76.3	-	51.0	253	1743

TABLE 2. (Continued)

HARDNESS CONVERSION CHART
(INTERMEDIATE HARDNESS)

| Brinell Hardness Tungsten Carbide Ball 3000 kg | Rockwell Hardness | | | Approximate Tensile Strength | |
	A Scale 60kg	B Scale 100kg	C Scale 150kg	(ksi)	(MPa)
477	75.6	-	49.6	243	1674
461	74.9	-	48.5	235	1619
444	74.2	-	47.1	225	1550
429	73.4	-	45.7	217	1495
415	72.8	-	44.5	210	1447
401	72.0	-	43.1	202	1378
388	71.4	-	41.8	195	1343
375	70.6	-	40.4	188	1295
363	70.0	-	39.1	182	1254
352	69.3	-	37.9	176	1213
341	68.7	-	36.6	170	1171
331	68.1	-	35.5	166	1144
321	67.5	-	34.3	160	1102
311	66.9	-	33.1	155	1068
302	66.3	-	32.1	150	1033
293	65.7	-	30.9	145	999
285	65.3	-	29.9	141	971
277	64.6	-	28.8	137	944
269	64.1	-	27.6	133	916
262	63.6	-	26.6	129	889
255	63.0	-	25.4	126	868
248	62.5	-	24.2	122	840
241	61.8	100.0	22.8	118	813

TABLE 2. (Continued)

HARDNESS CONVERSION CHART
(LOWER HARDNESS)

Brinell Hardness Tungsten Carbide Ball 3000 kg	Rockwell Hardness			Approximate Tensile Strength	
	A Scale 60kg	B Scale 100kg	C Scale 150kg	(ksi)	(MPa)
235	61.4	99.0	21.7	115	792
229	60.8	98.2	20.5	111	765
223	-	97.3	20.0	109	751
217	-	96.4	18.0	105	723
212	-	95.5	17.0	102	703
207	-	94.6	16.0	100	689
201	-	93.8	15.0	98	675
197	-	92.8	-	95	655
192	-	91.9	-	93	641
187	-	90.7	-	90	620
183	-	90.0	-	89	613
179	-	89.0	-	87	599
174	-	87.8	-	85	586
170	-	86.8	-	83	572
167	-	86.0	-	81	558
163	-	85.0	-	79	544
156	-	82.9	-	76	523
149	-	80.8	-	73	503
143	-	78.7	-	71	489
137	-	76.4	-	67	461
131	-	74.0	-	65	448
126	-	72.0	-	63	434
121	-	69.8	-	60	413
116	-	67.6	-	58	400
111	-	65.7	-	56	386

6   Notched-Bar Impact Tests

See also: Dowling, Section 4.8

Notched-bar impact tests measure the work done (or energy dissipated) required to fail a specimen containing a stress concentration – the notch, under high strain-rate loading conditions, at different temperatures. The **Charpy Impact Test** is one common notched-bar impact test. In the Charpy test, a notched bar is subjected to three-point bending under impact loading conditions, Figure 4, and the energy to break the specimen is measured. Both U-notch and V-notch configurations are employed; the V-notch is used more frequently since it provides a more severe stress concentration. In this lab, we will use U-notched bars to assess the effects of differing heat-treatments of 1045 steel on the Charpy Impact Energy.
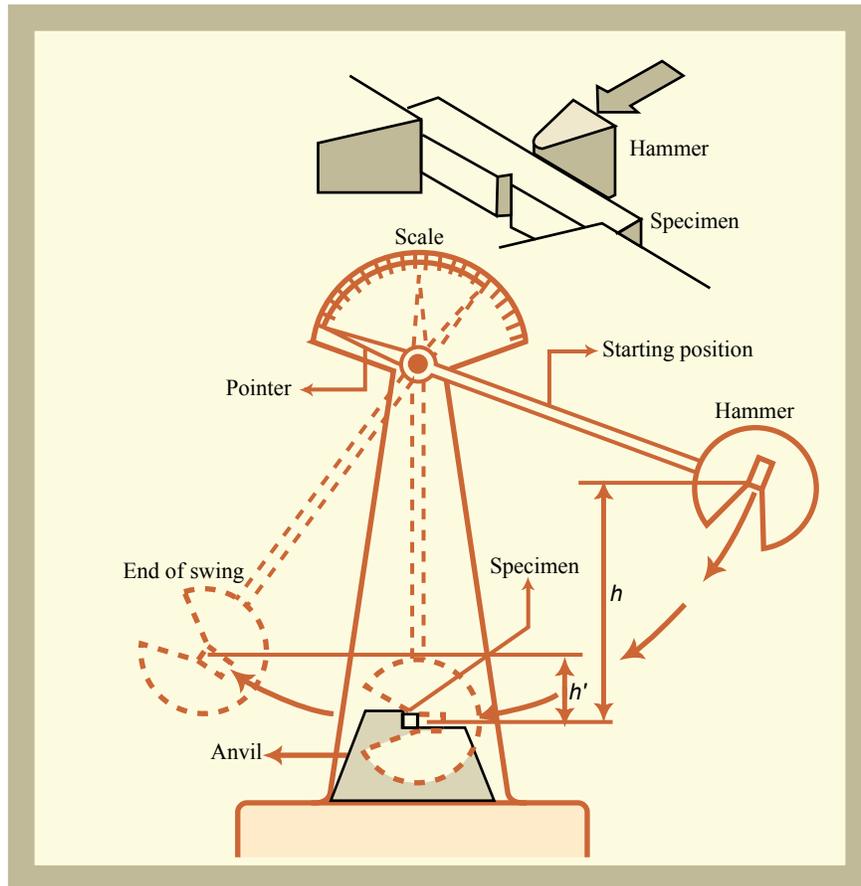
Geometry of Charpy impact test.

Figure 4: Charpy impact testing machine, and detail of three-point bending specimen and fixture, prior to impact.

The impact loading conditions are achieved by a swinging pendulum, see Figure 4.

1. The pendulum of standard mass is raised above the specimen; the pendulum height (and thus its potential energy) is recorded.

2. The pendulum is then released, the weight swings through and strikes the specimen in three-point bending; the (maximum) pendulum height after breaking the specimen is then recorded.

3. The difference between the initial and final potential energies is the energy required to break the specimen.

Note that this "loss" of energy represents a simple accounting that encompasses many phenomena, including

1. energy dissipated in the plastic deformation of the specimen,

2. the energy loss in driving the local fracture processes at the notch root,

3. frictional losses as the specimen slides over the supports,

4. kinetic energy acquired by the specimen,

5. acoustic energy lost (you can hear the impact), etc.

Nonetheless, provided standard-sized and -shaped specimens are used, the quantitative differences in impact energy are provide important **qualitative** indicators of the expected relative toughness (or brittleness) of the material, in the metallurgical condition tested, during potential service in an engineering product at test temperature, under high loading rates, and in the presence of stress concentration.

Indeed, the Charpy impact test has long been used in both alloy development studies, as well as being incorporated into many materials test specifications.