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Cold and Cryogenic Treatment of Steel

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COLD TREATNG OF STEEL is widely accepted within the metallurgical profession as a supplemental treatment that can be used to enhance the transformation of austenite to martensite and to improve stress relief of castings and machined parts. Common practice identifies –84 °C (–120 °F) as the optimal temperature for cold treatment. There is evidence, however, that cryogenic treatment of steel (also referred to as deep cryogenic treatment, or DCT), in which material is brought to a temperature on the order of –184 °C (–300 °F), improves certain properties beyond the improvement attained at cold treatment temperatures. This discussion explains the practices employed in the cold treatment of steel and presents some of the results of using cryogenic treatment to enhance steel properties.

Cold Treatment of Steel

Cold treatment of steel consists of exposing the ferrous material to subzero temperatures to either impart or enhance specific conditions or properties of the material. Increased strength, greater dimensional or microstructural stability, improved wear resistance, and relief of residual stress are among the benefits of the cold treatment of steel. Generally, one hour of cold treatment for each 2.54 cm (1 in.) of cross section is adequate to achieve the desired results.

All hardened steels are improved by a proper subzero treatment to the extent that there will be less tendency to develop grinding cracks and therefore they will grind much more easily after the elimination of the retained austenite and the untempered martensite.

Hardening and Retained Austenite

Whenever hardening is to be done during heat treating, complete transformation from austenite to martensite generally is desired prior to tempering. From a practical standpoint, however, conditions vary widely, and 100% transformation rarely, if ever, occurs. Cold treating may be useful in many instances for improving the percentage of transformation and thus for enhancing properties.

During hardening, martensite develops as a continuous process from start (M_s) to finish (M_f) through the martensite formation range. Except in a few highly alloyed steels, martensite starts to form at well above room temperature. In many instances, transformation essentially is complete at room temperature. Retained austenite tends to be present in varying amounts, however, and when considered excessive for a particular application, must be transformed to martensite and then tempered.

Cold Treating versus Tempering. Immediate cold treating without delays at room temperature or at other temperatures during quenching offers the best opportunity for maximum transformation to martensite. In some instances, however, there is a risk that this will cause cracking of parts. Therefore, it is important to ensure that the grade of steel and the product design will tolerate immediate cold treating rather than immediate tempering. Some steels must be transferred to a tempering furnace when still warm to the touch to minimize the likelihood of cracking. Design features such as sharp corners and abrupt changes in section create stress concentrations and promote cracking.

In most instances, cold treating is not done before tempering. In several types of industrial applications, tempering is followed by deep freezing and retempering without delay. For example, such parts as gages, machineways, arbors, mandrils, cylinders, pistons, and ball and roller bearings are treated in this manner for dimensional stability. Multiple freeze-draw cycles are used for critical applications.

Cold treating also is used to improve wear resistance in such materials as tool steels, high-carbon martensitic stainless steels, and carburized alloy steels for applications in which the presence of retained austenite may result in excessive wear. Transformation in service may cause cracking and/or dimensional changes that can promote failure. In some instances, more than 50% retained austenite has been observed. In such cases, no delay in tempering after cold treatment is permitted, or cracking can develop readily.

Process Limitations. In some applications in which explicit amounts of retained austenite are considered beneficial, cold treating might be detrimental. Moreover, multiple tempering, rather than alternate freeze-temper cycling, generally is more practical for transforming retained austenite in high-speed and high-carbon/high-chromium steels.

Hardness Testing. Lower-than-expected Rockwell C hardness (HRC) readings may indicate excessive retained austenite. Significant increases in these readings as a result of cold treatment indicate conversion of austenite to martensite. Superficial hardness readings, such as HR15N, can show even more significant changes.

Precipitation-Hardening Steels. Specifications for precipitation-hardening steels may include a mandatory deep freeze after solution treatment and prior to aging.

Shrink Fits. Cooling the inner member of a complex part to below ambient temperature can be a useful way of providing an interference fit. Care must be taken, however, to avoid the brittle cracking that may develop when the inner member is made of heat treated steel with high amounts of retained austenite, which converts to martensite on subzero cooling.

Stress Relief

Residual stresses often contribute to part failure and frequently are the result of temperature changes that produce thermal expansion and phase changes, and consequently, volume changes.

Under normal conditions, temperature gradients produce nonuniform dimensional and volume changes. In castings, for example, compressive stresses develop in lower-volume areas, which cool first, and tensile stresses develop in areas of greater volume, which cool last. Shear stresses develop between the two areas. Even in large castings and machined parts of relatively uniform thickness, the surface cools first and the core last. In such cases, stresses develop as a result of the phase (volume) change between those layers that transform first and the center portion, which transforms last.

When both volume and phase changes occur in pieces of uneven cross section, normal contractions due to cooling are opposed by transformation expansion. The resulting residual stresses will remain until a means of relief is applied. This type of stress develops most frequently in steels during quenching. The surface becomes martensitic before the interior does. Although the inner austenite can be strained to match this surface change, subsequent interior expansions place the surface martensite under tension when the inner austenite transforms. Cracks in high-carbon steels arise from such stresses.

The use of cold treating has proved beneficial in stress relief of castings and machined parts of even or nonuniform cross section. Features of the treatment include:

- Transformation of all layers is accomplished when the material reaches –84 °C (–120 °F).
- The increase in volume of the outer martensite is counteracted somewhat by the initial contraction due to chilling.
- Rewarm time is controlled more easily than cooling time, allowing equipment flexibility.
- The expansion of the inner core due to transformation is balanced somewhat by the expansion of the outer shell.
- The chilled parts are handled more easily.
- The surface is unaffected by low temperature.
- Parts that contain various alloying elements and that are of different sizes and weights can be chilled simultaneously.

Advantages of Cold Treating

Unlike heat treating, which requires that temperature be controlled precisely to avoid reversal, successful transformation through cold treating depends only on the attainment of the minimum low temperature and is not affected by lower temperatures. As long as the material is chilled to –84 °C (–120 °F), transformation will occur; additional chilling will not cause reversal.

Time at Temperature. After thorough chilling, additional exposure has no adverse effect. In heat treating, holding time and temperature are critical. In cold treatment, materials of different compositions and of different configurations may be chilled at the same time, even though each may have a different high-temperature transformation point. Moreover, the warm-up rate of a chilled material is not critical as long as uniformity is maintained and large temperature gradient variations are avoided.

The cooling rate of a heated piece, however, has a definite influence on the end product. Formation of martensite during solution heat treating assumes immediate quenching to ensure that austenitic decomposition will not result in the formation of bainite and cementite. In large pieces comprising both thick and thin sections, not all areas will cool at the same rate. As a result, surface areas and thin sections may be highly martensitic, and the slower-cooling core may contain as much as 30 to 50% retained austenite. In addition to incomplete transformation, subsequent natural aging induces stress and also results in additional growth after machining. Aside from transformation, no other metallurgical change takes place as a result of chilling. The surface of the material needs no additional treatment. The use of heat frequently causes scale and other surface deformations that must be removed.

Equipment for Cold Treating

A simple home-type deep freezer can be used for transformation of austenite to martensite. Temperature will be approximately –18 °C (0 °F). In some instances, hardness tests can be used to determine if this type of cold treating will be helpful. Dry ice placed on top of the work in a closed, insulated container also is commonly used for cold treating. The dry ice surface temperature is –78 °C (–109 °F), but the chamber temperature normally is approximately –60 °C (–75 °F).

Mechanical refrigeration units with circulating air at approximately –87 °C (–125 °F) are commercially available. A typical unit has these dimensions and operational features:

- Chamber volume, up to 2.7 m^3 (95 ft^3)
- Temperature range, 5 to –95 °C (40 to –140 °F)
- Load capacity, 11.3 to 163 kg/h (25 to 360 lb/h)
- Thermal capacity, up to 8870 kJ/h (8400 Btu/h)

Although liquid nitrogen at –195 °C (–320 °F) may be employed, it is used less frequently than any of the previous methods because of its cost.

Cryogenic Treatment of Steels

Cryogenic treatment, also referred to as cryogenic processing, deep cryogenic processing, deep cryogenic treatment (DCT), cryogenic tempering, and deep cryogenic tempering, is a distinct process that uses extreme cold to modify the performance of materials. (The use of the word *tempering* is a misnomer, because this is not a tempering process.)

The process is differentiated from cold treatment by the use of lower temperatures, the presence of distinct time/temperature profiles, and its application to materials other than steel (Ref 1). Cryogenic treatment has been in existence only since the late 1930s, making it a relatively new and emerging process. The late development of the process is mainly due to the fact that cryogenic temperatures have been available in useful commercial quantities only since the early 1900s.

Cryogenic treatment can provide wear-resistance increases several times those created by cold treatment with hardened steels (Ref 2). The process is not confined to hardened steels, but also shows results with most metals, cemented carbides, and some plastics (Ref 3–5). Use of the process on metals other than steel produces similar affects as with steel. Results of the process include relief of residual stresses (Ref 6); reduced retained austenite (in hardened steel); the precipitation of fine carbides in ferrous metals (Ref 7, 8); and increased wear resistance, fatigue life, hardness, dimensional stability, thermal and electrical conductivity, and corrosion resistance (Ref 9).

What are cryogenic temperatures? The scientific community generally defines cryogenic temperatures as temperatures below −150 °C (−238 °F, or 123 K) (Ref 10). This, admittedly, is an artificial upper limit. Temperatures used presently in cryogenic treatment are generally –185 °C (–300 °F, or 89 K). These are temperatures easily reached with liquid nitrogen. Some work is being done with liquid helium at temperatures down to –268 °C (–450 °F, or approximately 6 K).

Cryogenic treatment was made easier to achieve and more successful by the development of microprocessor-based temperature controls in the 1960s and 1970s and by the pioneering research by Randall Barron of Louisiana Tech University. Research into the process has been accelerating. The Cryogenic Society of America maintains a database of peer reviewed research papers (Ref 11).

Cryogenic Treatment Cycles

One distinct difference from cold treatment is that cryogenic processing requires a slow drop in temperature in order to reap all benefits of the process. The ramp down in temperature usually is on the order of 0.25 to 0.5 °C/min (32.5 to 32.9 °F/min). The object of this slow ramp down is to avoid high-temperature gradients in the material that can create harmful stresses, and to allow time for the crystal lattice structure to accommodate the changes that are occurring.

Typical cryogenic treatment consists of a slow cool-down from ambient temperature to approximately –193 °C (–315 °F), where it is held for an appropriate time. Hold periods range from 4 to 48 h depending on the material. At the end of the hold period, the material is brought back to ambient temperature at a rate of approximately 2.5 °C/min (36.5 °F/min). The temperature-time plot for this cryogenic treatment cycle is shown in Fig. 1. By conducting the cool-down cycle in gaseous nitrogen, temperature can be controlled accurately, and thermal shock to the material is avoided. Single-cycle tempering usually is performed after cryogenic treatment to improve impact resistance, although double or triple tempering cycles sometimes are used.

It is worthy of note that most time-temperature profiles have been empirically developed. Some research is being done to optimize the profiles for individual steels. For instance, some research indicates the holding time for AISI

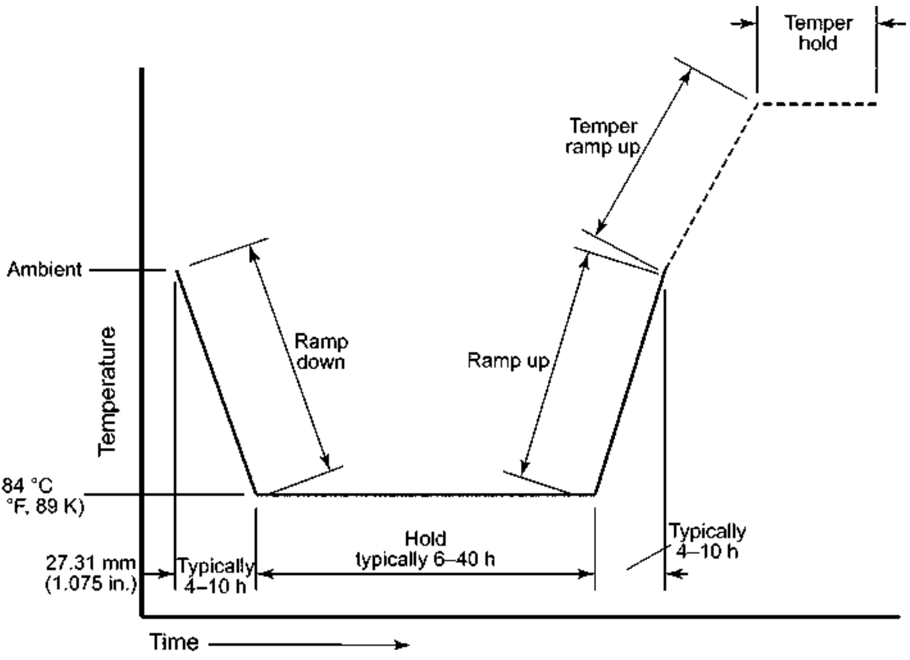

Fig. 1 Plot of temperature vs. time for the cryogenic treatment cycle. Tempering may or may not be necessary, depending on the material treated. Some materials require multiple tempering cycles. Some companies are now treating materials down to −268 °C (−450 °F, or 6 K)

T42 steel should not be longer than 8 h (Ref 12). In contrast, research indicates that the hold time should be 36 h for AISI D2 (Ref 13). This indicates there is much research to be done to optimize the process for all materials. Research into the optimal ramp down times, hold times, and ramp up times would maximize the effect of the process and minimize the time needed to accomplish its results.

There are several theories behind the effects of cryogenic treatment. One theory involves the more nearly complete transformation of retained austenite into martensite. This theory has been verified by x-ray diffraction measurements. Another theory is based on the strengthening of the material brought about by precipitation of submicroscopic carbides as a result of the cryogenic treatment (Ref 7, 8). Allied with this is the reduction in internal stresses in the martensite that happens when the submicroscopic carbide precipitation occurs. A reduction in microcracking tendencies resulting from reduced internal stresses also is suggested as a reason for improved properties. Studies also show reduction in residual stresses. Another theory postulates that the extreme cold reduces the free energy of the crystal structure and creates a more orderly structure. Another area to consider is the basic effect of cold on the crystal structure of metals. Point defects in the crystal structure are temperature dependent. Lowering the temperature of the crystal structure will cause the number of point defects in the crystal structure to change according to:

$$N_{\mathrm{d}} = N \exp(-E_{\mathrm{d}}/kT)$$

where N_{d} is the number of defects present, N is the total number of atomic sites, E_{d} is the activation energy needed to form the defect, k is the Boltzmann constant, and T is the absolute temperature. Reducing the temperature at a suitably slow rate drives the point defects out of the structure to the grain boundaries. In other words, the solubility of vacancies and other point defects in the matrix drops. This could account for some of the effects seen in DCT.

In the past, the absence of a clear-cut understanding of the mechanism by which cryogenic treatment improves performance had hampered its widespread acceptance by metallurgists. Some confusion has arisen from the fact that there are a number of different effects on metals, many of which cannot be seen in simple microstructural examination of the material with a light microscope. The lack of easily detected microstructural changes led many to discount the process. Another reason was the generally accepted belief that nothing happens to solid objects as the temperature drops. Extreme cold has been available on earth only for about 100 years. Understanding of materials science developed with the observation that heat changes properties. Much of the early research was centered around determining whether or not cryogenic treatment actually provided the advantages claimed. Because the early research and actual industry usage have proven the validity of the process, research now is turning to determine why the results are seen and how to maximize those results.

Uses of Deep Cryogenic Treatment

Deep cryogenic treatment is used in many ways to reduce wear. It is in common use to control distortion of metal objects, modify the vibrational characteristics of metals, increase fatigue life, reduce abrasive wear, and reduce electrical resistance. It is safe to say the applications for this process are extremely broad.

The process is in commercial use for high-speed steel (HSS) and carbide cutting tools, knives, blanking tools, forming tools, and more. Research as far back as 1973 indicates that deep cryogenic processing results in over three times life increase in end mills, 82 times life improvement in punches, over two times the life in thread dies, six times the life in copper resistance electrodes, six times the life in progressive dies, and over four to five times the life in broaches (Ref 2). Research estimates a 50% reduction in tooling costs with H13 and M2 steels that have been deep cryogenically treated (Ref 14).

Other studies have shown that DCT increases abrasion resistance of cast iron. Cast iron brake rotors consistently show a three to five times life increase when tested to SAE2707 brake dynamometers (Ref 15). This has been validated against real-world experience in passenger cars, racing cars, trucks, and mining vehicles.

Deep cryogenic treatment also has been proven to create a phase change in cemented carbide (Ref 5). A study by the National Aeronautics and Space Administration (NASA) proved the release of residual stresses in welded aluminum (Ref 6), and other studies prove increases in fatigue life in steel springs (Ref 16) and in load capacity of gears (Ref 17).

Deep cryogenic treatment is used in the automotive racing industry to increase life in virtually every engine component. Drive line components such as transmission and differential gears, suspension springs, torsion bars, axles, suspension members, and, of course, brakes are treated.

Deep cryogenic treatment also is in commercial use by musical instrument makers. Yamaha Wind Instruments has done extensive testing of DCT and offers the process on its wind instruments (Ref 18). There is much activity in the high-performance stereo industry in treating vacuum tubes, wire, power cords, vacuum tubes, transformers, connectors, and more.

Equipment for Cryogenic Treatment

All cryogenic treatment equipment is comprised of a thermally insulated container and some means of extracting the latent heat of the payload to reach the desired low temperature. In most cases the insulation is a solid material that contains small closed cells of trapped still air. The thermal conductivity of such insulation essentially is that of still, nonconvecting air, assuming that the solid material that encloses the air pockets is of thin cross section and low conductivity. \Examples are polyurethane foam, aerogel, and expanded glass foam. Fifteen centimeters (6 in.) of any of these will conduct approximately (15 Btu/h.ft^2) across a temperature differential of 204 °C (400 °F), which exists between the interior of a refrigerator at –195 °C (–320 °F) and ambient temperature of 26 °C (80 °F).

These solid insulating materials are relatively inexpensive and, in the case of foamed-in-place polyurethane, can readily fill irregularly shaped cavities. They all suffer from one important drawback: temperature cycling establishes a temperature gradient across the insulating slab that results in differential contraction in the material. Repeated temperature cycles ultimately result in fatigue cracking of the insulation. Energy expenditure to sustain the temperature difference goes up, and temperature uniformity within the refrigerator may deteriorate.

The use of vacuum insulation in cryoprocessor design avoids these problems. A vacuum insulated container consists of two concentric shells, usually cylindrical, separated by a small distance relative to their diameters, which are joined around the perimeter of one end of the shells. The space between the shells contains reflective insulation and is evacuated to a pressure of approximately 533 Pa (10^{-6} torr). This essentially eliminates heat flow by conduction and convection because most of the conducting or convecting gas has been removed. Heat gain via infrared radiation is minimized by multiple reflective layers placed in the vacuum space. Heat flow across a vacuum-insulated space, given a temperature difference across the walls of 204 °C (400 °F), is (0.008 Btu/h.ft^2), a factor of 1900 better than solid insulation of 15 cm (6 in.) thickness (Ref 19). The principle mode of heat transmission into the interior of a vacuum-insulated container is metallic conduction through the perimeter that joins the inner and outer shells.

In addition to providing a barrier to heat flow relative to solid insulation, the vacuum-insulated vessel is immune to thermal cycling fatigue. Additionally, the vacuum-insulated vessel can sustain elevated operating temperatures far in excess of that permissible with the use of polyurethane. This permits the post-refrigeration tempering of components in one device, eliminating the need for a separate tempering oven.

Heat extraction from the payload is effected by the phase change of a low-boiling-point fluid. If mechanical refrigeration is used, a high-pressure fluid is allowed to expand and become a gas within an evaporator coil inside the insulated space. The evaporator coil is a heat exchanger that absorbs heat from the payload via convection, natural or forced, within the chamber. This ensures the relatively slow cooling of the payload and avoids thermal shock resulting from too rapid cooling. Rapid cooling can cause shrinkage of the outside of the cooled component while the relatively warm interior does not shrink. Tensile stress induced this way can lead to cracking or the initiation of residual stress, especially at sharp edges. Reaching cryogenic temperatures by mechanical refrigeration for industrial size payloads requires multistage refrigeration. These are very expensive machines to build and maintain.

Fortunately, liquid nitrogen is abundant, readily available, and relatively inexpensive. It has a boiling point of –196 °C (–321 °F) and a heat of vaporization of approximately 150 Btu/liter. It is produced in huge industrial gas production facilities and delivered to the facility where the expansion and phase change occurs, free of the capital and maintenance expense demanded by in-house mechanical refrigerators.

Two other approaches have been tried but have difficulties: a hybrid of mechanical refrigeration and LN$_2$ (liquid nitrogen) cooling, and a controlled immersion of components into LN$_2$.

The hybrid approach uses mechanical refrigeration to do an initial cooling of the payload to some sub-atmospheric temperature that is well above the desired cryogenic range. At that point a spray of LN$_2$ droplets is showered onto the payload to bring the temperature down to the desired point. Unless the mechanical refrigeration has sufficient Btu removal rate, the payload will be substantially warmer than indicated by the thermocouple that monitors chamber temperature. This causes the LN$_2$ spray to come on prematurely, with the resultant rapid cooling of parts and the increased possibility of cracking.

The controlled immersion of components into LN$_2$ has been tried in two versions: the payload is lowered slowly into a pool of LN$_2$, or a chamber is slowly flooded with LN$_2$ so the liquid level rises to and eventually covers the payload.

Both versions suffer from a serious weakness arising from the effects of fundamental physics. First, the temperature gradient above a pool of LN$_2$ is very steep. Second, the rate of heat transport between warm solid and a cold gas at –195 °C (–320 °F) is much slower than the rate between the same warm solid and a liquid at –195 °C. Therefore, in either of the above versions, a slow decrease in the distance separating the part and the liquid does not ensure a slow rate of cooling of the part. The risk of thermal shock is increased by the steep temperature gradient above the liquid and the sudden increase in the heat transfer rate when liquid contact is made.

Cryogenic treatment is a process that holds great promise to modify and improve products in many markets, including reducing wear and extending the service life of many components. Continuing research efforts are being undertaken to understand the underlying science of DCT so process improvements can be made and the technology advanced.

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