Deep Cryogenic Treatment of Materials for Aerospace Applications

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The results of Rolling/Sliding Contact Fatigue (R/SCF) tests were compared for two aerospace carburizing heat-treatment processes. All samples were from one heat lot of AMS 6265J (Single Vacuum Melted SAE 9310H) gear material. The material was normalized, quenched, tempered, carburized, subcritical annealed, copper plated, quench hardened, tempered, copper stripped, ground and surface temper inspected to conventional aerospace specifications. All samples were processed in the same heat treat lot. Some were randomly selected as the baseline process for testing. Others were given an additional deep cryogenic treatment (-320°F) and temper after grinding. The following are presented: R/SCF test results (with end-of-test pit appearance criteria), metrological changes, microstructural changes (the presence of eta carbides and microcracking), and temper resistance behavior. Recommended heat treatment and cryogenic treatment processing specifications are included in the paper with a discussion of the benefits of tempering prior to deep cryogenic treatment. Eddy current examination and use of the Barkhausen Effect are discussed as test methods for confirming cryogenic processing. Also discussed in this paper are other potential benefits of cryogenic treatment for carburized gear materials such as cryogenic stress relief. Recommendations are given for additional work in this area.

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1 Introduction

During the heat treatment of steel parts, it has been a normal practice to include a -120°F cold treatment cycle. This process is performed after the austenitic steel is quenched, but before it is tempered. This process is performed to transform the remaining austenite in the quenched part into martensite. Martensite improves the wear characteristics and hardness of finished steel.

In recent years, devices for precisely controlling the temperature of components at -320°F have become available. This has allowed experimenters to evaluate the effect of cryogenic processing on heat treated steels. The cryogenic treatment of steel products has become a controversial process. Claims of improvement in performance properties and part life for cryogenically processed steel components have been made since the early 1940's. These testimonials to the improved properties of cryogenically treated components have been met with counter claims that the process does not always result in improved properties.

In the former Soviet Union, a cryogenic process referred to as "Shock Cooling" was developed to process steels. The steel was lowered into a bath of liquid nitrogen and held there until the part reached equilibrium (temperature); the steel was then allowed to return to room temperature. The materials that were treated included stainless steels and molybdenum alloy steels. It was claimed that the process improved the wear characteristics of the steel and the practice of treating steel with the Shock Cooling method became a widespread application in over 100 enterprises. Between 1977 and 1978, the State Committee on Patents and Discoveries in the Soviet Union determined that while there was considerable savings gained through the cryogenic process, the results were not consistent. In the survey taken, it was determined that only 70% of the 204 enterprises evaluated were obtaining consistent results.[1]

Additional development work on the cryogenic process and its control was performed in Great Britain. The process developed in Great Britain eliminated the possibility of thermal shock that could occur in the Shock Cooling process. This was accomplished by pre-cooling the steel prior to immersion into the liquid nitrogen. After removal from the liquid nitrogen, the steel was allowed to return to room temperature in air.[2]

In Canada, research by private corporations has been undertaken with the support of the Canadian Industrial Research Assistance Program. This research has developed a cryogenic process that has produced claims of improvements in wear from 40 to 300%. [3]

Research efforts were performed in the United States at the Louisiana Technical Institute to determine the effect of cryogenic exposure time on wear resistance properties in steel components. This study determined that holding steel at a temperature of -310°F for a period of over 20 hours would result in a component with improved wear resistance. [4]

2 Experimentation

2.1 Investigation

Experimentation was conducted at IIT Research Institute's "Instrumented Factory for Gears" Heat Treatment facility, which has a state-of-the-art Honeywell computer control system. This system has the capability of controlling the cryogenic chamber ramp-down at 20°F/hour. The chamber itself is equipped with a circulating fan that can operate at -320°F. Dual control-solenoid-valves eliminate the possibility of frozen controls during extended cycles. The carburization furnace heat zone is controllable to within ±3°F between the operating temperatures of 1475°F and 1700°F. This precision was verified using a standard nine-point survey.

The purpose of this experiment was to determine the effect of the cryogenic process on the material and any resulting material property improvements due to the process.

Changes in pitting resistance and temper resistance due to cryogenic processing were investigated. Changes in the pitting resistance of a cryogenically treated material correspond to changes in the wear characteristics of the material. Temper resistance was examined to see if cryogenically treated materials could withstand a higher-temperature environment than conventionally treated materials. Improvements in temper resistance would improve resistance to softening due to exposure to high temperature environments and would result in the ability of parts to withstand a more severe operating environment.

2.2 Methodology

This project began with a review of previous work in cryogenic treatment. A process was selected, and AMS 6265J (Single Vacuum Melted SAE 9310H) was chosen as the material of interest. A baseline utilizing the existing conventional hardening process was established and then compared to the results of the cryogenic process. The conventional process utilizes a standard cold treatment (-120°F). The cryogenic process was designed such that results would be obtained from both a pre-temper and a post-temper cryogenic cycle.

The selected method of testing for pitting resistance was the Rolling/Sliding Contact Fatigue (R/SCF) test. The selection of this test required that the material be machined into a set of mating parts consisting of a pin and a disk. This configuration was used for both the pitting resistance and temper resistance evaluation.

After machining the parts into the required pins and disks, they were normalized (to relieve any machining stresses). Normalization was accomplished by heating the material above its upper transformation temperature and slowly cooling the parts to room temperature. After normalization, the parts were reheated to 1500°F in a controlled-atmosphere integral-quench furnace, quenched in oil (120°F) and tempered at 300°F for three hours.

The material was carburized at a temperature of 1700°F to obtain a case depth of 0.040 in. The furnace was lowered in temperature to 1450°F and the parts were stabilized for one hour. The material was then allowed to cool in the furnace vestibule. Following this, the material was sub-critical annealed at 1175° F for three hours.

Randomization was utilized for division into a baseline lot and multiple cryogenic lots.

The parts were copper plated to a thickness of 0.001-0.002 in. prior to performing the hardening cycle. A controlled atmosphere integral quench furnace was used to austenitize the parts (1500°F) for two hours. The parts were then quenched in oil (120°F). The baseline and post-temper cryogenic lots were given a cold treatment (-120°F) for two hours and were tempered at 300° F for three hours. The pre-temper cryogenic lot was given a cryogenic treatment (-320°F). The cool down ramp was controlled at 20° F/hr. The parts were held at that temperature for a period of 20 hrs. The parts were returned to room temperature at a rate of 20°F/hr and then tempered at 300°F for two hours.

The post-temper cryogenic lot was given a cryogenic treatment (-320°F). Ramp-down was controlled to 20°F/hr and the material was held at temperature for 20 hrs. The parts were returned to room temperature at a rate of 20°F/hr and then tempered at 300° F for two hours.

After heat treatment, the copper plating was stripped and the parts ground to their final configuration. A surface temper etch inspection was performed to ANSI/AGMA 2007-B92 and ISO14104: 1995 specifications.

3 Results and Discussion

The parts were evaluated for retained austenite and residual stress by means of x-ray diffraction. Both the baseline and cryogenic lots exhibited similar retained austenite and residual stress. The retained austenite was measured at between 6.0-7.4%. Residual compressive stress was measured at between 77-88 ksi.

The microstructure of the baseline lot and the post-temper cryogenic lot was examined at a magnification of 30,000X using a SEM. The microstructure of each lot was similar with no noticeable differences. The parts were also evaluated for evidence of microcracking and none was found.

R/SCF tests were used to evaluate the wear characteristics of the baseline and post-temper cryogenic lots. The results of these tests are illustrated in Figure 1. Comparison between the baseline and the post-temper cryogenic lots demonstrates that the post-temper cryogenically treated components have a 50% extra pitting resistance life and have a 5% greater load carrying capacity. The cryogenically treated parts achieved a life of 7.42 million cycles compared to the baseline parts, which achieved a life of 4.9 million cycles. The tests were at a load of 400 KSI and 43% slip. The results of the retained austenite measurements indicate that the improvement is not a result of retained austenite being transformed into martensite.

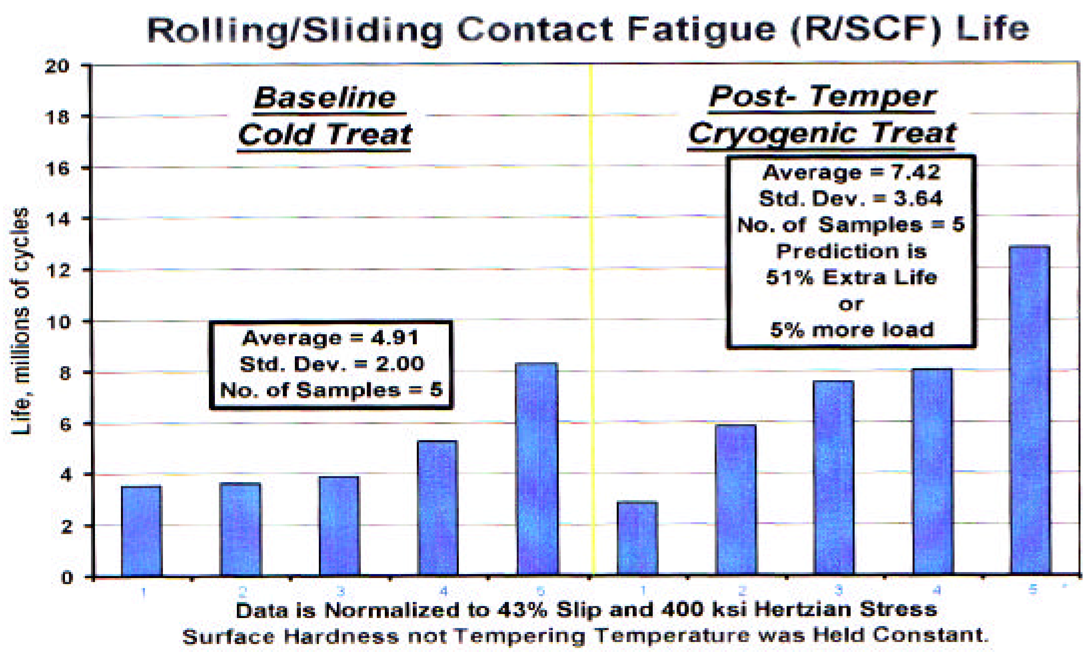

Figure 1 – Results of R/SCF tests performed on the baseline and post-temper cryogenic lots.

Research performed at the Iron and Steel Institute of Japan (ISIJ) supports this interpretation. The ISIJ study indicated that material performance could be improved without reducing retained austenite. This is accomplished through martensitic decomposition, formation of *h*-carbides, reduction of *e*-carbides, and a resultant finer martensitic structure.[5]

Temper resistance of the baseline, pre-temper cryogenic, and post-temper cryogenic lots was evaluated. Rockwell-C Hardness (HRC) was used as the response variable, and was checked on a calibrated Wilson hardness tester. The hardness readings of the parts were taken “as received,” and were also taken after the

Cryogenic treatments were performed. Parts from the post-temper lot and the pre-temper lot were further tempered at increasing temperatures in increments of 25F°, and the resultant hardness readings were recorded.

The data from the temper resistance evaluation indicates that cryogenic treatment improves the temper resistance of the treated steel. The analysis indicates that the pre-temper cryogenic treatment creates a greater improvement than the post-temper cryogenic treatment. The pre-tempering cryogenic treatment was able to withstand temperatures between 40 and 60F° more, to effect the same reduction in hardness. The results of the temper resistance evaluation are shown in Figure 2.

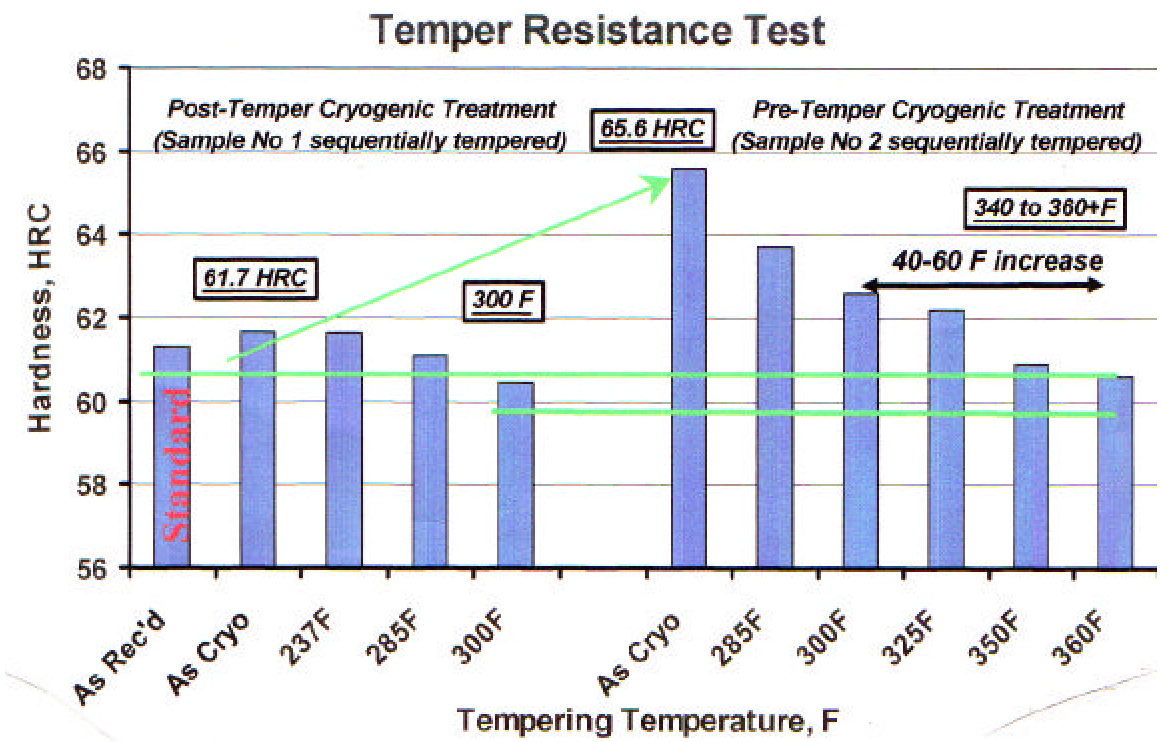

Figure 2 – Results of the temper resistance evaluation.

In an attempt to identify and distinguish between the effects of the cryogenic treatment, two evaluation methods were investigated: Eddy Current and Barkhausen Noise Analysis.

Eddy Current is a non-destructive test that has successfully been used to identify cracks in material and differences in heat treatment processes. The inspection method relies on the inducing of “eddy currents” in the material to be inspected. This is accomplished by bringing a coil with alternating current flowing through it into contact with the material to be tested. The voltage is monitored across the coil. Interruptions in the eddy current flow in the material being tested will change the loading on the coil by increasing or decreasing the effective impedance and this will effect a change in the instrument voltage.

The Barkhausen Noise Inspection method is used to detect grinding damage in gears. This relies on locating a magnetizing field near a ferromagnetic material. The field causes the material to undergo a net magnetization change. This change is a result of a microscopic movement of magnetic domain walls in the material. When the change occurs, a detectable electrical pulse is given off that can be compared against a measured standard.

The Eddy Current and Barkhauser test methods require that a standard be utilized as a baseline for examining differences. Several unsuccessful attempts were made to identify a standard for either method that would consistently identify cryogenically treated components. The failure to identify a standard for either evaluation method has prevented their use as a means to obtain data from the cryogenic treated parts.

4 Recommendations for Future Work

Additional evaluations are underway at IIT Research Institute in this area of study. A project investigating the effect of cryogenic treatment on stress relief and wear in Heat Treated SAE 4130/4140 steel tubes is just getting underway. Initial results indicate that cryogenic treatment of this steel can relieve stress due to manufacturing. Other areas of concern include:

- A method of identifying material that has been cryogenically treated and of quantitatively measuring the condition of the material needs to be developed.

- Efforts should be made to investigate the effect of combining Deep Cryogenic treatment with improvements in surface finishing and surface coating treatments.

- A sensitivity analysis needs to be performed on the process and materials to identify the most significant factors affecting these phenomena and to explain the mixed performance results often reported. This analysis will utilize the unique Laser Gas Analyzer recently installed on the atmosphere control system.

5. Conclusions

The following conclusions are drawn from the study:

- Cryogenic treatment improves Rolling/Sliding Contact Fatigue life.
- Cryogenic treatment improves temper resistance.
- Non-destructive testing did not distinguish the different in processes. The inability to identify and establish a measured standard restricted their usefulness.

6. Acknowledgements

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The R/SCF tests were performed at Penn State University's Gear Research Institute.

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