Table 2 .... Hardness and Retained Austenite Content of Hardened and Cold Treated Tool Steel[2]

Tool Steel	Hardening Treatment Temp, F	As-hardened		Cooled to −320 F	
		Rockwell hardness	% Retained austenite	Rockwell hardness	% Retained austenite
Plain carbon (1% C)	1450, water	C66	9	C67	3
Ball bearing (1% C, 1.5% Cr)	1550, oil	C64	7	C65	2
Tungsten die (1.2% C, 1.5% W)	1600, oil	C62.5	14	C63	7
Manganese die (1% C, 1.2% Mn)	1450, oil	C64.5	9	C67.5	6
M-2 high speed (0.8% C, 6% W, 5% Mo, 4% Cr, 1.5% V)	2220, oil	C65	20	C66	8
Tungsten high speed (0.8% C, 18% W, 4% Cr, 1% V)	2350, oil	C66	20	C67	8
High carbon, high chromium die (1.5% C, 12% Cr, 1% Mo, 1% V)	1900, air	C64.5	25	C66	15
Chromium die (1% C, 5% Cr)	1775, air	C65	33	C68	6

sult from thermal stresses caused by the localized heating action of the grinding wheel. This condition is further aggravated by the expansion of retained austenite transforming thermally or by the mechanical shock of the wheel causing conversion to martensite with severe surface stresses.

Tool steels which may show marked improvement by cold treatment are those high in carbon and alloy content such as high speed, high-carbon high-chrome, 1 percent carbon-5 percent chromium, and tungsten punch. A comparison of hardness and retained austenite content of several tool steels after cooling to −320 F is shown in Table 2.

If high speed steel is properly hardened and then tempered at 1050 F for 2½ hr, very little retained austenite should remain in the structure. With the usual addition of 2 or more multiple tempering cycles at 1050 F, negligible retained austenite should remain in the structure to warrant cold treatment. However, cold treatment may serve to reduce the number of required multiple tempering cycles. When the as-quenched retained austenite content is greater than normal, cold treatment may be more effective than multiple tempering. This condition may result from high quenching temperature, surface carburization, interrupted quenching, or the high alloy content in special steels. Increased hardness and cutting performance have been noted after cold treatment and retempering when applied to stock high speed tools. This improvement might be explained by the transformation of retained austenite formed during severe grinding of the cutting edge causing rehardening of a thin surface layer.

High-carbon high-chrome (1.5 percent carbon, 12 to 14 percent chromium, 1 percent molybdenum), 1 percent carbon, 5 percent chromium, 1 percent molybdenum die steels respond very well to cold treatment. Multiple cycling of high-carbon high-chrome steels between −120 F and 950 F produces greater stability than is possible by secondary hardening alone in the temper at 950 F; however the final hardness may be less than with the conventional quench and temper process. When these steels are tempered at 400 to 500 F, an increase in hardness is noted over the conventional treatment. Rockwell hardness inspection should never be made prior to a 300 F stress relief since there is a hazard of incipient cracks forming from the penetrator impression.

Wear resistance can be increased in the 1 percent carbon, 5 percent chromium steel by cooling to −150 F prior to a 400 F temper. This is particularly effective whenever the surface carbon content is increased by pack hardening in carburizing compound. This technique of carburizing certain tool steels to improve surface wear resistance is also used with the 0.5 percent carbon, 1–2 percent tungsten, 1 percent chromium punch and die steel.

Hardenable magnet steels, such as the 1 percent carbon types with either 3½ percent chromium, 3 percent cobalt or 5 percent tungsten, tend to retain austenite in the hardening process. This non-magnetic austenite may be converted to magnetic martensite by cooling to −150 F as a continuation of the quenching cycle with a resultant increase of about 5 percent martensite.

Salvage of out-of-size tools and dies is sometimes possible by cold treatment to compensate for shrinkage in heat treatment or dimensional errors in machining of close tolerance work. The expansion in volume gained by conversion of possible retained austenite will be slightly offset by shrinkage in the subsequent tempering of the freshly formed martensite.

MECHANISM OF PRECIPITATION HARDENING

Certain alloys may be hardened by a mechanism known as precipitation or age hardening rather than by allotropic transformation as in steel. This mechanism depends on the decreasing solubility in the solid state of solute atoms dissolved in the crystal lattice of a solvent solid solution. The heat treatment of such alloys consists of: (1) a solution heat treatment wherein complete solution of solute atoms takes place, (2) a rapid quench, usually in water, which produces a soft supersaturated solid solution at room temperature and (3) a precipitation heat treatment which tends to permit solute atoms to aggregate and coalesce into particles of the precipitation phase. The term *precipitation* is misleading in that maximum hardness and strength are achieved in this operation before any visible particles of the second phase are observed in the microstructure. It is believed that the strengthening of the crystal lattice occurs in a pre-precipitation stage of the process possibly caused by reorganization or distortion within the crystal lattice. With higher precipitating temperature, or increased time at temperature, coalescence and growth of the precipitating phase occurs to produce an overaged structure with loss in strength and hardness.

Those alloys which precipitate at normal atmospheric temperatures and hence do not require precipitation heat treatment for hardening, are referred to as natural age hardening alloys. They are normally furnished in the solution heat treated or annealed condition and are fabricated before natural aging occurs. With some alloys the time element is such that storage after the solution treatment would permit hardening to take place prior to fabrication unless steps were taken to delay the aging phenomenon. Cold treatment has been very effective in slowing down the aging rate in such alloys.

Aluminum alloy rivets made from 2017 (17S) and 2024 (24S) aluminum will normally start to age at 32 F within 16 hr after the solution treatment. They may be stored about a week at 0 F and held indefinitely at −40 F without hardening taking place until after the riveting operation. A recent trend has extended the use of cold treatment to solution treated castings, forgings and sheet metal blanks for storage prior to cold working operations. To promote dimensional stability and better machined finishes in precipitation hardenable aluminum and magnesium castings, a combination of cold treatment at −140 F followed by overaging 50 F above the aging temperature has been employed. This allows permanent growth to take place prior to the final dimensioning of precision parts and minimizes possible service failures caused by temperature fluctuations over the same range of temperature.

Modifications of the 17-7 type austenitic stainless steels have been developed recently to satisfy aircraft and missile operating temperatures up to 800 F. These semiaustenitic steels are made hardenable by cold treatment at −100 F to form martensite or by a combination of cold treatment at −100 F followed by precipitation treatment in the range 750 to 1050 F.

PROCESSES INVOLVING PHYSICAL CHANGE

The preceding discussion has covered cold treating applications involving permanent structural change in metals. Other commercial applications of cold treatment involve reversible physical change where temperature is the controlling factor. Such processes include expansion fitting, shrink disassembly, and a unique application employing frozen mercury for precision casting patterns.

Expansion fitting differs from shrink fitting in that an internal part is thermally contracted by cold treatment, inserted into a mating part, and allowed to expand to a tight fit upon returning to atmospheric temperature. Shrink fitting on the other hand involves the heating of the external part with subsequent shrinkage when assembled and allowed to cool around an inside part. A combination of both expansion and shrink fitting may be utilized when greater assembly strength is required. Shrink disassembly of bushings, sleeves and hollow inserts is possible if a suitable refrigerated coolant can be allowed to flow through the internal part.

The use of cold treatment in the assembly of parts offers a definite advantage in ease of operation with freedom from oxidation, distortion and softening caused by tempering of hardened parts when heated for shrink fitting. A disadvantage of the process is the limited size change found possible when cooling with liquid nitrogen to −320 F compared to the thermal expansion resulting from heating through several hundred degrees.

In the Mercast process (developed by Mercast Corp.), cold treatment is employed to control the phase change between liquid and solid mercury used as replica material in place of the wax or plastic employed in the familiar *lost wax* or precision investment casting process. Mercury is rapidly frozen in a master mold by cooling to −100 F, removed as a solid replica of the part, and then held below −40 F during the investment proccess.

METHODS OF COLD TREATMENT

Temperatures below −150 F are seldom required for commercial cold treatment of metals except in connection with expansion fitting. In fact, a temperature of −120 F is generally satisfactory except where maximum dimensional stability warrants the higher cost of lower temperature intallation and operation. The various means of producing sub-atmospheric temperatures and the temperatures obtainable are:

Salt and ice	− 6 F
Mechanical refrigeration, single stage	− 50
Solid CO_2 and methyl alcohol or acetone	− 100
Solid CO_2	− 109
Mechanical refrigeration, multiple stage	− 150
Liquid air	− 314
Liquid nitrogen	− 320
Liquid helium	− 458

Although solid CO_2 and alcohol or acetone mixtures require a relatively small initial investment, continued use on a production basis is inconvenient and costly. Mechanical refrigeration provides the best means of meeting variable production demands. Such units are generally operated continuously at the desired temperature. Both liquid baths and circulated air chambers are used for cooling parts, however, the rapid heat transfer in the refrigerated liquid bath is somewhat offset by the greater tendency toward cracking. Liquid nitrogen has limited commercial use except in special applications that warrant the relatively high cost.

In cold treatment sufficient time must be allowed for parts to come to uniform thermal equilibrium at the temperature of the cooling medium. The following times were required to cool machined steel cylinders, four inches in length, from an ambient temperature of 75 F down to an alcohol bath temperature of −100 F as measured by thermocouples embedded at the middle of the bar:

Diameter, In.	Time, Min.
1	9
1½	14
2	16
2½	20
3	26
4	38

It will be recalled that in those cold treating and stabilizing processes involving conversion of retained austenite to martensite, transformation takes place with decreasing temperature and is not a function of time at temperature.

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