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Process to Minimize Distortion during High Pressure Gas Quenching Processes

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Abstract

A gas quenching method was developed by DANTE Solutions, in conjunction with the U.S. Army Combat Capabilities Development Command Aviation & Missile Center (DEVCOM AvMC), to control distortion in difficult to quench geometries. This new method addresses the nonuniform cooling inherent in most gas quenching processes. A prototype unit was constructed and tested with the aim of controlling the martensite formation rate uniformity in the component being quenched. With the ability of the DANTE Controlled Gas Quenching (DCGQ) unit to control the temperature of the quench gas entering the quench chamber, thermal and phase transformation gradients are significantly reduced. This reduction in gradients yields a more uniform phase transformation, resulting in reduced and predictable distortion. Being able to minimize and predict distortion during gas quenching, post heat treatment finishing operations can be reduced or eliminated, and as such, fatigue performance can be improved. This paper will discuss the prototype unit performance. Mechanical testing and metallographic analysis were also performed on Ferrium C64 alloy steel coupons and will be discussed. The results obtained showed that the slower cooling rate provided by the prototype did not alter the microstructure, hardness, strength, ductility, toughness, or residual stress of the alloy.

Introduction

Distortion can generally be divided into two categories: size change and shape change. Solid-state phase changes occurring in steel alloys during thermal processing can result in permanent size change, due to the difference between the starting microstructure and the microstructure after heat treatment. Size change is unavoidable but can easily be predicted and accounted for in part design. Permanent shape change is a result of nonuniform plastic strain, caused by nonuniform phase transformations, thermal strains, or creep while at high temperature, and is more difficult to predict and control. The nonuniformities can be a result of alloy segregation, uneven heating or quenching, poor support while at high temperature, thermal expansion or contraction restrictions, or residual stresses from prior manufacturing operations.

Traditionally, liquid quenchants were used to quench most steel parts to obtain a martensitic microstructure. Liquid quenchants undergo a unique phenomenon, comprised of three stages, when a red-hot part is immersed into the liquid [1]. First, a thin vapor film is formed around the red-hot part, with extremely slow heat transfer rates. Nucleate boiling commences as the vapor blanket breaks down. Nucleate boiling results in the fastest heat transfer due to a combination of the latent heat of vaporization and aggressive convection. Convective cooling, the final stage, begins as the nucleate boiling subsides [2].

The continually changing heat transfer rates associated with liquid quenching can severely affect the cooling uniformity of a given part. First, the breakdown of the vapor blanket rarely occurs evenly on all part surfaces, being dependent on the part surface temperature, local flow behaviour, and the liquid properties, creating brief periods of nonuniform heat transfer. The chaotic nature of this phenomenon is difficult to predict and can lead to inconsistent distortion within a single load of parts. Part geometry and immersion orientation also play a significant role in nonuniform cooling when quenching in liquids [3 – 5].

High pressure gas quenching (HPGQ) does not involve a phase change of the quenching media, and therefore, has a more stable heat transfer rate. However, due to its low density and specific heat, gas is unable to absorb energy as well as liquids, and will suffer a temperature change as heat is removed from the part. Gases' low density also make it more susceptible to local flow variations. HPGQ equipment can also significantly contribute to local flow variations [6].

In response to large distortion during HPGQ of complex geometries, DANTE Solutions devised a novel process, termed DANTE Controlled Gas Quenching (DCGQ), by which the martensitic phase transformation is controlled during gas quenching [7]. Since the transformation from austenite to martensite is driven by a reduction in temperature, and is not time dependent like the diffusive phase transformations, the simplest way to control the martensite transformation is to control the rate of temperature change within the component. By controlling the uniformity of martensitic transformation throughout the part, distortion can be significantly reduced, easily predicted, and consistently reproduced. This paper will examine the DCGQ prototype unit design and operation. Material property testing was also conducted, and discussed in this paper, to show that a slowly transformed martensitic structure was equivalent to a rapidly formed structure.

Equipment Description and Operation

Equipment Design and Construction

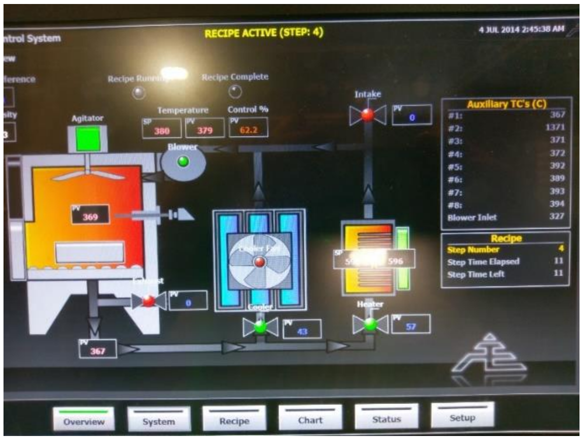
The DCGQ process was developed by DANTE Solutions after hundreds of hours were spent evaluating DANTE quenching models and determining temperature gradients which allowed for minimal distortion of difficult to quench geometries, generally encountered in power transmission applications. It was determined that by maintaining a set temperature difference between the fastest cooling point and the slowest cooling point on a part, distortion could be significantly reduced. If the temperature difference is kept sufficiently small, shape change can be completely eliminated, and only the resulting size change from the phase transformations is realized.

Atmosphere Engineering (now part of United Process Controls), in Milwaukee, WI was contracted to design and construct the DCGQ prototype unit, with specifications defined by DANTE Solutions. The system includes a one (1) m^3 working zone within the quench chamber, separate hot and cold chambers for temperature manipulation of the quench gas, a human machine interface (HMI) for system manipulation and process monitoring, and custom program logic developed by Atmosphere Engineering to follow time-temperature recipes by mixing gases from the hot and cold chambers.

Figure 1 shows the front of the quenching chamber, where the parts are loaded. Figure 2 shows the back of the unit. The electrical panel, with the HMI unit, is in the foreground of Figure 2, with the cold chamber directly behind it, and the hot chamber to its right. Figure 3 shows the HMI. The HMI allows access to, and manipulation of, the system logic and parameters, as well as being where recipes are entered, and processes monitored. Shown in Figure 3 is the process monitoring function, which allows the user to view the recipe setpoint and the actual temperature of the gas entering the quench chamber, while also monitoring the position of all the various valves required to operate the equipment. The tables on the right of the screen show the temperature values of several thermal couples located within the chamber or thermocouples attached to a quench probe or part.

Figure 1: Front of the DCGQ prototype unit.

Figure 2: Back of the DCGQ unit.



Auxiliary TC's (C)
#1: 367
#2: 1371
#3: 371
#4: 372
#5: 392
#6: 389
#7: 393
#8: 394
Blower Inlet 327

Recipe
Step Number 4
Step Time Elapsed 11
Step Time Left 11

Figure 3: Human Machine Interface on DCGQ prototype unit showing process monitoring functionality.

Equipment Operation

The unit was constructed at Atmosphere Engineering and shipped to Akron Steel Treating in Akron, OH, where it was installed and tested. This first prototype unit requires heating to be performed in a separate furnace, with the part transferred by rail cart to the DCGQ unit after austenization is complete; future production equipment should integrate controlled heating, as well as cooling. The DCGQ unit is preheated to a predefined temperature during the austenitization process in preparation for quench; the preheat temperature is alloy and part geometry dependent. The DCGQ recipe programmed into the HMI begins once the front-loading door is closed. Figure 4 shows a comparison of the temperature of the quench gas entering the chamber ("Chamber Inlet PV") and the recipe setpoint temperature ("Chamber Inlet SP") with no hot part in the chamber. The recipe consists of 50° C temperature reductions over two minutes, starting at 425° C, with twenty-minute holds at each temperature step.

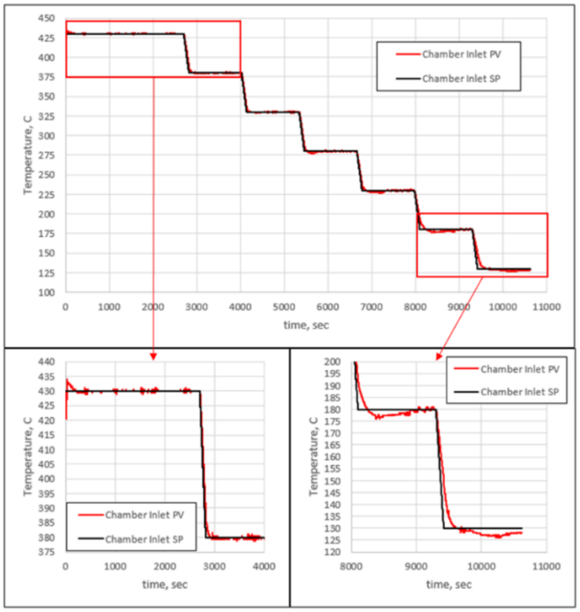


Figure 4: DCGQ Prototype unit temperature comparison between quench gas entering the quench chamber and the recipe setpoint temperature, with an empty chamber.

Figure 4 shows that the unit logic works well, and the system has no issues following the prescribed time-temperature recipe. The system did struggle a bit at lower temperatures, but still maintained the process temperature within 5° C of the recipe setpoint temperature, which was the tolerance programmed into the unit.

Figure 5 shows the unit performance using a recipe with 25° C temperature ramps over two minutes, with two-minute holds between temperature reductions. Figure 6 shows the unit performance using a recipe with a 100° C temperature reduction over five minutes, with a 5-minute hold, followed by a 120° C reduction over five minutes. In all three cases, Figures 4 – 6, the unit performed exceptionally well.

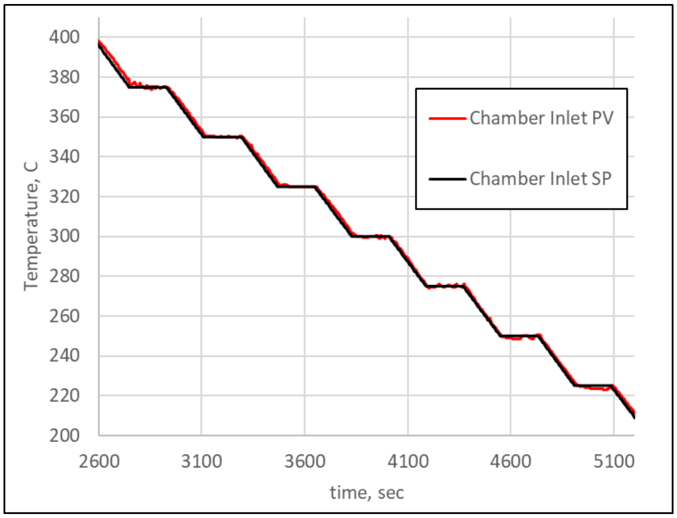


Figure 5: DCGQ Prototype unit temperature comparison between quench gas entering the quench chamber and the recipe setpoint temperature, with an empty chamber.

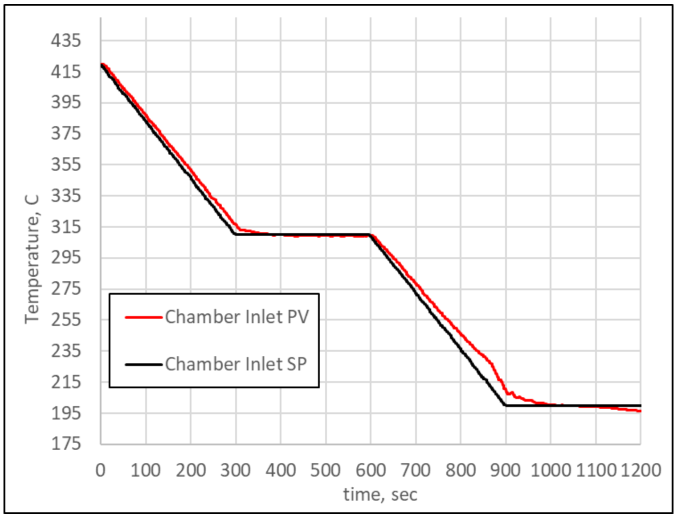


Figure 6: DCGQ Prototype unit temperature comparison between quench gas entering the quench chamber and the recipe setpoint temperature, with an empty chamber.

The three time-temperature recipes shown in Figures 4 to 6 represent schedules required for various geometries. For geometries with a thin, uniform cross-sectional thickness, such as rings, the designed temperature reductions can be relatively large, and the ramp and hold times can be short, since the part cools relatively quickly and uniformly. For thin geometries with a slightly nonuniform cross-sectional thickness, such as ring gears, temperature reductions should be kept small, due to the mass differences in the part, but the ramp and hold times can be relatively short, as the part will cool quickly. For parts with significant unbalanced mass distributions, such as crankshafts or eccentric bores, temperature reductions should be kept small, and ramp and hold times should be long.

To reduce recipe design time, and ensure an optimal recipe is achieved, with respect to distortion minimization and processing time, the equipment must be thermally characterized so heat treatment simulation and design software, such as DANTE, can be used for DCGQ recipe design. The following section describes the characterization of the DCGQ prototype unit.

Equipment Characterization

To properly characterize the DCGQ prototype unit for modeling and process design, or any other type of thermal equipment, it is necessary to determine the heat transfer coefficients (HTCs) and ambient temperatures acting on the component being treated. For liquid quenching operations, the convective HTC should be described in terms of part surface temperature, due to the significant difference in heat transfer between the vaporization, nucleate boiling, and convective stages of liquid quenching. The ambient temperature is assumed to remain constant, due to liquids' large specific heat and overall volume in the quench vessel.

However, for gas quenching operations, it is assumed that the convective HTC remains constant, since there is no phase change associated with cooling in gas, and the ambient temperature is a function of process time, due to gases' low specific heat (generally a magnitude less than liquids) and volume in the quench vessel. The ambient temperature as a function of time will vary for any single piece of gas quenching equipment, operating at the same conditions, and is dependent on the total mass being quenched, surface area of the load, and the initial temperature of the load.

Figure 7(A) shows a cylindrical quench probe, made of AISI 304 stainless steel, used to characterize the DCGQ equipment. The cylinder has a 100 mm diameter and 100 mm height. There are five holes drilled to mid-height, four approximately 3 mm from the outer diameter and one in the center, which are fitted with K-type thermocouples. Thermocouples are also located in the DCGQ chamber at various distances from the probe to measure the ambient temperature. Using the time-temperature history of the quench probe and recorded ambient temperature for a given DCGQ recipe, along with a DANTE model of the quench probe, shown in Figure 7(B), the HTC and ambient temperatures can be determined.

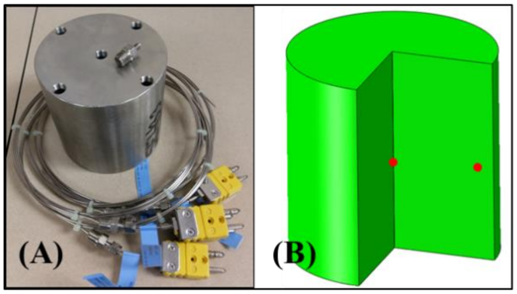


Figure 7: (A) Quench probe used for DCGQ equipment characterization, and (B) DANTE model of the quench probe.

For DCGQ processes, the ambient temperature as a function of time is dependent on total mass, surface area, and initial temperature, just as it is with HPGQ, but the ambient temperature for DCGQ is also dependent on the temperature ramp and hold times. Figure 8 shows the results of a comparative analysis between the setpoint and actual temperatures at the ends of the holds (left) and ramps (right) for various DCGQ schedules. Ideally, to make design and analysis of the DCGQ process simpler and more intuitive, the setpoint and actual temperatures should be equal at the end of each temperature ramp and hold. For the processes shown in Figure 8, HR4 and HR7 abide to this equality, but as the ramp and hold times get shorter, these two temperatures begin to deviate; HR5 had the shortest ramp and hold times. This fact does not negate the use of modeling in recipe design, but rather solidifies the fact that processing equipment behavior should be thoroughly understood, within normal operating conditions, if the process is to be modeled. Once the behavior is understood, it can be incorporated into the models for more accurate results.

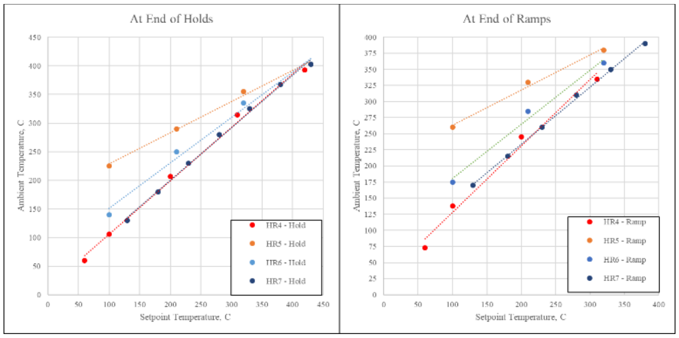


Figure 8: Relationship between recipe setpoint and ambient temperatures at the end of the Holds (left) and Ramps (right) used for DCGQ recipe design and analysis.

Figures 9 and 10 show time-temperature results comparing the DANTE simulation, which used the single HTC and relevant ambient temperatures determined from experiments, and the actual time-temperature data. In addition to the two tests shown in this paper, five more recipes were executed, with their data also used in characterizing the DCGQ equipment. Figures 9(A) and 10(A), 1000° C to 400° C, and Figures 9(C) and 10(C), 400° C to 100° C, show the average temperature history for the near surface thermocouples during the HR4 and HR5 tests and DANTE simulation of these two tests. Figures 9(B) and 10(B), 1000° C to 400° C, and Figures 9(D) and 10(D), 400° C to 100°

C, show the temperature history for the core thermocouple during the HR4 and HR5 tests and DANTE simulation of these tests. There is good agreement between simulation and experimental results, with the tests not shown showing similar agreement. Each DANTE model of the DCGQ process used the same HTC, but the ambient temperature varied depending on the temperature ramp and hold times of the given recipe.

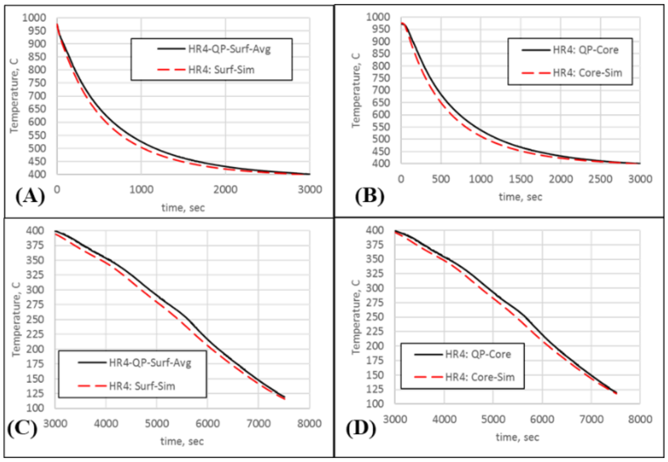


Figure 9: Comparison of experimental and simulation results for one DCGQ recipe.

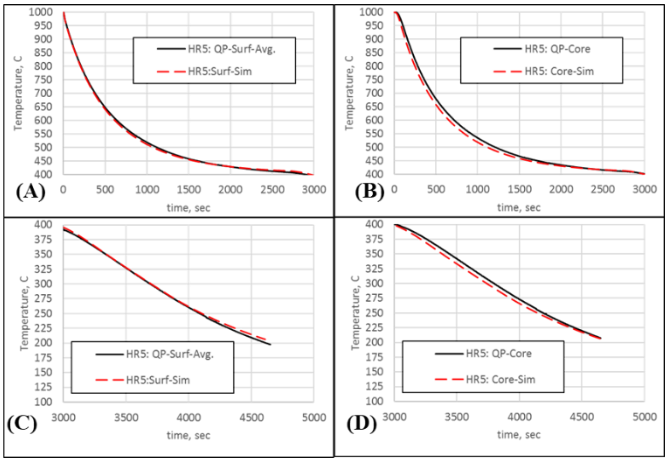


Figure 10: Comparison of experimental and simulation results for one DCGQ recipe.

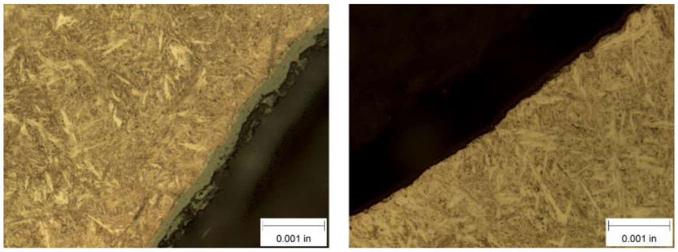
Material Testing Results

The previous section showed that the temperature of quench gas, at atmospheric pressure, could be controlled within the range of martensitic formation for high hardenability steels. However, doubt still existed as to whether or not a martensitic structure formed over such a long time period could perform as well as a standard high pressure gas quenched structure. Therefore, a testing program was launched to compare DCGQ to a standard high pressure gas quenching process.

Ferrium C64 was chosen as the candidate alloy, due to its high hardenability, use in high stress powertrain applications, and high tempering temperature; since the martensite transformation occurs slowly using DCGQ, self-tempering may occur during quench if the tempering temperature is less than the martensite start temperature (M_S). Further testing is needed to evaluate this effect. All comparative testing was performed on identical coupons machined from one, 100 mm diameter bar. For tests which required carburization, a single LPC process was executed by Solar Atmospheres in Souderton, PA on all coupons as one batch using a predefined LPC recipe. The HPGQ coupons were processed first, at Solar Atmospheres, and the austenitizing step was analyzed such that the time the coupons spent in the austenite phase was noted and duplicated with the DCGQ coupons. This was done to ensure any additional carbon diffusion occurring during austenitizing was similar between the two sets of coupons. All processed coupons, DCGQ and HPGQ, were also subjected to cryogenic and tempering treatments. The testing included, on carburized coupons, microstructural evaluation, hardness, and residual stress and on noncarburized coupons, tensile, impact strength, and distortion.

Microstructural Evaluation

Previous experiments had been conducted on base carbon Ferrium C64 prior to the design and construction of the DCGQ prototype unit to evaluate a slow transformation rate on C64's microstructure. Those preliminary results showed no noticeable difference between slowly and rapidly transformed martensitic microstructures. Figure 11 shows the microstructure, magnified 1000X, of a carburized (A) DCGQ processed coupon and (B) HPGQ processed coupon. As with previous experiments, there is no discernable difference between the two microstructures. The thin grey film on the DCGQ coupon is oxidized copper, which was removed prior to testing. The microhardness profiles for the coupons are shown in Figure 12 and confirm that the slow transformation rate did not alter the microstructure of the DCGQ processed coupons, compared to coupons processed using a standard 2-bar HPGQ process.



(A) (B)

Figure 11: Microstructure of a carburized coupon processed using (A) DCGQ and (B) HPGQ, magnified 1000X.

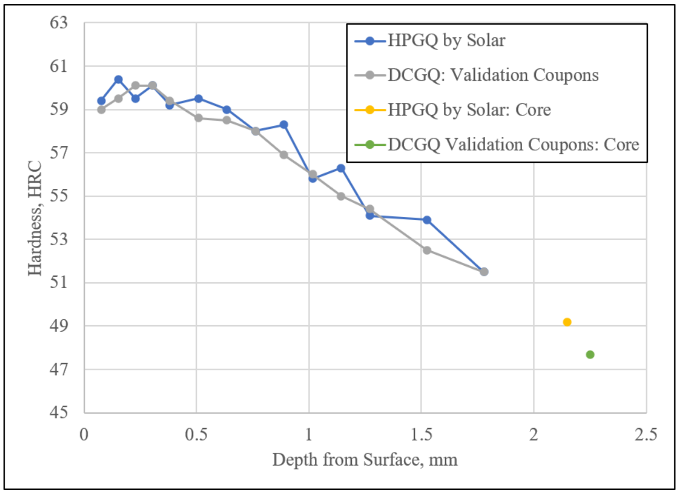


Figure 12: Hardness profile comparison of carburized coupons processed using DCGQ and HPGQ.

Mechanical Testing

Tensile testing and Charpy V-notch impact testing were conducted at room temperature to compare the mechanical properties of DCGQ processed C64 and HPGQ processed C64. Table 1 shows the results from these tests and reveal equivalent tensile and yield strengths, elongation and reduction of area, and impact energy for Ferrium C64 processed using HPGQ and DCGQ. These tests are promising, as they indicate that a slow martensitic transformation can be used for Ferrium C64 without degrading the microstructure or mechanical properties. Materials with similar M_S and tempering temperatures should also be suitable for DCGQ, though further testing is required.

Table 1: Tension and Charpy impact results comparing DCGQ and HPGQ processed Ferrium C64 coupons.

	Tensile Strength (MPa)	Yield Strength (MPa)	Elongation (%)	RA (%)	CVN Energy (J)
DCGQ	1627	1405	17.75	71.2	25.35
HPGQ	1625	1401	16.75	71.0	24.00

Residual Stress and Distortion

Residual stress profiles for a carburized coupon were compared between the two processes, at two locations, and are shown in Figure 13. There is no significant difference between the residual stress profiles. This, combined with the microstructural and mechanical property evaluations, bode well for fatigue performance being similar between parts subjected to the two different processes.

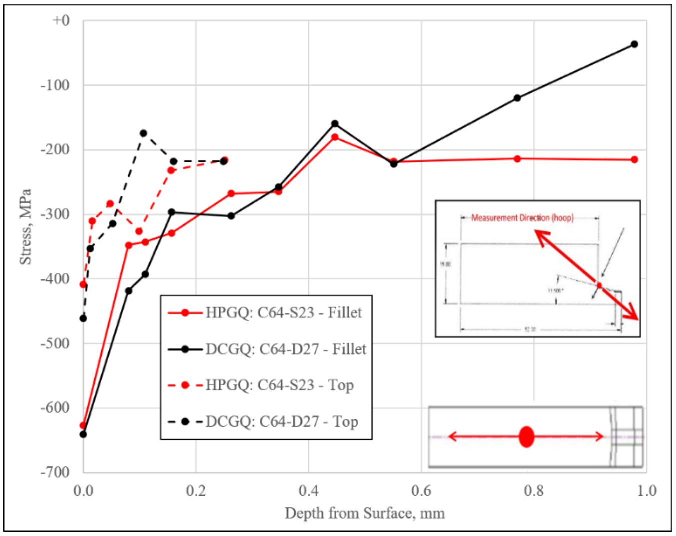


Figure 13: Residual stress profile comparison of carburized coupons processed using DCGQ and HPGQ.

Bend fatigue testing was conducted during this work; however, the results were impacted by surface oxidation on the DCGQ bend fatigue coupons. The oxidation was caused by the DCGQ coupons requiring open air processing with the prototype unit and was verified by SEM; production equipment can eliminate this problem by operating the process in a protective atmosphere. Although the DCGQ coupons were copper plated, the integrity of the copper was not maintained throughout the complete cycle, with damage likely occurring during the 1000°C austenitizing step, and subsequently worsened during the 500° C tempering cycle. The DCGQ coupons which did not fail after a few hundred cycles due to crack initiation at an oxide, would runout with stress levels comparable to HPGQ. Therefore, operating DCGQ under a protective atmosphere for all processing steps, should not render a decrease in in-service performance compared to HPGQ processed components.

The above discussion focused on showing that certain materials, particularly those which are tempered at temperatures below their M_S, have the potential to acquire a similar structure and properties after a relatively slow transformation from austenite to martensite, compared to a rapid transformation from austenite to martensite. However, the benefit of DCGQ is its capability to significantly reduce and control distortion of difficult to quench geometries. One such geometry was designed by DANTE Solutions to maximize nonuniform cooling and create large distortion. The coupon, shown in Figure 14, was used to show the distortion reducing capabilities of DCGQ compared to HPGQ.

Out-of-round distortion was the chosen mode of distortion to evaluate, due to its ease of measurement. A 50 mm eccentric bore was drilled through a disc having a 100 mm diameter and 100 mm height, creating a 6 mm thin section and 44 mm thick section. A bore gauge is used to compare the measurements of the bore at the location between the minimum and maximum thicknesses and at 90° to this location. The two measurements are made at five points along the height, as shown in Figure 14.

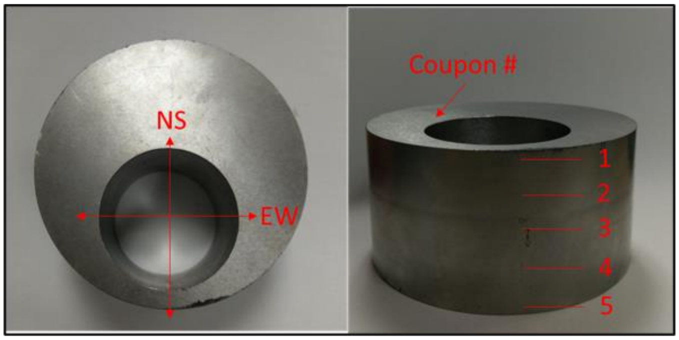


Figure 14: Eccentric bore coupon used to evaluate out-of-round distortion, showing the two directions used to define out-of-round (left) and the five axial measurement points (right).

Two (2) eccentric bore coupons were processed using HPGQ and two (2) using DCGQ. These coupons were processed alongside the mechanical test coupons. The out-of-round distortion results for the four coupons at the five axial locations, along with the average for each coupon, are shown in Table 2. For the given geometry and distortion mode, DCGQ reduced the distortion by 100 µm, or 50%, compared to HPGQ. This is significant, as the DCGQ recipe used was designed for the bend fatigue coupon, which has a thinner and more uniform cross-section. The recipe was designed as such to ensure testing was conducted using a DCGQ recipe representative of the geometry being tested. Slowing the DCGQ process down would yield less distortion of the eccentric bore geometry. Regardless, a 50% reduction in distortion was achieved when compared to the currently used processing conditions for Ferrium C64.

Table 2: Out-of-round distortion results comparing DCGQ and HPGQ processed Ferrium C64 coupons.

Axial Position	**Coupon DCGQ 4 (mm)**	**Coupon DCGQ 5 (mm)**	**Coupon HPGQ 1 (mm)**	**Coupon HPGQ 2 (mm)**
1	0.11	0.09	0.15	0.23
2	0.09	0.11	0.21	0.21
3	0.07	0.11	0.23	0.21
4	0.08	0.12	0.22	0.20
5	0.11	0.09	0.25	0.23
AVERAGE	**0.092**	**0.104**	**0.212**	**0.216**

DANTE simulations have been conducted on various geometries, using various DCGQ recipes, and it has been shown that it is possible to design a DCGQ recipe which completely eliminates the shape change distortion, with only the uniform volumetric size change from the initial to final volume microstructure occurring. Generally, this recipe takes too much time to be practical. However, the processing time can be reduced to determine acceptable distortion and processing time; the DCGQ recipe used to reduce distortion in the eccentric bore coupon by 50% was executed in one hour, where the recipe resulting in no shape change of this geometry, determined by DANTE modeling, requires seven hours to complete.

Conclusions

The DANTE Controlled Gas Quenching (DCGQ) process has the potential to process difficult to quench part geometries without the use of expensive press quench tooling and reduce the amount of post-heat treatment processing required. The work presented here concluded that it is possible to control the temperature of quench gas entering a quench vessel, at atmospheric pressure, in order to follow a time-temperature recipe required to control the martensitic transformation rate in high hardenability steel alloys. The prototype unit constructed was able to achieve great control within the temperature range of 400 – 100° C, using varying rates of temperature change. The work further concluded that for Ferrium C64, the relatively slow transformation rate from austenite to martensite did not alter the microstructure, mechanical properties, or residual stress when compared the current standard quenching process. Furthermore, DCGQ was shown to significantly reduce distortion in a difficult to quench geometry when compared to HPGQ.

Besides having the capability to significantly reduce distortion for difficult to quench part geometries, DCGQ has also been shown, through the use of DANTE modeling, to be less sensitive to nonuniform convective cooling conditions created by equipment design. The DCGQ process can also result in more dimensionally consistent components. All of DCGQ’s benefits are a result of controlling the martensitic transformation. By controlling the martensitic transformation throughout the part, it is possible to reduce the nonuniform cooling effect created by geometry and equipment design, ensuring that the transformation proceeds in a consistent way, part after part. Consistent and predictable distortion allows the part’s pre heat treatment configuration to be redesigned such that the post-heat treated shape is within design tolerances and a minimal amount of post-heat treatment processing is required.

Acknowledgments

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