

YES, CRYOGENIC TREATMENTS CAN SAVE YOU MONEY! HERE'S WHY ---

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Abstract

In recent years, we have seen an increase in the use of cryogenic metal treatment to improve the wear resistance of cutting tools, gears, etc., in the Container Industry. Little work, however, had been done in documenting why this treatment produced such remarkable results, and many sceptics disputed the validity and worth of such treatment. The author has completed an extensive research program on exactly what cryogenic treatment does to tool steel, and this paper presents documented evidence of how and why processes such as the Perm-O-Bond dry cryogenic treatment increase wear resistance in tools used in the Container Industry.

Introduction

There have been many "spin-offs" from our space program, and many have involved the realm of super-cold or cryogenics. One such practical application of sub-zero temperatures involved the quenching of metals in liquefied gases to improve the properties of the materials. Metals were quenched from temperatures on the order of 1600°F to 1800°F (870°C to 980°C) in liquid nitrogen (-320°F or -196°C) in a cryo-quenching process used by Grumman Aircraft Corporation to avoid warping of thin components, such as gaskets.^{1,2,3} A slower cool-down rate actually resulted with cryogenic quenching, compared with brine quenching, because the part was blanketed by a film of poorly-conducting nitrogen vapor; whereas, the part came in direct contact with the liquid in the case of a brine quench.

During the 1950's tests were made to determine the amount of retained austenite in carbon steels after cooling to cryogenic temperatures.⁴ It was found that large amounts of austenite were transformed to the harder, tougher martensite structure when the part was cooled down to liquid nitrogen temperatures. Since the austenite-martensite transformation is irreversible (unless the material is heated back to high temperatures), this observation suggested that cryogenic treatment could be used to make metal parts last longer in wear situations, because more of the cryogenically-treated steel would be in the martensite form than would be the case for normally heat-treated steels.

During the 1960's a few companies began developing practical procedures for treating such metal parts as cutting tools, gears, slitter knives, etc., to improve the wear resistance of these parts. One company--Materials Improvement, Inc., near Detroit--has developed a process, called Perm-O-Bonding, which brings out the best properties of tool steels. It was found that the greatest improvement in wear resistance could be achieved when the part was cooled very slowly, soaked at a temperature on the order of -310°F or lower for many hours, and then warmed up to room temperature slowly. The temperature-time diagram for a typical Perm-O-Bonding process is illustrated in Figure 1. In addition, the

Perm-O-Bond process involved cooling the parts in a completely dry environment--no liquid nitrogen was used. This dry process avoided the problems of thermal shock that some people had experienced in cryogenic treatment by quenching in liquid nitrogen. The use of dry cooling also allowed precise control of the temperature of the part being cooled.

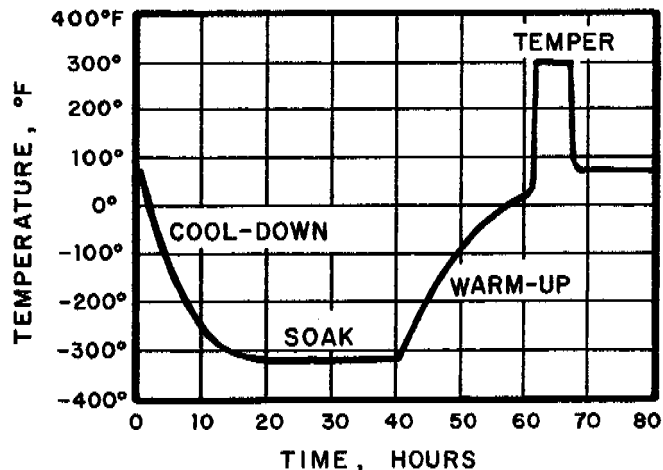


Figure 1.
Temperature-Time Diagram for Perm-O-Bond Process

Examples of Lifetime Improvements

Many companies have used cryogenic treatment to improve metal part lifetimes, and several examples reported in the literature^{5,6,7} are listed in Table 1.

Corrugating plants in many areas of the country have noted marked increases in wear life of slitters, slotters, cut-off blades, tape-cutters, and other cutting tools after the tools were treated in the Perm-O-Bond Process. For example, the average service life of untreated slitters is 6 to 8 weeks; the average service life of Perm-O-Bonded slitters exceeds 6 months. At the new Owens-Illinois plant at Cleveland, Tennessee, every slitter blade in the plant was Perm-O-Bonded, and these blades ran for well over a year without needing sharpening.

Machine down-time to change worn-out gears can be quite costly, because the machine may be out of service for many hours. Several plants, such as Union Camp (Monroe, Michigan), Consolidated Packaging (Flint, Michigan), and Packing Corp. of America (Anderson, Indiana), have found that Perm-O-Bonded gears run many months longer than untreated gears.

Cryogenic treatment of high-speed tool steels used in

Table 1. Lifetime improvements after cryogenic treatment

ITEM	Company	Average Life before treatment	Life after treatment	Ratio
2-inch end mills used to cut C-1065 steel	A	65 parts	200 parts	3.07
Hack-saw blades used to cut boss on M107 shell	A	4 hours	6 hours	1.50
Zone punches used on shell casings	A	64 shells	5820 shells	82.5
Nosing thread dies used on M485 shells	A	225 shells	487 shells	2.12
Copper resistance weld tips	B	2 weeks	6 weeks	3.00
Progressive dies used in metal working	B	40,000 hits	250,000 hits	6.25
Blanking of heat treated 4140 and 1095 steel	C	1000 pieces	2000 pieces	2.00
Broach used on a C1020 steel torque tube yoke	D	1810 parts	8602 parts	4.75
Broaching operation on forged connecting rods	D	1500 parts	8600 parts	5.73
Gang milling T-nuts from C1018 steel with M-2 cutters	E	3 bars	14 bars	4.67
AMT-38 cut-off blades	F	60 hours	928 hours	15.4

Company:

- A - Louisiana Army Ammunition Plant, Sperry Rand Corp., Minden, La.
- B - Hawthorne Metals, Detroit, Michigan
- C - S. K. Wellman Div., Abex Corp., Bedford, Ohio
- D - Detroit Area Manufacturing Div., Chrysler Corp., Detroit, Michigan
- E - DeVlieg Machine Co., Detroit, Michigan
- F - Boise Cascade Composite Can Div., Hazelwood, Missouri

lathe tools⁸ has been shown to increase the time between regrinding of the tools by a factor of from 2 to 5. The Sperry Rand Corporation at the Louisiana Army Ammunition Plant in Minden, La., used this process to increase the life of the tools used in their Metal Parts Division.

Some Partial Concepts About Cryogenic Treatment

The cryogenic treatment process is a relatively new technology, so little has been written about it. Few people are familiar with the process and the reasons that it improves wear life so dramatically. In fact, some partially true concepts have been advanced about cryogenic treatment during the past years. Some of these ideas were based on tests conducted at soak temperatures on the order of -60°F to -120°F, at which the full benefits of cryogenic treatment are not realized. Other

tests involved plunging the parts into liquid nitrogen and allowing the part to soak for a few minutes before being warmed to room temperature. This type of treatment resulted in high thermal stresses (which are not encountered during a slow cool-down), so the cryogenic treatment was considered unsatisfactory. Finally, some of the ideas were based on an extrapolation into the cryogenic temperature range of data obtained at ambient temperature and higher.

One publication⁹ purported to "clear up misconceptions" about cryogenic treatment; however, the report was somewhat biased against the use of low-temperature treatment. For example, the statement was made that "Properly heat treated tool steels ordinarily do not require refrigeration because they normally will contain little if any retained austenite." The statement that properly heat treated tool steels contain little

retained austenite is correct; however, it has been found¹⁰ that the austenite-martensite transformation is not the only mechanism involved in improving the wear life of high-alloy steels, such as tool steels. A time-dependent mechanism is also involved, because a long soak time (greater than 8 hours) is needed to improve wear life effectively in cryogenic treatment. Thus, a well-heat-treated tool will perform well in wear situations; however, a cryogenically treated tool will generally perform better.

It has been said that cryogenic treatment does not affect the grain size of the treated material. Experimental tests by Mazur¹¹ have shown that the grain size was actually reduced somewhat (4% to 6%) by cryogenic treatment. On the other hand, the atomic lattice parameters were unaffected by the low-temperature soak. This means that permanent dimensional changes during cryogenic treatment of a properly heat-treated steel are negligible, and improved dimensional stability is achieved by cryogenic treatment.

In ferrous alloys and most nonferrous alloys, bulk martensite transformation takes place at a speed equal to approximately one-third of the speed of sound in the material (5500 ft/sec. or 1680 m/s transformation speed for steel)¹², so the transformation may be considered to be essentially instantaneous at a particular temperature. Surface martensite formation has been observed for the Fe-Ni and Fe-Cr-Ni alloys¹³. Surface martensite crystals formed at cryogenic temperatures have been observed to grow at speeds several orders of magnitude slower than martensite plates formed within bulk specimens.¹⁴ Thus, the statement that it is not necessary to refrigerate gradually to -320°F is only partially true, because the improvement in wear life is a time-dependent process at cryogenic temperatures. This is an example of a conclusion formed by erroneously extrapolating data from room temperature down into the sub-zero range. Actually, a rapid cool-down will improve the part life somewhat; however, a slow cool-down will result in a greater improvement.

Results of Research on Cryogenic Treatment

In order to determine the factors responsible for the increase in wear resistance caused by cryogenic treatment, an extensive research program was conducted in the Mechanical Engineering Department at Louisiana Tech University. In this test program, five tool steel alloys (52100, D-2, M-2, A-2, and O-1) were subjected to various cryogenic treatments. More than 150 samples were soaked at various temperatures, from -200°F to -310°F, for times ranging from 480 minutes to 1200 minutes. Some samples were not tempered after cryogenic treatment, some were tempered once, and some were tempered twice.

Wear tests

Accelerated wear tests were conducted on a wear apparatus similar to the Tabor Model 503 Abrasive Testing Instrument (Teledyne Tabor Co., North Tonawanda, N.Y.), except the specimen was in the form of a rectangular bar, 0.394 in. x 0.394 in. x 2.25 in (10 mm x 10 mm x 57 mm), with the end pressed against a coarse-grit abrasive wheel 5 in. (127 mm) in diameter. A sketch of the apparatus is shown in Figure 2.

The results of the wear tests indicated that the cryogenic treatment improved the wear resistance by factors ranging from 1.2 to 11 times, depending upon the number of tempering

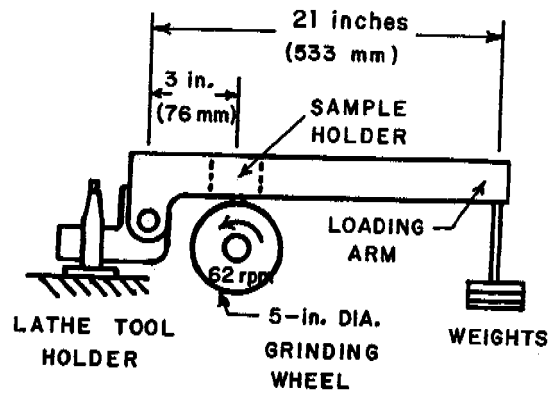


Figure 2
Wear Test Apparatus

cycles, the soak temperature, and the soak time. For example, the wear resistance ratio for 52100 tool steel could be correlated by:

$$\frac{R_w(\text{treated})}{R_w(\text{untreated})} = F_1(N) F_2(T_L) F_3(t_L) \quad (\text{Eq. 1})$$

$$\text{where: } F_1(N) = 2.393 - 0.550 N \quad (\text{Eq. 2})$$

$$F_2(T_L) = 0.247 (530^\circ\text{R}/T_L)^{1.908} \quad (\text{Eq. 3})$$

$$F_3(t_L) = 1.0614 \exp[6.73 t_L (10^{-4})] \quad (\text{Eq. 4})$$

In this equation,

N = number of tempering cycles (0, 1, or 2)

T_L = soak temperature, °R (150°R to 260°R)

t_L = soak time, minutes (480 min. to 1200 min.)

The wear resistance is defined by:

$$R_w = \frac{F V}{W H_v} \quad (\text{Eq. 5})$$

where: F = normal force on the sample, N

V = surface sliding velocity, mm/s

W = wear rate, mm^3/s

H_v = Vickers hardness, MN/m^2

Similar correlations were developed for the four other alloys tested.

The results of the wear tests indicated that the greatest increase in wear resistance was achieved when the material was cooled slowly (on the order of 0.5°F per minute) to a temperature below -300°F (-184°C), and soaked at this temperature for

long times (on the order of 1200 minutes or 20 hours). This optimum treatment process closely parallels the Perm-O-Bond process developed by Materials Improvement, Inc.

Typical numerical values for the wear resistance ratio may be found from the previous equation for 52100. For example, for N=1 tempering cycle, soaking at 150°R (-310°F or -184°C) for 1200 minutes,

$$F_1(N) = 1.843$$

$$F_2(t_L) = 2.746$$

$$F_3(t_L) = 2.381$$

$$\text{And, } R_w(\text{treated})/R_w(\text{untreated}) = 12.05$$

Hardness tests

Hardness measurements of the test specimens were taken before each wear test. Typical results are given in Table 2.

Table 2. Comparison of tool steel hardness before and after Perm-O-Bond treatment.

ALLOY	HARDNESS, R _c		CHANGE R _c
	Before Treatment	After Perm-O-Bonding	
52100	57.5	57.5	0.0
D-2	62.2	60.9	-1.3
A-2	60.5	59.9	-0.6
M-2	63.6	63.8	+0.2
0-1	60.1	61.2	+1.1

From a statistical analysis of the hardness data, it was found that the differences between the hardness of the treated and untreated samples of 52100, A-2, and M-2 were not statistically significant, i.e. cryogenic treatment had no significant effect on the hardness. The analysis of the data for the D-2 and 0-1 alloys did indicate, however, that the cryogenic treatment caused a change of approximately 1 R_c in the hardness of these alloys.

Microhardness tests taken over regions about 25 μm or 0.001 in. in size showed that the hardness of the carbide particles in the steels was unaffected by the cryogenic treatment, and any changes in bulk hardness resulted from changes of the matrix material around the hard carbide particles.

Microstructure

To determine the changes in microstructure caused by cryogenic treatment, photomicrographs were taken at 1000X magnification of the tool steels before and after treatment. Before cryogenic treatment, the microstructure consisted of relatively large carbide particles (about 100 microinch or 2.5 μm in size) dispersed within a matrix of tempered martensite. After cryogenic treatment, smaller carbide particles (about 20 microinch or 0.5 μm in size) were found distributed in the

background matrix. Any retained austenite present in the untreated samples was observed to have been transformed into martensite. This was particularly obvious in those samples which were not tempered after cryogenic treatment, since the untempered martensite showed up as a dedentrite structure in the photomicrograph.

Impact tests

After the wear tests were completed, V-notch Charpy impact tests were run on the samples. Little, if any, changes in the Charpy impact strength were observed between the cryogenically treated and the untreated samples. This result would indicate that the impact strength or the dynamic toughness of the material was relatively unaffected by low-temperature treatment.

Test on Corrugating Slitter Knife

To determine the advantages of cryogenic treatment on an item used in the Container Industry, tests were conducted on a Zenith G-63 corrugating slitter knife. This knife was circular, with a 9-1/8 inch OD (232 mm Ø) and a thickness of 5/16 inch (7.9 mm). The blade was cut in half, and one half was tested in the as-received condition. The other half was Perm-O-Bonded by soaking at -310°F (-190°C) for 19 hours, followed by 1-1/2 hours tempering at +300°F (149°C).

Hardness and wear tests were conducted on the two knife halves, and the results are shown in Table 3. As noted from the table, the hardness of the knife was relatively unaffected by the cryogenic treatment; however, the wear resistance was improved considerably. A photograph of the wear samples is shown in Figure 3.

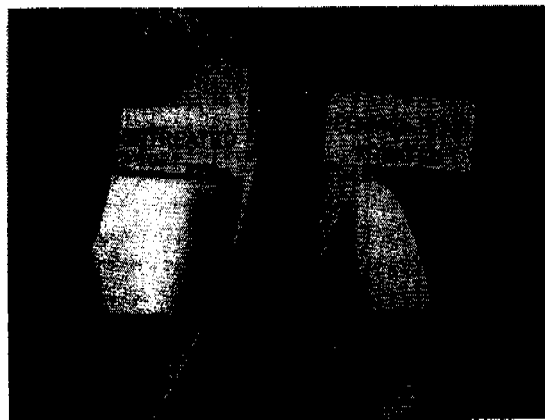


Figure 3
Comparison of Untreated and Treated Slitter
Knives from Wear Test

Table 3. Results of accelerated wear tests on a Zenith G-63 circular corrugating slitter knife.

QUANTITY	UNTREATED SAMPLE	PERM-O-BONDED SAMPLE
Initial mass	459.9 g 1.014 lbm	454.0 g 1.001 lbm
Mass loss	4.8 g 0.011 lbm	1.1 g 0.002 lbm
Volume loss	645.3 mm ³ 0.0394 in ³	147.8 mm ³ 0.0090 in ³
Time of run	30 min.	30 min.
Wear Rate, W	0.3585 mm ³ /s 21.8 x 10 ⁻⁶ in ³ /s	0.0821 mm ³ /s 5.0 x 10 ⁻⁶ in ³ /s
Hardness, H _v	59.4 Rc 6700 MN/m ²	58.9 Rc 6590 MN/m ²
Force on sample, F	431 N 96.9 lbf	431 N 96.9 lbf
Surface velocity, V	412.5 mm/s 81.2 fpm	412.5 mm/s 81.2 fpm
Dimensionless wear resistance, R _w = F V/W H _v	74.0	537.0
Wear resistance ratio, R _w (treated)/R _w (untreated) = 7.26		

The increase in wear resistance of more than 7 times, as determined from the laboratory test, was in agreement with field tests on similar slitter knives. The average life of an untreated slitter blade was 6 to 10 weeks between sharpenings; whereas, Perm-O-Bonded knives lasted from 6 to 18 months between sharpenings.

Can Cryogenic Treatment Save You Money?

It is obvious from the previous discussion that cryogenic treatment of such items as corrugating slitting knives can save a lot of money -- not only in cost of new knives, since a knife will last 4 to 10 times as long after cryogenic treatment, but also in cost of down-time in changing and resharpening the knives. For example, a G-63 blade costs \$9.25. It costs \$3.50 to resharpen the slitter blade, and the blade may be resharpened an average of three times during its useful life. Over a two-year period, four sets of blades will have been used (cost - \$37.00), and the blades will have been sharpened 12 times (cost - \$42.00), for a total material cost of \$79.00. In addition, the blades will have been changed 16 times. It takes 4 to 8 hours to change a complete set of upper and lower slitters, so the knife changing is quite expensive in terms of both man-hours and hours of lost production.

For the cryogenically treated knives, however, only one set of knives will have been used over the two-year period (cost - \$9.25 plus \$5.20 for Perm-O-Bond, or \$14.45), and the blades will have been sharpened three times (cost - \$10.50), for a total material cost of \$24.95. Note also that the blades

will have been changed only four times. The savings in material cost is 68% of the cost of the untreated blades, and 75% of the labor costs are saved by using cryogenically treated knives. These figures are based on a minimum wear resistance ratio of 4; the laboratory tests found a ratio of 7.26, and field tests have shown ratios as high as 10 or 12.

Yes, cryogenic treatment definitely can save you money-- both in material and in decreased labor and down-time costs.

How Does Cryogenic Treatment Work?

Although the cryogenic treatment of tool steels is a relatively new technology and little is found in the technical literature about the process, the way that low-temperature treatment works to improve the wear life of a part is fairly straight-forward. From our experimental program, we found two primary mechanisms in action.

First, any retained austenite is transformed into martensite during the cryogenic soak, and the martensite is tempered after the soak. The martensite structure resists plastic deformation much better than the austenite structure, because the small carbon atoms in the martensite lattice "lock together" the iron atoms more effectively than in the more open face-centered cubic austenite lattice. Tempering the martensite makes it tougher and better able to resist impact than untempered martensite.

Second, the cryogenic treatment of high-alloy steels,

such as tool steels, results in the formation of very small carbide particles dispersed in the martensite structure between the larger carbide particles present in the steel. This strengthening mechanism is analogous to the fact that a concrete made of cement and large rocks is not as strong as a concrete made of cement, large rocks, and very small rocks (coarse sand). The small hard carbide particles within the martensite matrix help support the matrix and resist penetration by foreign particles in abrasive wear.

In the case of grinding the material to sharpen the blades, the wear mechanism involves a high-speed scratching, plastic flow, and microcracking at the surface. Under these conditions, the cryogenically treated material is abraded at about the same rate as the untreated material, because the hardness in each case is practically the same. This means that the cryogenically treated blade is no more difficult to sharpen than an untreated blade. This conclusion has been supported by field tests, also. Since the cryogenic treatment affects the material through-and-through and is not a surface treatment, sharpening does not reduce the wear resistance of the blade.

Summary

As a result of extensive laboratory wear tests, it has been demonstrated that cryogenic treatment involving a slow cool-down to temperatures on the order of -310°F (-190°C) and a long soak at this temperature increases the wear resistance of tool steels by a substantial factor (from 4 to 10 times). This increase in wear resistance does not result in the material becoming more brittle, and neither is the treated part, such as a slitter knife, any more difficult to sharpen after cryogenic treatment.

From a practical standpoint, field tests of cryogenically treated parts in service have shown a similar increase in part lifetime after cryogenic treatment. Because fewer slitter blades are needed per year and the blades run longer before they must be sharpened, considerable cost savings may be realized by cryogenic treatment of the blades.

Finally, through the wear tests, hardness tests, and photomicrographs taken on more than 150 samples of five different tool steels, the basic reasons why cryogenic treatments work so well have been identified. The wear resistance of a cryogenically treated tool steel is increased because: (a) any retained austenite is transformed into the hard, strong martensite structure, and (b) additional small carbide particles are formed within the martensite matrix to combat wear further.

Acknowledgment

Financial support for the laboratory research reported in this paper was provided by Materials Improvement, Inc., Hazel Park, Michigan.

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