Increasing Hardness Through Cryogenics

Dr. John Cesarone

INFAC experiments examine how carburizing process variables and types of cryogenic treatments affect the microstructure of 9310 alloy steel.

Introduction

The Instrumented Factory for Gears (INFAC) conducted a metallurgical experiment that examined the effects of carburizing process variables and types of cryogenic treatments in modifying the microstructure of the material. The initial experiment was designed so that, following the carburizing cycles, the same test coupons could be used in a future experiment.

Background

The aerospace industry, specifically in the manufacturing of helicopters, has always used cryogenic treatments to stabilize both the geometry and microstructure of the precision gears used in the transmissions. The loading and noise requirements for these gears call for dimensional stability because of its effect on the center spacing of the gear set in operation. Research has shown that material containing levels of approximately 20% retained austenite yields parts with a higher fatigue life than that observed in parts containing lesser amounts. An increase in gear life endurance of 166% has been observed when the retained austenite level was increased from 17% to 40% (Ref. 1). However, accompanying the increase in fatigue life is a loss of geometric shape and an increase in drivetrain noise during gear set "run-in." Though these phenomena are usually not a significant problem in automotive and agricultural applications, a reduction in the amount of retained austenite in these materials may also be necessary as the need for quieter, higher performing transmissions in cars and trucks increases. The mechanism that increases a gear's performance is the mechanical shearing of the austenite, resulting in a finer martensitic structure than thermomechanically (deep frozen to -110° F) formed martensite (Ref. 2).

New helicopter designs and modifications to existing weapons systems are always taxing a material's useful life. New alloys are being adopted or modified for use. Although some of these "new" alloys have existed for as long as 25 years, most have not been used in production transmissions. SAE AMS 6265H (SAE 9310), Pyrowear 53 and Vasco X2 are the three most commonly used alloys in today's helicopter transmissions. This initial investigation focuses exclusively on SAE AMS 6265H.

Aerospace manufacturers currently specify cryogenic treatment of -110°F and generally utilize carburizing cycles that result in microstructures effectively treated at this temperature. Cryogenic processing of materials has been performed for a wide range of materials with varying levels of success based on the material chemistry. Specifications for carburized parts in current aerospace gearing materials include cryogenic treatments that improve dimensional stability, minimize retained austenite levels, increase surface hardness and improve wear properties. Efforts to shorten carburizing cycles continue despite the negative consequences that often accompany shorter carburizing cycles. Although increases in either the carbon potential or the carburizing temperature can shorten a carburizing cycle, both methods have drawbacks. For example, an increase in temperature may lead to an improper grain size or increased intergranular oxidations (IGO), and an increase in carbon potential may lead to high levels of retained austenite and a greater tendency to form carbides.

Most previous research in cryogenics has been done on tool and die materials. One reason that research has not focused on gears is that a temperature of -60°F allows complete martensitic transformation. The bulk of current research in deep cryogenics is conducted on alloy tool steels that have higher carbon contents than gear steels, which may be another reason gears have not been candidates for deep cryogenic research. However, in the carburization of a gear, the carbon level in the carburized region becomes either equal to or higher than the carbon level in the tool steels. Therefore, this series of experiments focuses on improving the properties of the gear teeth without affecting the tough, low-carbon core.

In this presentation, *deep cryogenics* refers to treatment at temperatures lower than -110°F. Deep cryogenics does not add a step to a manufacturing process that already includes freezing parts at -110°F. The present commercial availability of liquid nitrogen and the sophistication of the process control systems make -300°F treatment readily

Dr. John Cesarone
is the program director at INFAC, the Instrumented Factory for Gears, a research facility of the U.S. Army Aviation and Troop Command, located in Chicago, IL.

applicable. A relatively new proprietary process developed by Nu-Bit, Inc., call the Nu-Bit Process (NBP), involves the immersion of room temperature parts into liquid nitrogen (-320°F) for the length of time required for the temperature of the part to equilibrate with the liquid nitrogen. The part temperature equilibration is achieved when the liquid nitrogen stops boiling. A criticism of liquid nitrogen processes is that they may impart thermal shock to the parts and/or that microcracking may result (Ref. 3).

Treatment at -110°F is adequate when decreasing levels of retained austenite is the main goal, but if treating at -300°F or lower results in consistently superior performance, specification for treatment at -300°F may be appropriate. The cryogenic temperature of -110°F corresponds to the use of dry ice as a method for low temperature treating, which is still being used, as is treatment in mechanical freezers, which can freeze as low as -140°F.

Several recent research projects and various successful manufacturing processes provide valuable information regarding deep cryogenics on gear materials. One research project reports dramatic wear resistance with 10% improvement on low carbon steels and cast iron as a result of deep cryogenics treatment, although problems arise with thermal stresses when using liquid nitrogen as a quenching (cryogenic) medium (Ref. 4). According to James Smith, vice president and co-founder of 3X Kryogenics, the only sure way to use ultra-low temperatures is in carefully controlled environments with tight control over the entire cycle and in cycles that take time—days as opposed to hours. Tight control over long cycles also provides a built-in safety against thermal shock (Ref. 5).

Cryogenic treatment has been more successful on alloyed steels than on plain carbon steels (Ref. 6). It has been suggested that when martensite constitutes more than 70% of the microstructure, 10–15% retained austenite can help reduce cracking (Ref. 7). Lower percentages of retained austenite transform under work-hardening to a very fine crystalline martensite that has a tendency to resist surface cracking rather than contribute to it. As a result, 10–20%, and in some instances, up to 25% retained austenite in the case surface of carburized gears is acceptable and may be beneficial for most applications.

Debate among engineers continues about the best level of retained austenite in the case surface or carburized and hardened parts. Some argue that the retained austenite may transform to untempered martensite, either by work-hardening or by exposure to extremely cold climates, and cause tensile cracking of the surface. Others object to the presence of retained austenite in the case of a carburized part that is to be subsequently ground. Under certain grinding conditions, even small amounts of retained austenite can cause severe grinding burns and cracking. Research published by the International Nickel Company (INCO) indicates that levels of less than 50% retained austenite enhance contact fatigue and compressive stresses (Ref. 8). Other research indicates that 15–30% retained austenite is desirable in carburized direct quenched gears as long as hardness is HRC 57 or more (Ref. 9).

The purpose of this experiment is to examine the effects of the carburizing atmosphere and the cryogenic treatment on the microstructure of the material to determine how to modify effectively the material. Input variables for the experiment include the carburizing atmosphere and the cryogenic treatment. Output variables include measurements of retained austenite, surface hardness, case depth and carbon and nitrogen gradients. Because this experiment is the first phase of another experiment in which the same samples will be used, the carburizing cycles were designed to be nondestructive to the test coupons. The information contained in this report will also provide data for the next experiment. The future experiment will include more cryogenic data for determining what cryogenic treatment is necessary to yield a desired microstructure as well as performance data for further differentiating between the cryogenic treatments.

Experimental Procedure

Eleven different carburizing cycles, designed to yield eleven different carbon gradients and eleven different retained austenite levels, were performed on test coupons. Table I lists the carburizing cycle conditions. The coupons were machined from 0.40" thick AMS 6265 (AISI 9310) steel. The

Table I Matrix for the Carburizing of the Cryogenic Coupons

Process	Temperature (°F)	% Carbon	Time (hours)	NH_3
A	1600	0.6	12	Yes
B	1600	0.8	12	Yes
C	1600	0.8	12	No
D	1600	1.0	8.5	No
E	1700	0.8	6	No
F	1700	1.0	4.5	No
G	1700	1.2	3.5	No
H	1800	0.8	3	No
I	1800	1.0	2.5	No
J	1800	1.2	2	No
K	1800	1.4	1.5	No

coupons consisted of discs with teeth hobbed into them, as shown in Fig. 1, and stamped with an identification on each test area, as designated in Table II. Sections of each test coupon were removed for analysis at the appropriate step of the process, leaving the remaining part of the coupon to continue through the cycle.

The coupons were reheated and integrally quenched in a gas-fired, vertical, radiant-tube, integral quench furnace (GVRT-IQ) before deep-freeze treatment. The -110°F deep freeze consisted

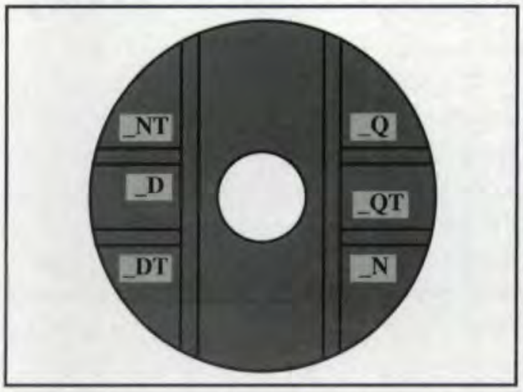

Fig. 1 — Test coupon showing identification location, position and labelling.

Table II Coupon Identification Assignments		
Designation	Treatment	Time
D	Dry ice deep freeze (-110°F)	120
DT	Dry ice deep freeze (-110°F) followed by 300°F temper	90
N	Liquid nitrogen immersion	15
NT	Liquid nitrogen immersion followed by 300°F temper	90
Q	None (as-quenched)	–
QT	300°F temper	90

Note: Identification is as follows: Each coupon is identified for the cycle and treatment, e.g., A-D, A-DT, A-N, A-NT, A-Q, A-QT; B-D, B-DT, etc. for the remaining carburizing cycles.

of one hour exposure in a chamber lined with dry ice; the -310°F deep-freeze treatment consisted of immersion for 15 minutes in liquid nitrogen. The transfer time from quench to deep freeze was less than two minutes. Samples were tempered at 300°F for one hour.

Measurements of retained austenite, Rockwell "C" hardness (HRC) at the surface and case depth Knoop hardness (KHN) were conducted at each step of the process. Six measurement points were chosen to observe the material behavior at each step in the process. The retained austenite measurements were conducted on a Windows-based TEC x-ray analyzer using a standard setup. The Rockwell hardness tests were conducted according to ASTM Specification Designation E 18-89a. The case depth measurements were conducted in accordance with ASTM Specification Designation E 384-89. Carbon and nitrogen gradient bars of material identical to the coupons were machined into 4" long bars, approximately 1" in diameter. The carbon and nitrogen gradients were conducted by collecting the turnings that were generated from the lathe without lubricant, but with deliberate care to prevent burning. The turnings were collected and bagged according to the depth turned, based on micrometer measurements. These turnings were broken into smaller pieces, and a 1-gram sample was analyzed in a Leco Carbon Determinator; nitrogen gradients were generated on a Leco Nitrogen Determinator. The carbon and nitrogen gradients were determined after the quench step, since they are not affected by further processing as long as no further high temperature processing occurs. Carbon and nitrogen gradient results appear in Table III.

Table III Carbon and Nitrogen Gradient Results (weight percent by depth in inches)

Run	A	B	C	D	E	F2	G	H	I	J	K	A	B
Depth	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Carbon	Nitrogen	Nitrogen
0.000	Bar 26	Bar 28	Bar 15	Bar 16	Bar 17	Bar 30	Bar 19	Bar 20	Bar 21	Bar 24	Bar 25	Bar 27	Bar 29
0.005	0.519	0.703	0.687	0.816	0.715	0.832	0.884	0.926	0.725	0.797	0.853	0.250	0.370
0.010	0.487	0.653	0.674	0.756	0.624	0.742	0.774	0.845	0.602	0.685	0.741	0.260	0.280
0.015	0.429	0.566	0.609	0.670	0.547	0.644	0.658	0.697	0.509	0.572	0.617	0.210	0.250
0.020	0.418	0.555	0.556	0.572	0.488	0.564	0.578	0.601	0.461	0.508	0.522	0.180	0.200
0.025	0.398	0.529	0.466	0.463	0.412	0.478	0.482	0.495	0.400	0.422	0.425	0.110	0.130
0.030	0.342	0.452	0.392	0.356	0.357	0.392	0.372	0.398	0.327	0.348	0.342	0.069	0.087
0.035	0.280	0.384	0.328	0.282	0.296	0.314	0.295	0.302	0.279	0.288	0.274	0.054	0.041
0.040	0.243	0.329	0.278	0.219	0.250	0.256	0.231	0.243	0.243	0.246	0.230	0.030	0.028
0.045	0.211	0.248	0.232	0.180	0.212	0.208	0.192	0.193	0.204	0.206	0.194	0.013	0.016
0.050	0.182	0.197	0.193	0.159	0.180	0.170	0.163	0.165	0.182	0.168	0.150	0.022	0.012
0.060	0.143	0.153	0.157	0.124	0.144	0.153	0.136	0.133	0.143	0.138	0.130	0.016	0.017
0.070	0.128	0.129	0.130	0.122	0.126	0.128	0.118	0.127	0.128	0.122	0.117	0.013	0.012
0.080	0.123	0.121	0.118	0.115	0.118	0.116	0.116	0.122	0.122	0.115	0.121	0.010	0.011
0.090		0.116	0.120	0.109	0.118	0.113	0.112	0.124	0.120	0.106	0.114	0.018	0.010
0.100		0.119	0.126	0.115	0.117	0.114	0.112	0.115	0.117	0.112	0.113	0.012	0.013

Data Analysis

A desired case depth of 0.030–0.040" was achieved and verified by microhardness examination. Table IV lists the retained austenite measurement results for the various steps of processing. The highest levels of retained austenite were observed in the high carbon potential carburized samples (G, J and K) and in the ammonia-addition carburized samples (B). The ammonia additions had no effect on the oxygen probe response, as verified by shim stock test results included in Table VI. It is possible that the addition level was too low to dramatically affect the atmosphere. The two ammoniated cycles had identical levels of added ammonia; the cycle with the higher carbon level (B) had the largest amount of retained austenite.

In general, higher carbon levels for carburizing result in larger amounts of retained austenite. This expected phenomenon was observed for the as-quenched condition; however, it did not prove to be the general convention with atmospheres of high carburizing potentials. The 1600°F runs (A, B, C and D) showed varying amounts of retained austenite after deep freezing, attributable to the ammonia addition. The 1700°F runs (E, F and G) followed the convention of increased carburizing carbon levels resulting in increased levels of retained austenite. The 1800°F runs (H, I, J and K) did not follow the convention and contained varying amounts of retained austenite not attributable to ammonia addition, since none was added during these cycles.

The differences in retained austenite between cryogenics and deep cryogenics are illustrated in Figs. 2–4. In general, NBP treatment decreased retained austenite levels more than the dry ice treatment. When the parts were tempered, the dry ice treated parts transformed more retained austenite than the NBP treated parts; therefore, the levels of total percent change in retained austenite for either cryogenic treatment were approximately equal.

Recent research by the Iron and Steel Institute of Japan (ISIJ) indicates that performance properties may be improved without evidence of further retained austenite reduction. This is explained by martensitic decomposition, formation of beneficial η-carbides, reduction of detrimental commonly formed ε-carbides and a finer martensitic structure. The formation of η-carbides was observed by electron microscopy only when the parts were treated to deep cryogenics. Conventional cryogenic treating to -110°F does not form η-carbides (Ref. 10).

Previous research (Ref. 11) indicates that increased carbon levels assist nitrogen diffusion because the carbon atoms open up the crystal lattice to allow easier nitrogen atom diffusion. The observed nitrogen gradient results for this experiment do not indicate this to be true for 9310. The nitrogen gradients appeared similar between runs A (0.6% C) and B (0.8% C); however, this may not be enough of a difference in carbon level to observe an effect of the nitrogen diffusion. Other research (Ref. 12) indicated much higher amounts of nitrogen in a carburized case than that achieved in the investigation presented in this report. The higher nitrogen case levels involve Raw Town Gas (RTG), which contained a different CO/CO_2 ratio than an endothermic based atmosphere. This research suggests that the amount of nitrogen diffusion into the case is not only dependent on temperature, but also on the CO/CO_2 ratio. The different CO/CO_2 ratio also resulted in lower levels of carbon diffusion into the case.

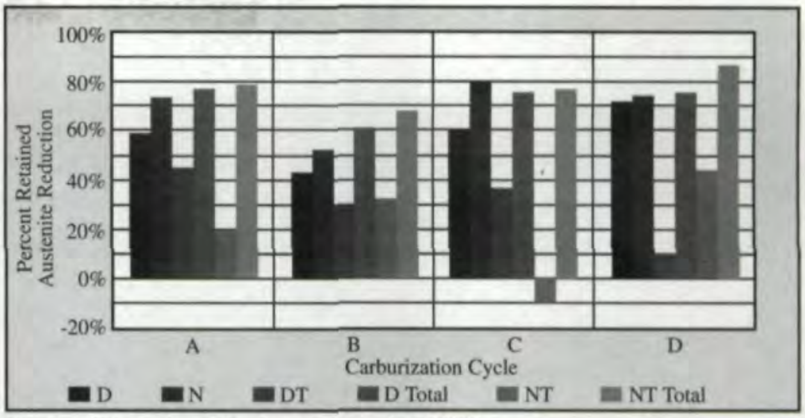

Fig. 2 — Change in retained austenite in the 1600°F runs.

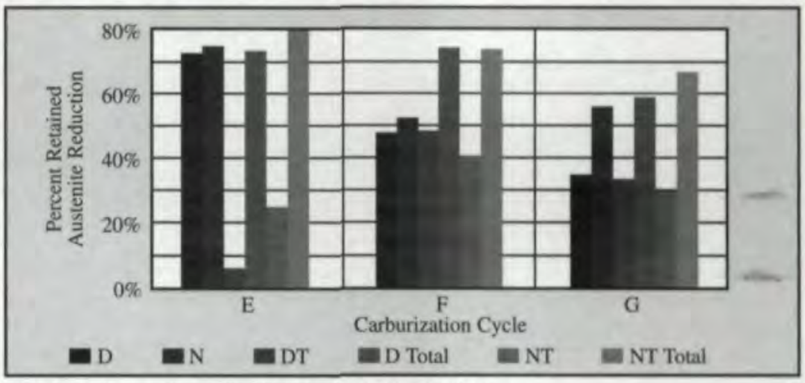

Fig. 3 — Change in retained austenite in the 1700°F runs.

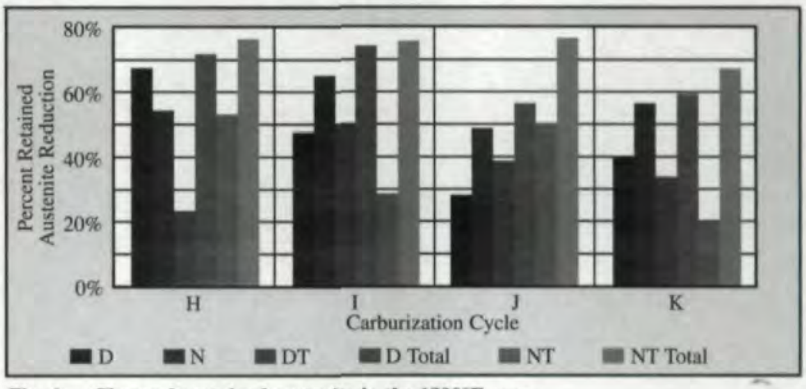

Fig. 4 — Change in retained austenite in the 1800°F runs.

Table IV Retained Austenite Levels Before and After Cryogenic Treatments of -110°F and -320°F						
Run	Q	QT	D	N	DT	NT
A	41.4	35.9	17.0	11.2	9.4	8.9
B	83.2	72.2	48.9	40.9	33.8	27.4
C	38.3	35.6	15.3	8.0	9.5	8.8
D	29.5	25.4	7.9	7.7	7.1	4.3
E	41.2	37.2	1.5	10.5	10.8	8.0
F	55.1	45.5	29.2	25.8	15.0	15.4
G	66.2	57.0	41.5	31.5	27.1	21.9
H	42.7	34.4	15.1	20.6	11.7	9.9
I	51.6	46.4	26.2	17.6	13.0	12.5
J	63.0	57.3	44.7	31.8	27.3	15.9
K	68.5	64.7	40.9	29.0	27.8	23.2

Table V Rockwell "C" Surface Hardness Test Results						
Run	Q	QT	D	N	DT	NT
A	62.4	59.3	62.8	63.2	62.3	62.7
B	61.0	58.7	62.4	65.6	63.6	64.3
C	60.0	58.4	64.2	65.1	61.4	61.2
D	58.0	56.5	63.7	63.8	62.5	63.0
E	60.9	58.8	64.8	68.0	62.5	65.5
F	59.5	56.4	63.9	62.8	62.1	62.2
G	55.6	58.3	63.1	65.3	64.8	62.8
H	61.6	57.7	64.4	64.4	60.5	61.3
I	59.8	56.6	64.6	63.4	61.1	61.1
J	57.6	53.8	61.9	63.5	61.2	61.8
K	55.0	48.9	62.3	62.4	60.6	61.8

The HRC results are listed in Table V, and a project data summary in Table VI. The results indicate that hardness is not necessarily a function of retained austenite. Using 30% retained austenite as a reference point, hardness values varied from a high of 68.0 HRC to a low of 56.5 HRC for amounts less than 30%, and from a high of 65.6 HRC to a low of 48.9 HRC for amounts greater than 30%. The maximum hardness resulted from the NBP treatment.

Conclusions

- Only the runs at 1700°F showed increased levels of retained austenite as a result of increased carbon levels.
- Increasing the carburizing temperature shortened the cycle times and did not increase the amount of retained austenite. Providing the furnace does not leak, increasing the carburizing temperature does not contribute to increased intergranular oxidation (IGP).
- The 1800°F carburizing cycles resulted in an increased grain size of the core material for the as-quenched samples; however, this may be controlled by a proper reheat and quench cycle.
- The effect of the NBP process is not apparent by hardness, retained austenite, residual stress or metallographic structure.
- Treatment at -110°F or NBP both reduced the retained austenite levels significantly and increased hardness from a range of HRC 49–59 to a range of HRC 60–65.
- NBP treated samples exhibited no signs of thermal shock or cracking.

Future Work

The effect of the NBP process on the performance properties of wear and single tooth bending will be studied in the next experiment. Gear teeth were cut on every sample coupon to simulate real-world material applications. The test coupons were tempered after deep freeze treatment to lock in the properties until performance testing could proceed. This will determine if the deep cryogenics impart similar benefits to gears as those observed in the tool and die materials in which they have been used successfully.

Future experiments may include expanding to other "new" gear materials such as Vasco, which was developed by Boeing Vertol and Teledyne Vasco for high hot hardness and the ability to be carburized.

Further studies on atmosphere addition will be investigated using higher ammonia additions, higher temperatures and higher carbon levels. ○

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