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Study of the effects of Thermal Aging coupled with Shot-peening Treatment of Carbonitrided DIN 1.6587 Low Alloy Steel on Retained Austenite and Residual Stresses

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ABSTRACT

This work investigated the effects of thermal aging and shot-peening of carbonitrided 1.6587 steel grade on retained austenite and residual stresses. After carbonitriding samples contained maximum of 0.87% carbon, 0.34% nitrogen and 50 mass.-% retained austenite. Thermal aging conditions investigated were -30°C, 90°C and 150°C while the aging period were 14, 96 and 720 hours. After thermal aging, some of the samples were subjected to shot-peening treatment using standard cut wire shots StD – G3 - 0.6 mm VDFI 8001 with intensity in the range of 0.25 to 0.30 mmA and coverage of 1.00 to 1.24 x 98%. It was found that the quantity and size of finely disseminated precipitates increase with increasing aging temperature and time accompanied by darkening of martensite plates. The magnitude and distribution of hardness in the case layer was highly influenced by aging conditions with an increase of up to 50 HV for peak hardness and up to 120 HV at a depth of 50 µm on aging at -30°C. Aging at 90°C, hardness distribution remains relatively unaffected while at 150°C for 720 hours the magnitude of hardness is slightly improved mainly to decomposition of retained austenite to bainite. Aging at 90°C, retained austenite remains relatively stable and readily transforms on aging at -30°C and 150°C for 720 hours; transformation of up to 20% of the initial retained austenite can be reached which enhances compressive residual stresses of up to -120 MPa in retained austenite and up to -250 MPa in martensite phase. Aging coupled with shot-peening treatment induces transformation of up to 48% of the initial retained austenite and enhances compressive residual stresses of up to -1200 MPa in both phases. It can be concluded that thermally aging coupled with shot-peening treatment reduces retained austenite and significantly enhances residual stresses which is essential to improved ductility and fatigue life of carbonitrided parts.

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INTRODUCTION

Carbonitriding is one of the thermochemical treatments which simultaneously diffuses carbon and nitrogen

interstitial atoms into the subsurface of the steel part being treated. The carbonitriding process has been extensively studied (Katemi and Epp, 2022; Katemi and Epp,

2021; El-Hossary et. al, 2001) as an alternative method to carburizing which improved (Katemi and Epp, 2022; Katemi and Epp, 2021; El-Hossary et al, 2001) as an alternative method to carburizing, improving surface mechanical properties for power transmission components. The process is considered to be more advantageous over carburizing process mainly due to the presence of nitrogen atoms that lead to improved hardenability, resistance to wear and softening at elevated service temperature, more advantageous than the carburizing process mainly due to the presence of nitrogen atoms that lead to improved hardenability, resistance to wear and softening at elevated service temperature, and higher fatigue limits (Herring, 2011). Further, carbonitriding process is carried at lower temperature of about 850°C and shorter times which renders less grain coarsening and less distortion during quenching.

In the last two decades, substantial work has been carried to investigate the effect of carbon and nitrogen potentials of carbonitriding atmosphere and resulting oxidation/porosity defects (Ohki, 2006; Bischoff et.al, 2010; Winter, 2011), analysis of phase compositions and residual stresses in both phases (Katemi et.al, 2014) as well as its influence on mechanical properties (Kukurova et. al, 2013; Nasser et. al, 2023). The magnitude of residual stresses and mechanical properties of the case-hardened layer highly depend on retained austenite after quenching and post-treatments.

In the previous work of the author (Katemi and Epp, 2021), it has been found that depending on carbonitriding conditions and the nitrogen content attained, as high as 70% of austenite can be retained after quenching and tempering stage. Such high amount is not always welcomed as may negatively affect mechanical properties. In view of this, thermal stability during aging of retained austenite or its reduction by cryogenic or mechanical treatment is key to avoiding distortion of carbonitrided parts

while in ser-vice; particularly in close fittings. The extensive studies on aging and tempering process of carbonitrided parts focused on precipitation behavior of ϵ -like and α' -like precipitates (Cheng et al., 1990; Cheng et. al, 1992). However, limited information is available describing the effect of thermal aging coupled with shot-peening treatment on retained austenite and residual stress distributions of carbonitrided parts on the effect of thermal aging coupled with shot-peening treatment on retained austenite and residual stress distributions of carbonitrided parts is available.

This paper focuses on analysis of the effects of different aging temperatures and times on retained austenite and distribution of residual stresses in the case layer. Further, the work investigates thermal aging coupled with mechanical (shot-peening) treatment of retained austenite and residual stress distribution by shot-peening treatment. Additional analysis includes chemical composition, metallograph and hardness.

METHODS AND MATERIALS

Materials and Carbonitriding Treatment

The material used for this work was DIN1.6587 alloy steel with standard and OES determined chemical composition given in Table 1. Samples with a diameter of 34 mm and a thickness of 8 mm were machined from the 35 mm diameter rod. Then, the samples were subjected to a gaseous carbonitriding treatment to attain a pre-determined surface carbon and nitrogen content of 0.87 and 0.40 mass.-%, respectively and with the target of retaining high amount of austenite of about 50 mass.-% after quenching. The details of the carbonitriding treatment have been presented in previous work of the author (Katemi and Epp, 2019). After carbonitriding, samples were quenched into oil held at 60 °C for 15 minutes followed by conventional tempering at 170°C for 2

hours before being subjected to ageing and mechanical treatment.

Table 1: DIN 1.6587 Steel grade and OES determined chemical composition

Element [Mass.-%]	C	Cr	Ni	Mn	Mo	Si	Fe
(DIN 1.6587)	0.15–0.21	1.50–1.80	1.40–1.70	0.50–0.90	0.25–0.35	<0.40	Bal
OES composition	0.157	1.65	1.57	0.517	0.259	0.269	Bal

Aging conditions

After carbonitriding and conventional tempering, samples were subjected to different aging conditions (*temperature and time*) to study the microstructure changes such as decomposition of retained austenite and precipitates formations, and evolution of residual stresses which are likely to occur due to thermal loading in service. Table 2 presents the accelerated aging conditions considered for this study. The temperature of -30°C was attained using a controllable refrigerator.

Table 2: Aging conditions

No.	Temperature [°C]	Time [h]
1	-30	14 96 720
2	90	14 96 720
3	150	14 96 720

Shot-peening treatment

The mechanical treatment conducted was shot-peening treatment. This was conducted to investigate mechanical stability of retained austenite, induced plasticity transformation, and enhanced compressive residual stresses in both retained austenite and martensite phase. The aging conditions for samples subjected for shot-peening treatment are given in Table 3 whereas the shot-peening parameters are provided in Table 4.

Table 3: Aging conditions of samples subjected for shot-peening treatment

Temperature [°C]	Time [h]
-30	96 720
90	96 720

150	96	720
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Table 4: Shot-peening parameters

Blasting	StD – G3 -0.6 mm VDFI 8001
Almen intensity	0.25 – 0.3 mmA
Coverage	1.00 – 1.24 x t 98%

Determination of Phase composition and residual stresses by X-ray Diffraction

The diffractometer MZ VI E (GE Inspection Technology) was used to acquire diffraction patterns for analysis of phase compositions and residual stress distributions in both retained austenite and martensite phases. For the measurements, Cr-K α radiations ($\lambda_{\alpha 1}=2.28975 \text{ \AA}$) produced by a standard sealed X-Ray tube operated at 33 kV and 40 mA was used. The primary beam was defined by a collimator with 2 mm in diameter and vanadium was used as a filter for Cr-K β radiations. Phase analysis was performed using the Rietveld Method (Topas 4.2, Bruker Axs) using fundamental parameter refinement approach. The standard $\sin^2\psi$ method which assumes no macroscopic stress ($\sigma_{33\text{macro}} = 0$) perpendicular to the plane surface was used for analysis of residual stress distributions in both phases. For full details of acquisition of diffraction patterns for phase composition and residual stress analysis and analysis technique, the reader is referred to the author's previous works (Katemi and Epp, 2019).

RESULTS AND DISCUSSION

Carbon and nitrogen profiles and retained austenite after carbonitriding treatment

The carbon and nitrogen depth profiles after carbonitriding was determined using Optical Emission Spectrometry. The maximum surface concentration of the

carbonitrided samples were 0.87 mass.-% carbon and 0.36 mass.-% nitrogen. After quenching and temperature at 170°C for 2 hours, the initial amount was about 50 mass.-% at a depth of 50 microns from the surface. Low amount of austenite was retained in the first 50 μm which is attributed to subsurface oxidation and depletion of alloying elements

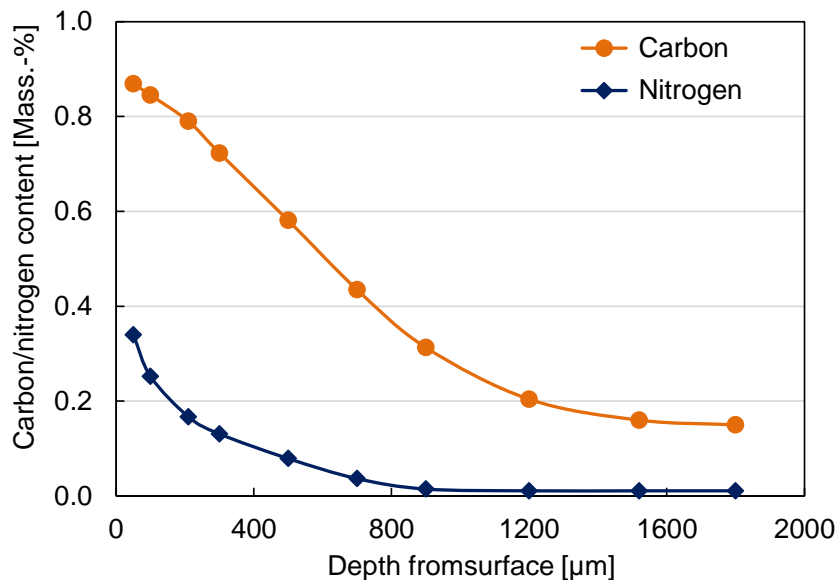


Figure 1: Carbon and nitrogen depth profiles after carbonitriding treatment.

Microstructures and microhardness distributions

Typical microstructures of the case layer and core microstructures after the extreme accelerated aging different conditions (720 hours) are presented in Figure 2. The case microstructures are characterized by plate martensite (dark), retained austenite (white) and finely disseminated precipitates of carbides and nitrides. On aging at different temperatures reveals no significant difference in the size of martensite plates and fraction of retained austenite. However, darkening of martensite plates on aging at 150 °C for 720 hours is observed, which can mainly be attributed to precipitation in martensite plate. Figure 3 shows unetched case

microstructures of the same samples characterized by subsurface inter- and/ or intra-granular oxidation which is common for carbonitriding treatments carried out under endothermic atmosphere (Sastry et. al, 1982).

Further, finely disseminated precipitates can clearly be observed. Under the same aging temperature, the quantity and size of precipitates increase with increasing aging temperature. On other hand, core microstructures which are essentially bainitic were hardly affected by aging conditions due to the fact that much of carbonitride precipitation or clustering has taken place by auto tempering during quenching process.

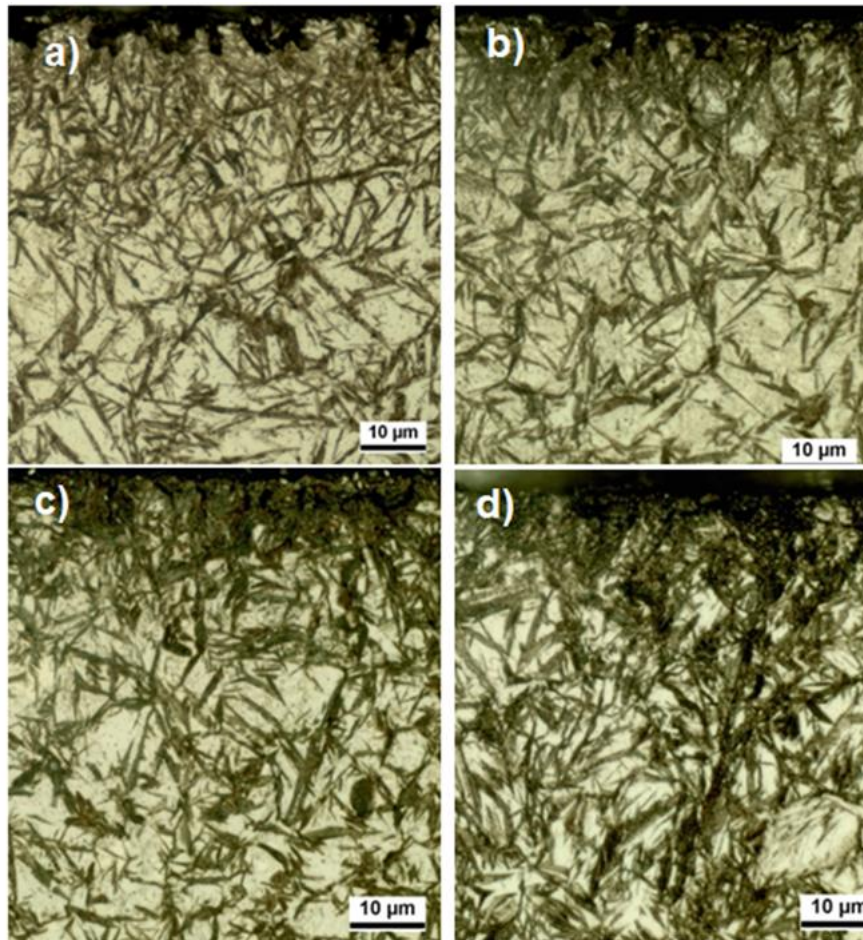


Figure 2: Typical Case and Core microstructures of samples after aging for 720 h (30 days) at: (a) conventional tempering (170°C/2 h); (b) -30°C; (c) 90°C; (d) 150°C.

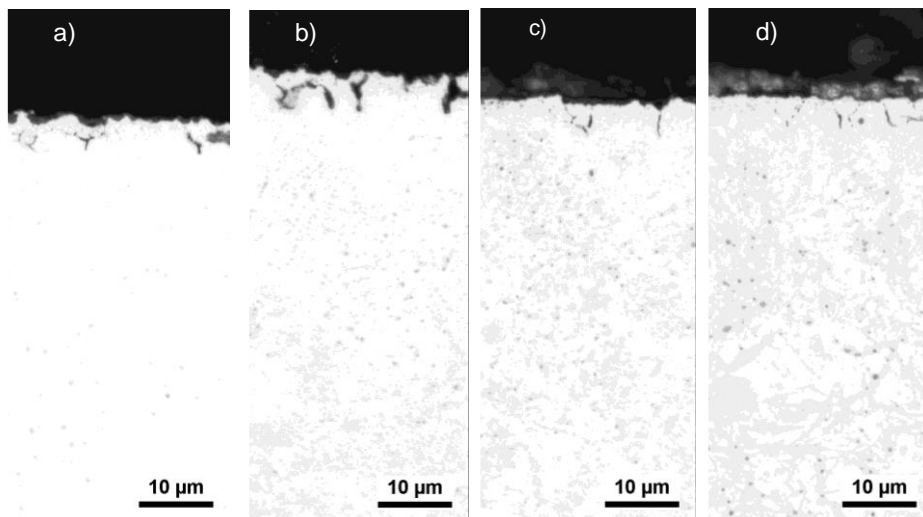


Figure 3: Unetched case microstructures after aging for 720 h (30 days) at: (a) RT; (b) -30°C; (c) 90°C; (d) 150°C.

Figure 4 illustrates the microhardness distributions after aging at different conditions. Generally, microhardness distribution, magnitude of peak hardness and location of peak hardness, and hardness

values in the first 400 microns from the surface are highly influenced by aging conditions. As expected, conventional tempering at 170°C for 2 hours lowers the peak hardness and a drop of up to 70 HV1

can be reached (Figure 4a). Aging at -30°C enhances hardness and shift the location of peak hardness toward the surface mainly due to continued martensitic transformation on cooling to -30°C . An increase of up to 50 HV for the peak hardness and of up to 120 HV at a depth of 50 μm from the surface are reached. Further, aging at -30°C , aging times seem to have negligible influence on the magnitude and distribution of hardness mainly because martensitic transformation is time independent transformation process. On aging at 90°C (Figure 4c) and presumably at RT, the magnitude and hardness distribution remain relatively unaffected. In contrast, aging at 150°C , the peak hardness and the magnitude of hardness in the first 50 microns highly depend on the aging time. Aging at this temperature for 720 hours decreases the peak hardness by 30 HV while improving the hardness at a depth of

50 μm by 30 HV. The enhancement of hardness can be connected to decomposition of retained austenite to bainite.

In all cases, the core hardness seems to be less affected by the aging conditions and remains slightly below 450 HV. Aging temperatures to 150°C reveal little effect on the martensitic core hardness of the carbonitrided low alloy steel as much carbide precipitation has taken place by auto-tempering. The core is essentially characterized by bainitic microstructure which is less affected by tempering temperatures up to 300°C . On the other hand, the hardness of the case layer is considerably affected as aging temperature exceeds 100°C when η -carbides precipitate, even more at 150°C when precipitation is more advanced and some of the retained austenite might transform (Parrish, 1999).

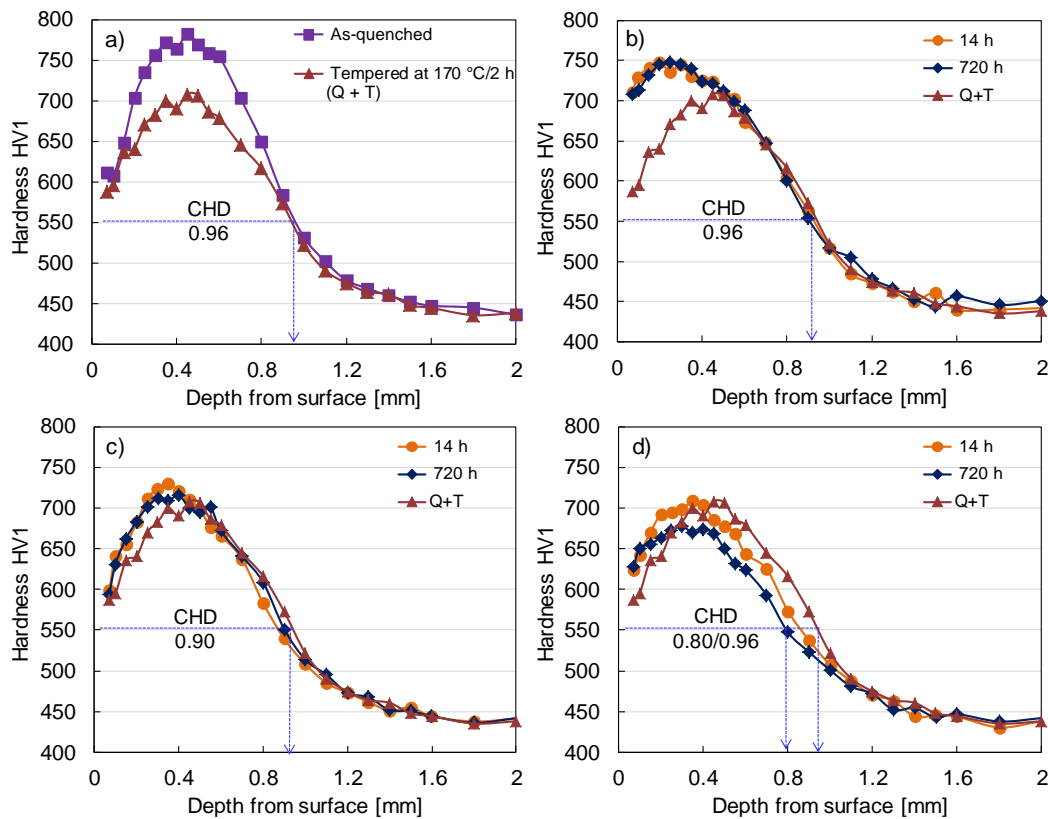


Figure 4: Microhardness distributions after aging for 14 h and 729 h at: a) Initial conditions; b) -30°C ; c) 90°C ; d) 150°C

Effect of aging time and temperature on retained austenite distributions

Figure 5 presents typical examples of variation of X-ray diffraction depth profiles collected after aging at different aging conditions. For all aging conditions, the intensity of retained austenite reflections $\gamma\{200\}$ and $\gamma\{220\}$ decrease with depth

reflecting the decrease in volume fraction of retained austenite with depth. On the other hand, the intensity of martensite/ferrite reflections $\alpha'\{200\}$ and $\alpha'\{211\}$ increase with increasing depth from the surface. Analysis of diffraction patterns collected at a depth of 50 μm after tempering gave 50 mass.-% as initial volume fraction of retained austenite.

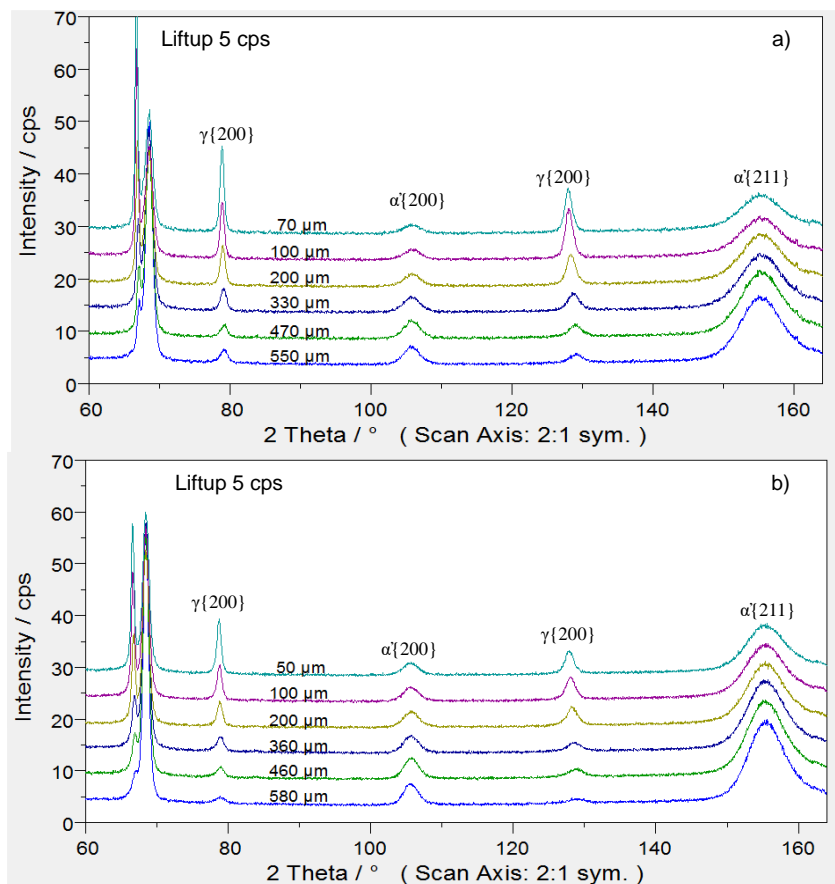


Figure 5: X-ray diffraction profiles after aging for 720 h at: a) 90°C; b) 150°C

Figure 6 shows retained austenite distributions after aging for 14, 96, and 720 hours at different temperatures. It can be seen from this figure that on aging at RT (Figure 6a) and at 90 °C (Figure 6c), no significant changes in fraction of retained austenite occur with aging time. At these temperatures, the retained austenite remains relatively thermally stable with discrepancies within the marginal scattering band. In the work of Neu and Sehitoglu (Neu and Sehitoglu, 1993) on carburized 4320 steel, it has been reported that thermally-induced transformation of about 20% of the initially retained austenite would take a year on isothermal holding at 90 °C.

On aging at -30 °C, the initial fraction of retained austenite is reduced by 10 mass.-% from about 50% to 40%. This is not only linked to the undercooling by -30 °C but also the aging time. On aging at 150 °C for aging time up to 96 h, retained austenite remains relatively thermally stable. On further holding for 720 hours at this temperature, retained austenite is significantly drop by 12% from 50 to 38%. According to Neu and Sehitoglu (Neu and Sehitoglu, 1993), such a decrease should take 1 day on isothermal holding at 150°C. The transformed retained austenite mainly transforms to bainite leading to slight

increase of compressive residual stresses in both phases and varies in the range of -25 to -50 MPa. Wang et al. (Wang et. al, 1986)

report that such improvement in compressive residual stresses can be linked to formation of carbides/nitrides.

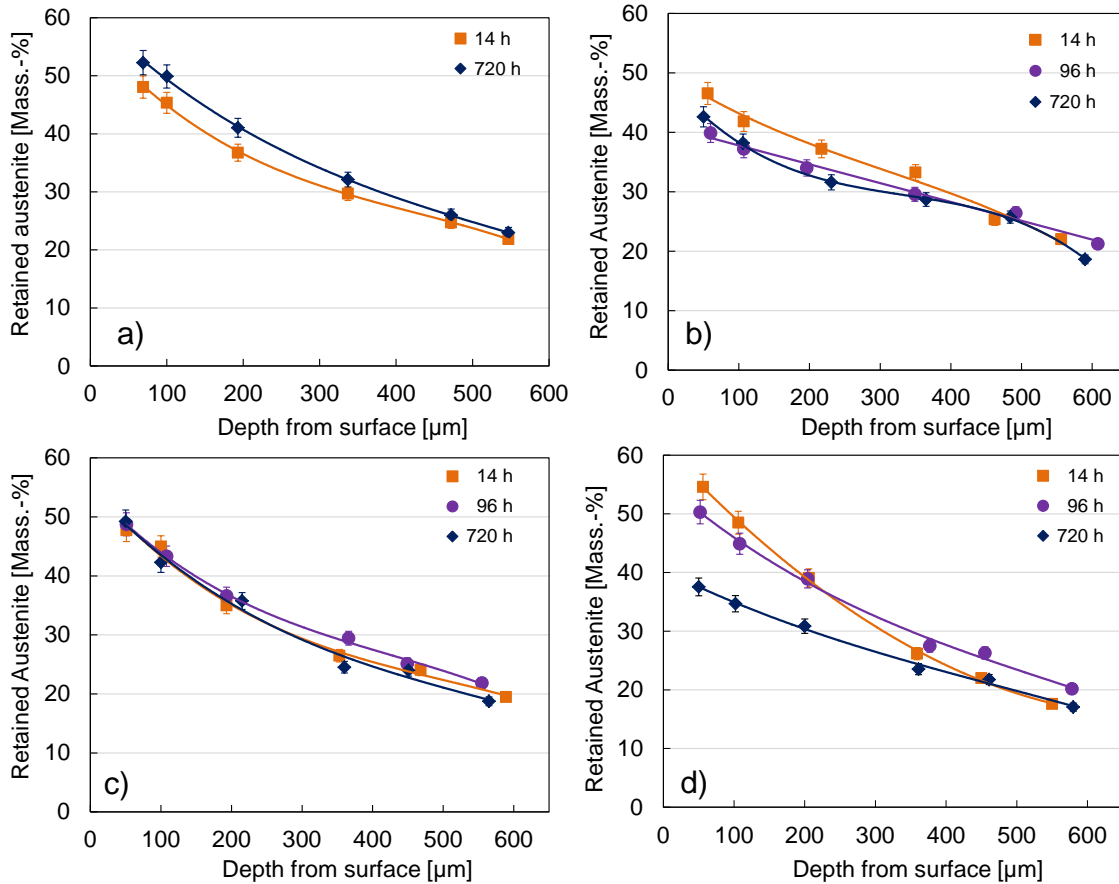


Figure 6: Retained austenite distributions after thermal cycles for 14 and 720 hours at different temperatures: a) RT; b) -30 °C; c) 90 °C; d) 150 °C.

Effect of aging conditions on residual stress distributions

Residual stress distributions in both retained austenite and martensite phase after at different aging conditions are given in Figure 7 through Figure 9. In addition, the graphs incorporate macro stresses distributions determined considering the volume fraction and magnitudes of residual stresses of each phase. It has to be pointed out that aging treatments were conducted after a conventional tempering at 170 °C for 2 hours in which residual stresses at a depth of 50 microns from surface was about -50 MPa in martensite phase while in retained austenite phase was about 0 MPa (Katemi and Epp, 2014). As observed from Figure 7 through Figure 9, aging conditions influence considerably the magnitudes and

distribution of residual stresses in both phases.

Aging at -30 °C for 14 hours considerably enhances the compressive residual stresses in both retained austenite and martensite phase with peak compressive residual stresses occurring in the first 50 μm from the surface. The volume expansion associated with the formation of new martensite would be constrained, and compressive stresses would be increased. Residual stresses up to about -120 MPa in retained austenite and -250 MPa in martensite phase is reached (Figure 7). The improved compressive residual stresses are mainly associated to the transformation of retained austenite to martensite resulting in volume increase in the case. Holding isothermally at -30°C for 720 hours

substantially alters the residual stress distribution pattern and relaxes the compressive RS in the first 100 μm from the surface. A relaxation of up to -55 MPa for retained austenite and up to -90 MPa for martensite (Figure 7b) occurred mainly due to clustering and segregation of carbon and nitrogen leading to areas with high dislocation density; consequently, loss of martensite tetragonality. No significant changes were observed on aging at 90°C for 14 and 720 hours in terms of residual stress distributions. However, a slight enhancement of compressive residual stress

of up to about -40 MPa in martensite phase can be observed. In contrary, aging at 150°C for 720 hours alters residual stress distributions and slightly improves compressive RS in both phases which varies in the range of 0 to -100 MPa (Figure 9b). The slight increase in compressive RS can be attributed to thermal-induced volumetric transformation strain. Such thermal induced transformation can be clearly observed in Figure 6d during which about 20 mass.-% of the initial amount of retained austenite (50 mass. -%) transformed.

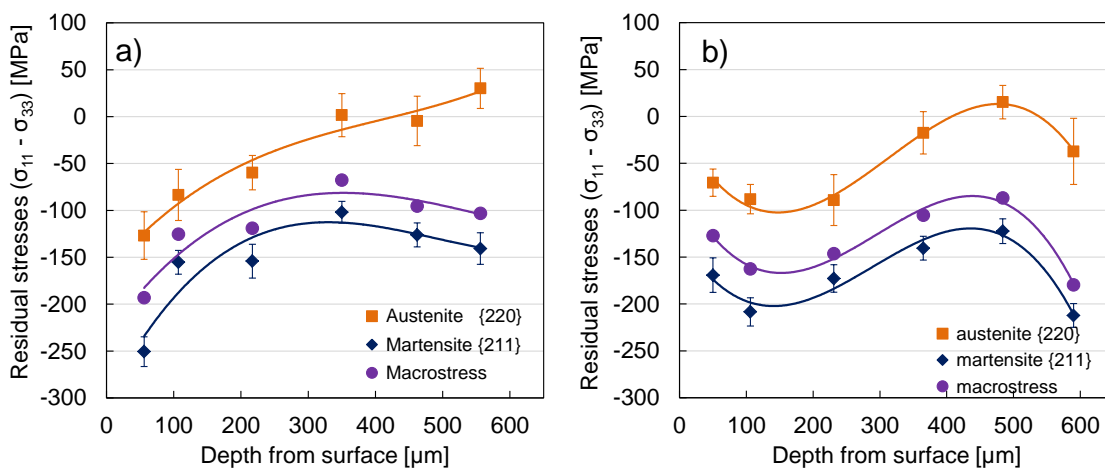


Figure 7: Residual stress distributions in retained austenite {220} and martensite phase {211} after aging at -30°C : a) 14 h; b) 720 h

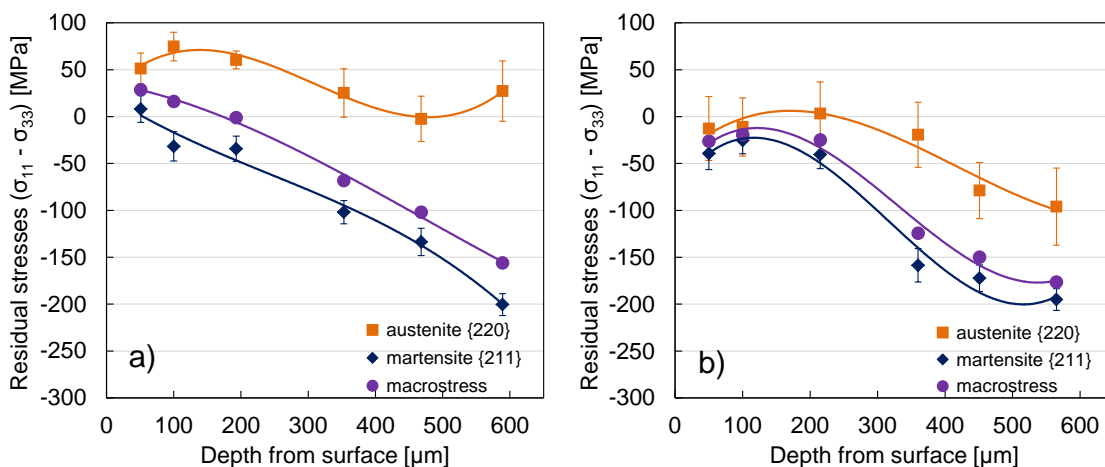


Figure 8: Residual stress distributions in retained austenite {220} and martensite phase {211} after aging at 90°C: a) 14 h; b) 720 h

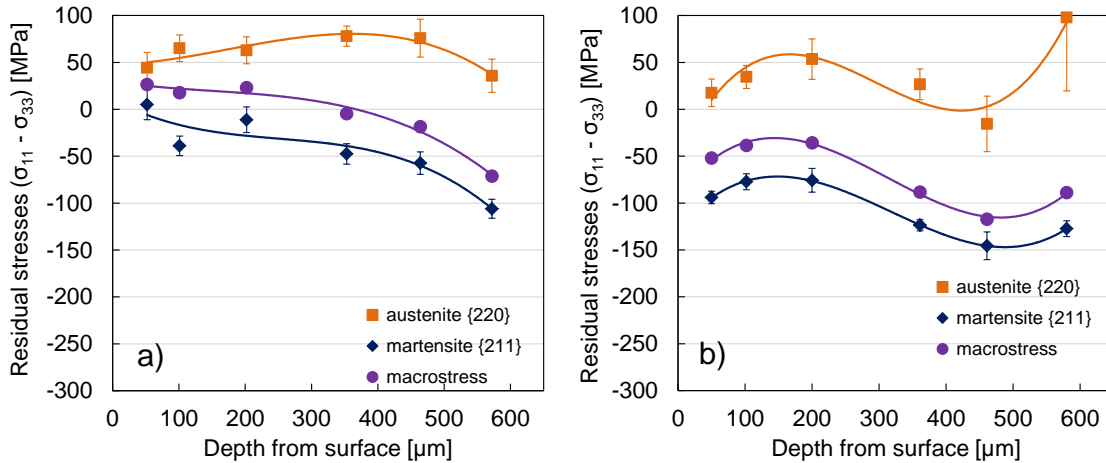


Figure 9: Residual stress distributions in retained austenite {220} and martensite phase {211} after aging at 150°C: a) 14 h; b) 720 h.

Mechanical stability treatment

In this case, the mechanical stability treatment conducted was shot-peening treatment aimed to investigate mechanical stability of retained austenite and enhancement of compressive residual stresses in both retained austenite and martensite phase as a result of induced plasticity transformation. In this case, prior to mechanical stability treatment the samples were thermally treated at various conditions. Samples aged at -30°C, 90°C, and 150°C whereas for 96 and 720 hours subjected to shot-peening treatment

Effect shot-peening treatment on retained austenite

Figure 10 compares volume fractions of retained austenite at a depth of 50 μm from the surface for as-quenched sample, tempered (CT) + aged sample, and aged plus shot-peened (SP) sample. As observed from this figure, shot-peening treatment induces further transformation of retained austenite to martensite. The fraction of transformed retained austenite ranges between 19% for samples aged at -30°C and 46% for samples aged at 150 °C. This implies that samples aged at -30°C are more mechanically stable and that the stability decreases with increasing temperature. Increasing the aging temperature raises the dislocations mobility

leading to the decrease in dislocation density; consequently, retained austenite becomes unstable and can readily transform.

It is also important to note that there is essentially no difference in mechanical stability of retained austenite for samples thermally aged -30°C for 96 and 720 hours. Similar behaviour is observed for samples thermally aged at 90°C. It is reasonable to assume that aging at -30°C and 90°C has little contribution to the mechanical stability of retained austenite because clustering/segregation of carbon and nitrogen atoms and carbide precipitation had taken place during prior conventional tempering at 170°C for 2 hours. On the contrary, a difference in the amount of transformed RA is observed on aging at 150°C for 96 and 720 hours. The higher amount of transformed retained austenite observed at 720 hours is attributed to the transformation of RA to bainite during thermal stabilization. In the work of Neu and Sehitoglu (Neu and Sehitoglu, 1993), a decrease of about 20% of the initial amount of retained austenite should take a day on isothermal holding at 150 °C. Additionally, such difference in transformed retained austenite may be associated to inhomogeneous deformation since the amount transformed depends on the extent of deformation.

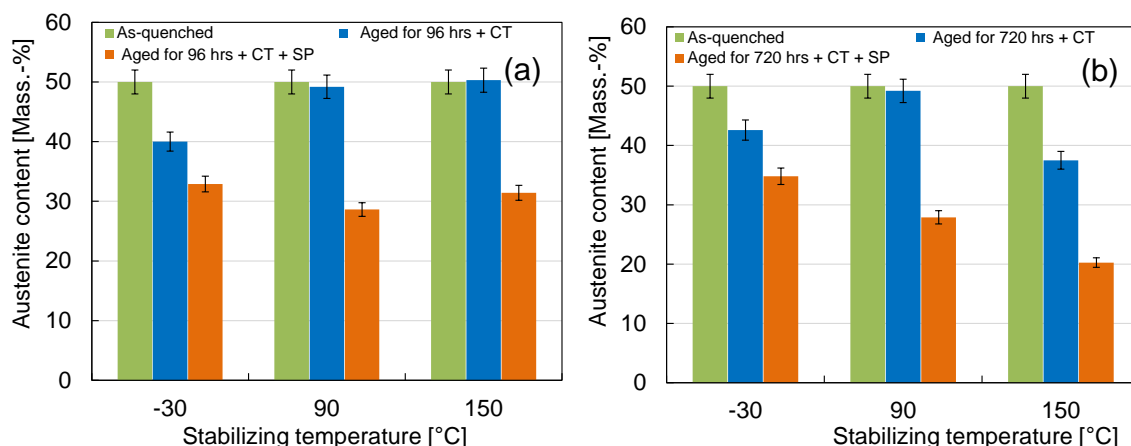


Figure 10: Volume fraction of retained austenite for as-quenched, aged + CT, and aged + CT + SP: (a) 96 h; (b) 720 h: CT – conventional tempering, SP: shot-peening

Effect of shot-peening treatment on residual stresses

Figure 11 and Figure 12 compare the residual stresses in retained austenite and martensite phase measured at a depth of 50 μm for thermally aged samples coupled with shot-peening treatment. As expected, shot-peening treatment induces high compressive residual stresses (RS) in both phases. After shot-peening, as high as -1200 MPa in retained austenite can be reached for samples aged for 720 hours whereas in martensite phase as high as -1200 MPa can be attained for samples aged for 96 hours. The level of compressive residual stresses induced after shot-peening are in good agreement with those observed

while investigating the effect of shot-peening parameters on residual stresses (Wu et.al, 2021; Lin et. al, 2020). The increase in compressive residual stresses is mainly attributed to the deformation-induced transformation of retained austenite to martensite leading to an increase in volume. The constraint of the associated volume expansion causes the development of high magnitude of compressive residual stresses and as a result improves the bending fatigue perform (Davis et. al, 2002). The improved bending fatigue results due to the fact that the induced compressive RS within the surface of the treated parts reduces the possibility of crack initiation at the surface.

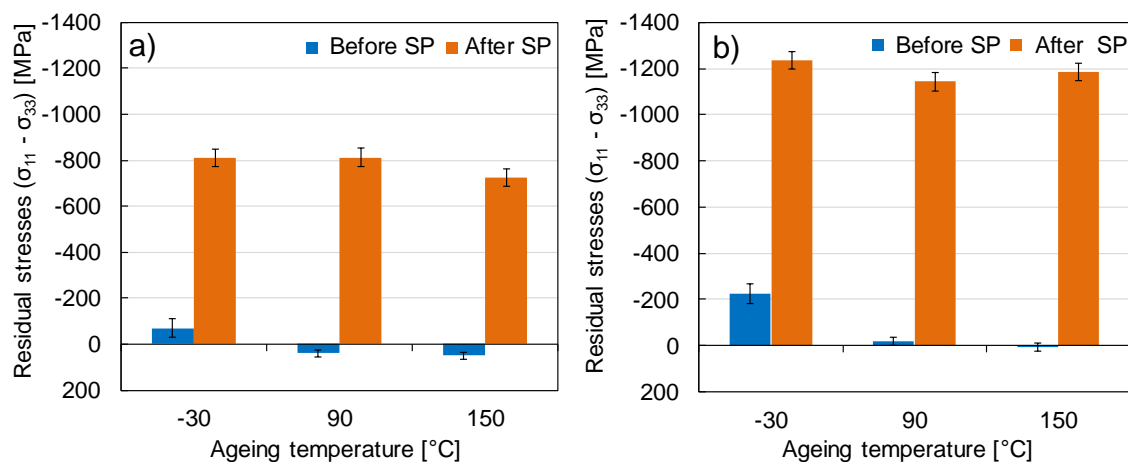


Figure 11: Residual stresses in retained austenite after shot-peening (SP) of samples with prior thermal treatment at different temperatures for: a) 96 h; b) 720 h

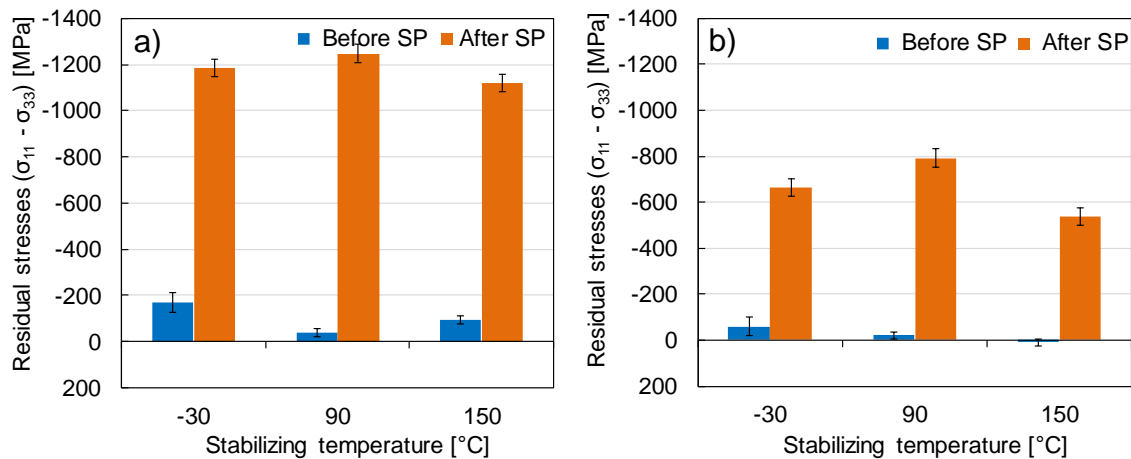


Figure 12: Residual stress in martensite after shot-peening (SP) of samples with prior thermal treatment at different temperatures for: a) 96 h; b) 720 h

CONCLUSION

This work investigated the effects of thermal aging and shot-peening of carbonitrided 1.6587 low alloy steel retained austenite and residual stresses distribution considering thermal aging conditions of -30°C, 90°C and 150°C with aging period of 14, 96 and 720 hours (1 month). It was found that the quantity and size of finely disseminated precipitates increase with increasing aging temperature and time. Darkening of martensite plates on aging at 150 °C for 720 hours is observed, which is linked to precipitation in martensite plate. On aging at 90°C, retained austenite remained relatively thermally stable. Aging at -30°C and 150°C retained austenite decomposes mainly due to continued martensitic transformation and transformation of retained austenite to bainite, respectively; consequently, enhances case-layer compressive residual stresses in both martensite and retained austenite phase. Aging at -30°C enhanced compressive residual stresses of up to -120 MPa in retained austenite and up to -250 MPa in martensite phase while aging at 150°C slightly improves compressive RS in both phases in the range of 0 to -100 MPa. Shot-peening treatment of thermally aged carbonitrided samples readily transforms retained austenite to martensite and improves compressive residual stresses in both retained austenite and martensite phase. The fraction of transformed retained

austenite on aging for 720 hours at -30 °C, 90 °C, and 150 °C was as high as 48%, 40%, and 34%, respectively. This in turn enhances compressive residual stresses and as high as -800 MPa in retained austenite and -1200 MPa in martensite phase can be reached.

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