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HEAT-TREATING OF MATERIALS

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PREREQUISITE KNOWLEDGE: Typical high-school students of math and science should understand basic concepts of crystals (solids) and glasses (super-cooled liquids) for elements (atoms) and compounds (molecules).

OBJECTIVES: To introduce those solid-state transformations of material structures, known as "heat-treatments".[1] To emphasize commercial importance of common processes for metals as well as non-metals.

EQUIPMENT & SUPPLIES: Furnace, torch, or burner (electric[2] or gas) for red-hot and re-heated specimens. Buckets (2-5 gallon capacity, say 9-22 litres) for water-oil-sand quenches. Grating (expanded-metal, wire-mesh) and firebricks (supports) for cooling-table. Stop-watch (or timer) and thermometer (dial or glass). Tongs, gloves, and safety glasses. Hammer, wrench, hack-saw, vise. Specimens of metals (ferrous and non-ferrous alloys) or non-metals (ceramics and polymers) from ware-house or scrap-pile.

PAUSE & PONDER: What is the best way to learn a new topic? Start with simple, familiar examples? Survey a broad field? Why not do both?[3] First, remember the blacksmith's dipping red-hot steel in cold-water, because "quenching" is a quick way to allotropically harden carbon-steel.[4] More broadly, define conventional heat-treating as: "controlled heating and cooling of metal alloys, to alter properties, in the solid state". Why order "special heats" from melting furnaces, needing long-term scheduling of large-tonnage orders in high-energy processes at high prices? Solid-state treatments are sooner performed on standard stock in small amounts, at market prices with fuel savings, available from local suppliers.

More specifically, define "heat treating" as the controlled heating and cooling of metal alloys in the solid-state. Start by "solutionizing", heating above a critical temperature and soaking 1-hour per inch, to get a single-phase solid-solution. Then, cool at various rates to anneal (slow), normalize (medium), or quench (fast). "Tempering" is merely re-heating, so as to draw brittleness from allotropically-quenched martensite, to graphitize malleable-iron from white cast-iron, or to age a T-6 aluminum or P/H stainless-steel fron quenched super-saturated solutions. "Hardening", the usual objective, may involve working-alloying-carburizing-coating-quenching-or tempering operations.[5]

STEEL EXPERIMENTS: There are dozens of separate and distinct heat-treating operations that may be performed on steel. In this laboratory exercise, only four of the commonly used heat-treating operations will be performed, with explanations of their purpose and effects upon steel properties.[6]

(a) Anneal - A part is heated to the proper temperature, where the structure becomes a single solid-solution of "austenite". After a suitable holding time at temperature, the part is slowly cooled, in hot-sand or furnace. This puts the steel in a condition having its lowest strength and softest properties. It is usually the best condition for machining - or for cold forming the material into parts. In slow cooling from red-hot state, austenite transforms into a predominately large-grain, lamellar-pearlite, micro-structure.

(b) Normalize - Has the same austenitic-temperature as for annealing; but, after a suitable holding time at temperature, the part is removed from the furnace and allowed to cool in still-air. This operation is less costly than anneal, produces slightly higher strength and hardness, but often is adequate for machining and cold-forming.

This heat-treatment may provide a uniform metallurgical structure, throughout the entire part. The slightly faster cooling of austenite from the normalizing temperature still produces a predominately pearlitic micro-structure; but, it is somewhat finer lamellar-structure, which contributes to slightly higher hardness and strength.

(c) Quench or Harden - A part is also heated to the proper temperature, where the structure becomes a single solid-solution of austenite. It is also held at temperature for a suitable time, to provide a homogenous solid-solution. For hardening, the cooling rate is all-important. The heated part must be cooled so rapidly that austenite does not expel carbon, nor become pearlite, but transforms into a wholly new constituent called "martensite". To do this, the rate of cooling must exceed a critical cooling rate (CCR) at critical cooling velocity (CCV).

Martensite is an interstitial, super-saturated, solid-solution of carbon in tetragonal body-centered iron. The "diffusionless" transformation from austenite-to-martensite results in a volume expansion of 7 to 9%. It is the hardest and strongest condition for a given grade of steel, say 60-63 Rockwell-C(R_c). However, in this condition it lacks toughness and is seldom used in the as-quenched condition. Additionally, in this hard expanded-volume condition, there frequently are high internal-stresses, which may result in cracking or shattering, depending upon the shape of the part. The next heat-treatment puts martensite into a suitable condition, for engineering applications.

d) Temper or Draw - This relatively low-temperature operation reduces the hardness and strength of martensite, but improves toughness, for a specified application. Temperature is between 1350-400^{0}F (730-205^{0}C), below the eutectoid temperature of the "Iron-Carbon" equilibrium diagram. For each part and application, the exact temperature for tempering is variable to provide a finished part with proper combinations of hardness, strength and toughness.[7] Since these changes in micro-structure are extremely difficult to evaluate, by viewing the changed structure, specify that structure as tempered-martensite of a certain Rockwell hardness.

Laboratory Exercise:
Heat treat 5-samples, all of the same grade of steel (say AISI #1040).

(a) Annealing - Heat sample to 1650^{0}F, hold for 1-hour per inch at temperature, shut off furnace, and allow sample to cool in furnace. Check and record hardness, using R_c-scale.
(b) Normalizing - Heat sample to 1650^0 F, hold for 1-hour at temperature, remove sample from furnace, and cool in still-air on a metal-grating. Check and record R_c.
(c) Hardening - Heat sample to 1650^{0}F, hold for 1-hour at temperature. Remove sample quickly from furnace, plunge in cold water and agitate during the cooling, to get an effective quench without insulating steam bubbles. Check and record R_c.

(d) Tempering - After recording the hardness of quenched sample (c) above, place in furnace and temper at 900° F. Hold for 1-hour at temperature. Remove from furnace and air-cool. Check and record R_c.

(e) Drawing - Heat sample to 1650°F, hold 1-hour, then quench 1" of the end. File off black mill-scale, watch for blue-oxide color being "drawn" to silvery tip, and then quench to stop re-heat. Check and record R_c.

ALUMINUM EXPERIMENTS:

Several metal alloys (mainly non-ferrous) can be hardened and strengthened by a 2-step process:
a) Solution Heat-Treating involves heating-soaking-quenching.
b) Precipitation Hardening includes natural aging (air) or artificially-accelerated aging (oven).

Wrought Al. Alloys - The "Aluminum Association" has a 4-digit system to designate various structural materials, from #1000-7000, plus tempers (F, O, H, W, T) followed by 1 or more digits. Hardening mainly involves the 2000, 6000, and 7000 series. Cast Al. Alloys are similar, but designated with 3-digits, a decimal point, and 4th digit.

Commonly alloyed with Aluminum are Cu, Mg and Si, whose "solid-solubilities" are greater at hi-temp (vs. room). Thus, an alloy is heated to maximum-solubility, then quenched (rapidly cooled) to become temporarily a super-saturated solid-solution at room temperature; i.e., the solution has dissolved more alloy-element than normal for equilibrium conditions. Such state can be maintained (or prolonged) by refrigerated storage. However, if allowed to "age" naturally (at room temp.) or "artificially" (at oven temp.), the alloy can precipitate small crystals of "inter-metallic compound". Visible only by electron-microscope (S.E.M.), these particles act as "keys" at inter-granular boundaries and slip-planes. The product is harder and stronger.

Select AA alloy #6061-T6 (formerly #61S), composed of Al-Mg-Si, commercially heat-treated and formed into bar-stock, 1.5" dia (say 4 cm). Cut samples approx. 1" long (2.5 cm). Demonstrate basic processes, such as:

a) Full Anneal - To insure all samples start with uniform softness, preheat the furnace to 532°C for 1-hr; then heat samples for 1-hour, shut furnace off, and let samples slowly cool to room temp. (about 12-hrs inside furnace). Measure hardness.
b) Normalize - Remove solutionized sample from 532°C furnace; let cool on grating in still-air to room temp. (about 23°C). Measure hardness.
c) Solution Heat-Treating - Samples heated to 532°C and soaked 1-hr.; then fast-quenched in water to room temp., and immediately tested for hardness.
d) Natural Aging - Store quenched samples at room temp., and regularly monitor the increasing hardness. Check with references for AA#6061-alloy to estimate the optimal-aging period (6-12 months) at room temp.
e) Artificial Aging - Three quenched samples of known hardness (from Test "c") are reheated (tempered) to 200°C for 20-40-60 minutes; then "quench" in water to room temp. and measure hardness. Check ASM Metals Handbook (Ref. 5, Vol. II) for optimal time, hardness and strength. Rockwell may exceed 50 R_c.

TIPS: Ferrous alloys (Fe over 50 w/o) are typically used for "allotropic quenching"; however, consider an "alpha-beta" titanium alloy (Ti 6 Al - 4 V) for precipitation-hardening (solutionize at 950°C, quench in water, age at 540°C). Common allotropic-metals include: Co, Cr, Mn, Ni, Sb, Sn, Ti, W, Zr.

Always define "percentages"; e.g., w/o by weight, v/o by volume. Always check hardness (or strength) of fresh (as-is) specimens, to establish a "bench-mark" (control standard) before heat-treatment.

Samples are usually steel (AISI #1010 - 1080; 4340) or aluminum (AA #6061; 2024) sliced from 1-1/2 to 2" dia. bars (say 3-5 cm). However, scrap-metals are cheap and disposable: springs, paper-clips, hacksaw blades, razor blades, street-sweeper tines, pallet-straps. For typical "soaking" (1-hr per in, 2.54 cm) a thin specimen (1/16", 1.5 mm) needs only 4-5 minutes (depending on reflectivity) to "solutionize" in the solid-state.. Surface sanding is avoided, unless micro-hardness (Vickers, Knoop) is tested.

To show property differences (from various treatments), the usual tests are tensile-pulls or macro-dents, which require descaled ends (coupons) or surfaces (disks, blocks). However, consider "Mohs" scratching (sawing or filing) or a drop-weight (Brinell-ball through glass-tube) with Brinell-scope (20x).

Magnetic tests are appropriate. Use a magnetized screwdriver, file-cabinet clip, or recipe-holder to show "ferro-magnetism" of steel, nickel, or cobalt specimens. Austenitic stainless-steel is usually not magnetized (contaminated welds may become ferro-magnetic). Black mill-scale (Fe_3O_4) is strongly "ferro-magnetic"; so, cleanup around hot-rolling mill, heat-treating furnace, or blacksmith-shop is easy. Of course, red-hot "austenite" (gamma iron) is <u>not</u> ferro-magnetic.

With small metal-barstock, twisting tests are fast and effective. Mount a heat-treated bar (square or hexagonal section) in a vise, and twist 90° by wrench (torque-wrench is better). Note differences in "stiffness" between the annealed, normalized, quenched, tempered specimens. Note the 90° return-twist is stiffer (annealed, normalized bars) or makes cracks (quenched, tempered bars) and gives higher torque-readings (work-hardening). Such testing also averts descaling or smoothing steps.

Tines, lying in the street (after curb-sweeping) are hi-grade spring steel. Fresh samples are available from maintenance shops (after brush-change). After heat-treating, each tine can be vise-gripped and sharply-flexed (90° to and fro) from vertical. A slight pull is needed, to concentrate bending at one spot (as metal work-hardens). Annealed tines may take 20-flexes, tempered "as-is" tines take 3, and quenched tines only 1/2 flex. More tests are done, in less time.

Furnaces are often too costly, bulky, hot, slow, and unreliable. Our labs, for 10-20 yrs., have successfully used dual-box units (440^v oven, 110^v control) with inter-changeable base (temp. range) and bench size (2'x 2' x 3' clearance)[2]. However, with thin samples, a gas-welding torch or bunsen-burner is adequate for demonstrations. Cooling in a furnace (slowest), then in air (slow), one might assume "sand-burial" to be moderate (slower than air). Students are surprised to find cold-sand (room temp.) gives a mild "quench" and more hardness than air-normalizing. So, <u>heated</u> sand (100-200°C) is needed, to better approach a "full-anneal"

Moreover, students are surprised, when "tool-steel" is hardened by an "air-quench", because hi-alloy content slows the transformation-cooling rate and avoids "normalizing."

Plastic sheets, such as poly-carbonate (G.E. Lexan) or Acrylic PMM (DuPont Lucite, or Rohm & Haas Plexiglas) can be "stress-relieved" at low-temperatures (say 100-400°C, plus 4-day slow-cooldown). Examine a tensile-coupon by polariscope to check results (no colored fringes, in unstressed photo-elasticity specimens). Melt-and-burn tests are also worth exploring, with common "ethenic" plastics.[8]

CONCLUSION:

With multiple lab-sections and repetitious testing, the materials lab has the opportunity of accumulating data (for several semesters) to run linear regressions, get dimensionless plots, or make a statistical analysis-of-variances. Studying trend-lines and correlation-coefficients will enhance comparisons:

a) Hardening vs. softening processes.
b) Cooling efficiency of quenchants (water, oil, sand).
c) Carbon content of steels.
d) Ferrous vs. nonferrous alloys.
e) Metals vs. non-metals.
f) Cooling efficiency of water-oil-sand quenchants.

Virtually all materials (metals, ceramics, polymers) are being commercially heat-treated. Materials lab experiments can expand beyond metallurgy into ceramic-glass tempering, refractory curing, thermoplastics manufacturing, thermo-setting, composites fabricating, repairing and maintaining.

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