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Impact of various heat treatments on the microstructure evolution and mechanical properties of hot forged 18CrNiMo7-6 steel

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Abstract

Carburizing is a method of enhancing the surface properties of components, primarily made from low to medium carbon steels, such as shafts, gears, bearings, etc. Carburized parts are generally quenched and tempered before being put into service; however, after quenching of carburized parts further annealing and hardening treatments can be employed before final tempering. This work analyses the impact of the two aforementioned heat treatment approaches on the development of subsequent microstructures and mechanical properties of hot forged 18CrNiMo7-6 steel. Moreover, this study aims to understand the impact of normalizing treatments prior to the two aforementioned heat treatment routes. Microstructural and mechanical tests were conducted on four as forged flat cylinder components that received a combination of the above-mentioned heat treatments. In general, better microstructure refinement, in terms of prior austenite grain size (PAGS), was obtained for carburized parts that received the intermediate annealing and hardening treatments after quenching and prior to the final tempering. Additionally, further refinement of the martensitic pockets/blocks was observed for parts that did not receive a normalising treatment prior to carburisation. The studied heat treatments appear to have a negligible effect on the mechanical properties of the hot forged flat cylinder components.

Introduction

Carburization is a widely used process for surface hardening of steels with low to medium carbon content where the same level of hardening cannot be achieved by conventional quenching and tempering. In this process, the component is subjected to a high carbon containing environment such as carbon monoxide, at a temperature above the austenitic phase transformation temperature. During this process, the carbon from the (carbon rich) environment diffuses into the surface of the component. This results in a thin, hard carburized layer on the surface of the component with a very high carbon content. The depth of this carburized layer depends on the carbon potential of the environment and the dwell time of the component submerged in that environment. Upon quenching, a hard case of martensitic microstructure develops on the surface of the parts due to the high amount of carbon diffused into the case. However, as the core of the material has a lower carbon content as well as a slower cooling rate, a softer and relatively ductile bainitic, martensitic or ferritic-pearlitic microstructure can develop in the core. Such a combination of microstructures is desirable for applications where higher toughness and impact resistance is required along with good core strength such as in armours, shafts, bearings, gears etc (1).

Due to the complexity of the controlling parameters in carburization, there has been relatively little work on the influence of process variables during the surface hardening process (2). One of the most important parameters affecting the mechanical properties of the carburised component is the process of quenching which governs the transformation of the austenite to martensite or bainite. Carburized parts may be either cooled to room temperature after carburizing and reheated for subsequent hardening or directly quenched from the carburizing temperature. In this work, four different heat-treatments were applied to the cylindrical shaped forged components of 18CrNiMo7-6 steel. The heat-treatments were chosen in order to understand the effect of the normalising treatment before carburisation, where the main purpose of normalising is to condition the component such that it responds satisfactorily to the hardening operation. Additionally, the effect of the above mentioned, two different quenching methodologies after carburisation were investigated in relation to the mechanical properties of this case-hardened steel.

Experimental Methods

The material used for the study was 18CrNiMo7-6 steel; the chemical composition of the steel is presented in Table 1. 18CrNiMo7-6 steel is a low carbon martensitic steel widely used in the manufacture of machine parts, shafts, toothed wheels etc. These components operate under high pressure, high impact, wear prone applications and therefore require a hard surface layer along with a relatively ductile core.

Table 1: Chemical composition of 18CrNiMo7-6 steel (3)

Element	C	Si	Mn	Cr	Ni	Mo	Fe
Wt. %	0.18	0.20	0.70	1.65	1.55	0.30	Balance

The material was received as cylindrical shaped preforms in the spheroidized and annealed condition. The preforms were forged to flat cylindrical shaped components at 1100ºC using an in-house Schuler screw press. A photograph of the preform and the forged cylinder is shown in Figure 1. The dimension conformity of the components were checked after forging and four flat cylinder components from one batch of forgings were supplied for this study. The components were subjected to four different carburising heat-treatments (forged flat cylinders are hereafter referred to parts 1 – 4) as stipulated in Table 2 below. The heat-treatment operation was outsourced to an external company.

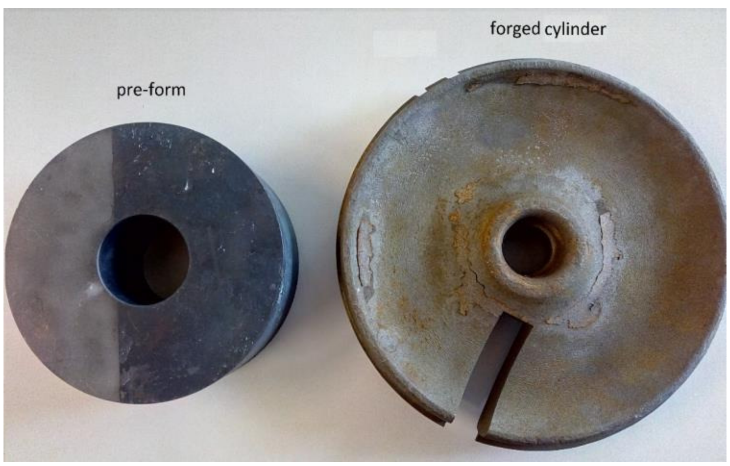

Figure 1: Image of the preform and the forged 18CrNiMo7-6 flat cylinder component (no scale bar given due to IP restriction)

After completion of the heat-treatments, a pair of cylindrical blank specimens were extracted from the centre of each of the components. The blanks were machined to the shape of tensile test specimens using an EDM machine. Two room temperature tensile tests were conducted for each part using Zwick 250 mechanical testing equipment. Strain during the tensile tests was measured using an extensometer placed directly at the gauge length of the specimen.

The remaining forged parts were sectioned using a Buehler Abrasimatic 300 abrasive wheel and a rectangular block of material was extracted from each of the forged parts. This block of material was then used to extract specimens for metallographic preparation and XRD analysis. The metallographic samples were used for microstructure analysis and hardness measurements.

Table 2: Different carburising treatments applied to the forged 18CrNiMo7-6 flat cylinder components

<table>
<tr><th rowspan="2">Heat-treatment ID</th><th rowspan="2">Part No.</th><th colspan="2">Heat Treatment</th></tr>
<tr><th>Normalising heat treatment
(Prior to carburising)</th><th>Carburising heat treatment</th></tr>
<tr><td>HT 1</td><td>Part 1</td><td>875°C for 30 mins + Air Cool</td><td rowspan="2">Carburising at 930°C until a 2.6 mm thick carburised layer is formed
Cool to 820°C and hold for 1 hour + Oil quench
Anneal at 670°C for 2 hours + Air cool
Harden at 800°C for 30 minutes + Oil quench
Sub Zero treatment at -80°C for 90 minute
Temper: 200°C for 2 hours + Air cool</td></tr>
<tr><td>HT 2</td><td>Part 2</td><td>Not applied</td></tr>
<tr><td>HT 3</td><td>Part 3</td><td>875°C for 30 mins + Air Cool</td><td rowspan="2">Carburising at 930°C until a 2.6 mm thick carburised layer is formed
Cool to 820°C and hold for 1 hour + Oil quench
Sub Zero treatment at -80°C for 90 minute
Temper at 200°C for 2 hours + Air cool</td></tr>
<tr><td>HT 4</td><td>Part 4</td><td>Not applied</td></tr>
</table>

A Struers hardness tester was used to measure the hardness of each forged part. The indents were made from the carburised case (surface) to the core of each part using a Knoop indenter with a fixed load of 100gF. Each indent was 0.3 mm apart from each other and each scan contains 28 indents, which covers almost 8 mm distance from the surface to the core. Five such scans were conducted on each of the parts and then their average taken, standard deviation was also calculated. For the reader’s convenience the Knoop hardness values (HK) were converted to Vicker’s hardness (HV) and plotted accordingly.

The microstructural characterisation was carried out using optical and scanning electron microscopy. The samples were etched using Nital (solution of 2% HNO_3 into ethanol) to reveal the general microstructure and prior austenite grains. The etched samples were examined using optical microscopy followed by Electron Backscattered Diffraction (EBSD) to determine the average effective grain size of the high angle martensitic packets and blocks. ImageJ was used to calculate the prior austenite grain size from the optical micrographs according to ASTM standard E112. EBSD data was acquired using AZtecHKL software operating with an accelerating voltage and working distance of 20kV and 20mm, respectively. The corresponding data processing was then carried out using HKL Channel 5 post processing software. Orientation mapping was performed on a rectangular grid with a step size of 0.5 µm at x1000 magnification. Only high angle grain boundaries (HAGB) were detected to determine the effective grain (martensitic packet and block) size and were defined by θ>15º. Detected martensitic packets/blocks with an area <2.5 μm^2 were considered to be noise and not included in the average effective grain size calculation.

Results and Discussion

Tensile test

Figure 2 shows the stress-strain curves from the tensile tests of the forged parts. The deformation in all specimens is almost identical, until the transition from elastic to plastic deformation. The yield stress for the aforementioned tests was calculated using a strain offset of 0.2% and the ultimate tensile strength was determined as the maximum stress value reached. In order to obtain a good statistical representation of the properties the obtained yield stress and ultimate tensile strength of the two tests for each part were averaged. The summary of the tensile test results are presented in Table 3.

Figure 3 shows a comparison between the measured average yield stress and ultimate tensile strength of the heat treated parts. No significant difference in tensile properties can be observed amongst all four forged parts, though parts 1 – 3 possess a slightly higher tensile and yield stress compared to part 4. It is noteworthy here that part 4 did not receive any normalising heat treatment nor did it go through an extra annealing and hardening step after carburisation as given to parts 1 and 3. Further to this, only a minor improvement in tensile properties can be observed for the parts that were normalized before carburising compared to those that were not (for part 1 compared to part 2 and for part 3 compared to part 4).

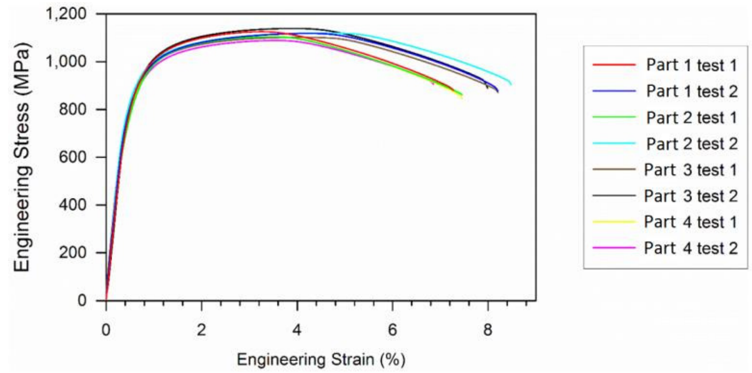

Figure 2: Stress-strain curve obtained from the tensile tests of the heat-treated 18CrNiMo7-6 forged parts

Table 3: Summary of the tensile test results

Test ID	0.2% YS (MPa)	UTS (MPa)	Elongation (%)
Part 1 test 1	927.2	1125.5	7.27
Part 1 test 2	915.2	1118.2	8.21
Part 2 test 1	913.4	1104.2	7.45
Part 2 test 2	915.8	1120.0	8.48
Part 3 test 1	909.1	1101.8	8.21
Part 3 test 2	932.7	1138.9	7.99
Part 4 test 1	910.0	1104.5	7.45
Part 4 test 2	903.5	1089.1	6.86

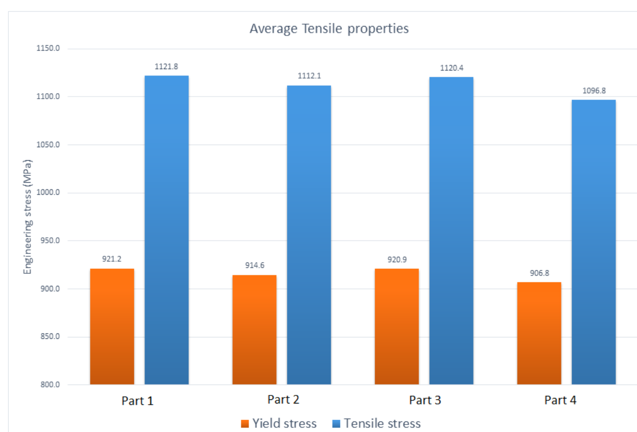

Figure 3: Comparison of the average yield stress and tensile stress of the heat-treated 18CrNiMo7-6 forged parts

Hardness

Figure 4 shows the change in hardness values for all four heat-treated forged parts from the carburised layer (surface) to the core. The hardness values are observed to be very high (750 – 800 HV) at the surface followed by a gradual decrease to circa

500HV in hardness with increasing depth (up to 2.6 mm). The core was found to be much softer with a hardness range 350 – 450 HV as compared to the surface (or case). These values are very similar to those reported in the literature, where the carburisation heat-treatment can result in a case hardness of 60 – 63 HRC, i.e. 740 – 810 HV with a core hardness of 300 – 380 HV (3). It should be noted that no significant difference is observed in terms of hardness for the four forged parts although they have experienced different heat-treatments.

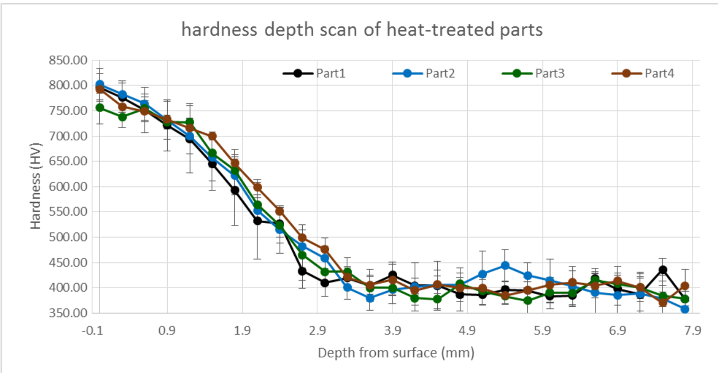

Figure 4: Hardness depth scans of heat-treated 18CrNiMo7-6 forged parts

Microstructural Analysis

Figure 5 shows the optical micrographs of the carburised layer (case) and the core of the four heat-treated forged parts. The austenite grains are transformed into martensite in the case and in the core upon quenching. However, the prior austenite grain boundaries can be seen, more prominently so in the core than in the case. In martensitic lath steels, such as the steel used in this study, there is a hierarchical substructure within the prior austenite grain boundaries. This substructure contains packets that consist of blocks that are made of individual sub-blocks containing laths (4).

The prior austenite grain size (PAGS) of the core material is measured using optical micrographs and ImageJ analysis software. During the quenching process, the austenite grains transform into high carbon martensite in the case and low carbon martensite in the core. However, the prior austenite grain size can still be obtained from the transformed microstructures. Coarser PAGS have been reported to result in lower yield strength, lower toughness, increased ductile-to-brittle transition temperature and higher residual stresses (1).

Figure 6 shows the average prior austenite grain size of the core material for all four forged parts as measured from the optical images. The average grain size of the forged parts undergoing two step quenching after carburisation (parts 1 and 2) is found in the range of 8 – 10 micron (G10 – G11 as per ASTM standard), whereas the parts directly quenched to room temperature after carburisation (parts 3 and 4) show average grain size of 18 – 20 micron (G8 – G8.5 according to ASTM standard). This indicates that a finer average grain size is obtained when carburisation is followed by the subsequent two step quenching, almost half the size of that obtained by direct quenching.

As reported elsewhere (5), the initial grain size in the sample affects both the case and the core of a case-hardened steel. A fine-grain microstructure i.e. G6 or finer (i.e.G7 - G9 or 15 - 45 micron) is desirable for achieving final properties. As observed in the current study, the annealing and hardening step after the carburisation (i.e. parts 1 and 2) results in a refined microstructure with a finer average prior austenite grain size (8 – 10 micron or G10 – G11 according to ASTM standard) as compared to other forged parts.

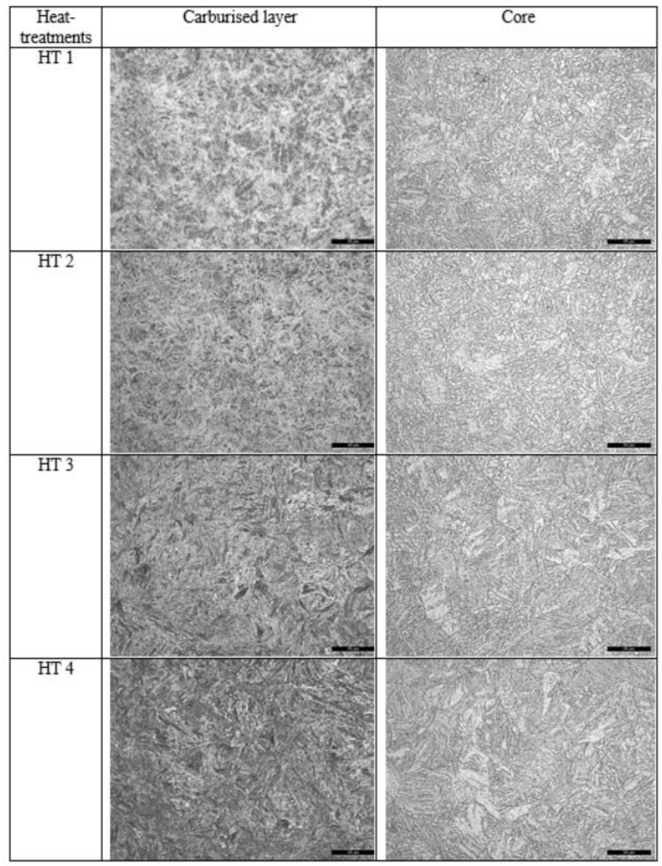

Figure 5: microstructure of heat-treated 18CrNiMo7-6 forged parts etched with Nital, showing core material and carburised layer (Marker on each micrograph is 20 microns)

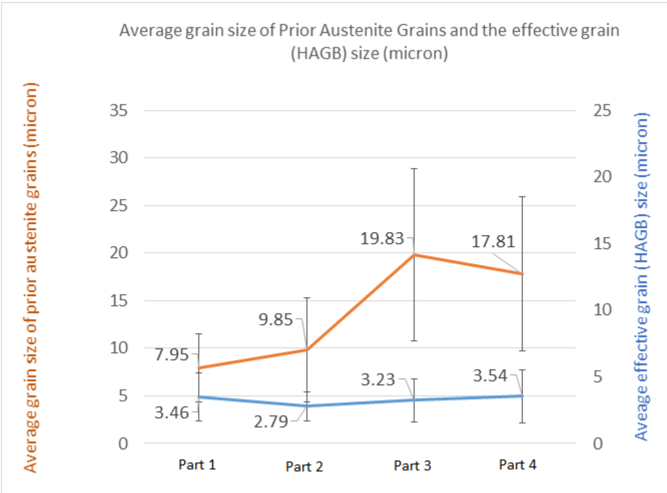

Figure 6: Average prior austenite grain size of the core material as measured from the optical micrographs as compared to the average effective grain size of the core material (high angle grain boundaries, HAGB, θ>15° of martensitic packets and blocks) measured by EBSD.

EBSD was utilised to determine the effective average grain size by measuring the high angle grain boundaries (HAGBs) of the martensitic packets and blocks within the prior austenite grain boundaries (PAGBs). Figure 6 shows how the effective average grain size changes as compared to the prior austenite grain size and Figure 7 shows the IPF colour maps in the Y/forging direction from the core of forged parts 1 to 4. As can be seen from Figure 6 and Figure 7, the part 2 has the smallest effective grain size, i.e. the part that has experienced no normalising heat treatment prior to carburisation. A Hall-Petch relationship between the effective grain size and the yield strength has been observed (6), but the same relationship was reported not to exist between the prior austenite grain size and the yield strength. However contrary to this a Hall-Petch relationship for both the effective grain size and prior austenite grain size with the yield strength has been observed elsewhere (7). In the same study it was also reported that only a 25% increase in the yield strength was achieved with a significant prior austenite grain refinement (from 166 μm to 6 μm) for 17CrNiMo6 steel. It was therefore concluded that grain refinement was not very effective in increasing the strength of martensitic lath steels (7). This can explain why the effective grain size has little effect on the reported yield strength and the UTS of the part 2, as compared to the other heat-treatments studied in the present work. Additionally, due to common $\{100\}_m$ cleavage planes in the parallel laths present in the blocks and in the packets within the martensitic lath substructure, the mechanism of transgranular fracture has been shown to be directly related to packet size and thus refinement of packet size can improve resistance to transgranular fracture (8). Therefore, the part 2 may have other microstructural advantages not explored in this paper. It has also been reported (9) that a Hall-Petch relationship exists between the yield strength and the prior austenite grain size, packet size and block size respectively and it was concluded that while the prior austenite grain size has a remarkable effect on the toughness and strength of the material, the block, comparable to the effective grain size in this case, is the smallest microstructure unit controlling strength and toughness. Moreover, EBSD investigation of lath martensite (10) has concluded that the block boundaries are the most effective sub-structure boundary in cleavage crack deviation due to the fact that all block boundaries were found to be of high angle, whereas only ~75% of the packet boundaries offered an effective barrier to crack propagation. In this study the effective grain size is measured in terms of HAGBs which provides crucial insight regarding effective barriers to the crack propagation.

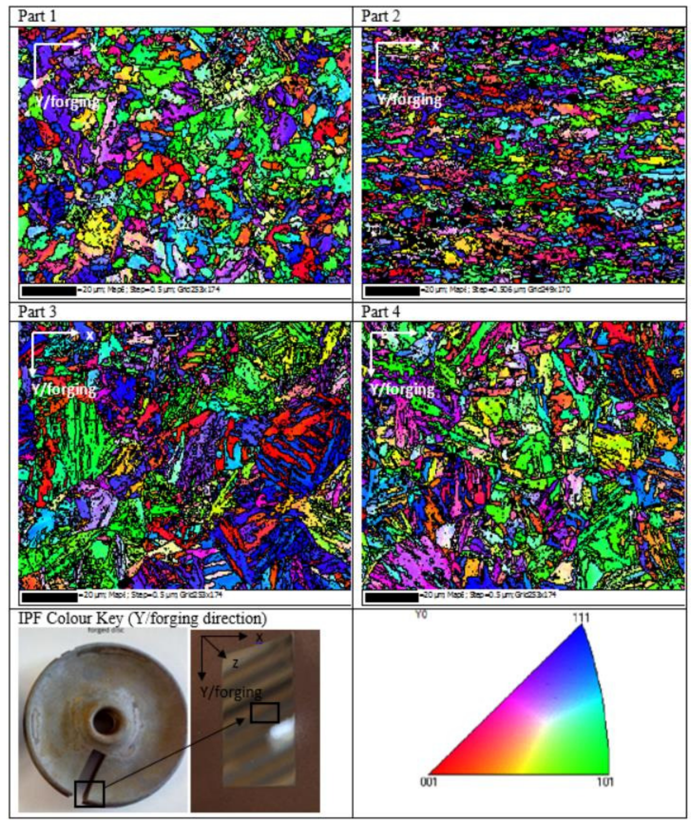

Figure 7: IPF colour maps in the Y/forging direction from the core of forged parts 1 and 4 as measured by EBSD.

Conclusions

1. The two-step quenching process (with an additional annealing step, followed by hardening and quenching) applied to part 1 and part 2 after the carburisation process was found to provide a more refined microstructure with a prior austenite grain size almost half the size of that achieved by direct quenching, in the case of part 3 and part 4, for the hot-forged case hardened 18CrNiMo7-6 steel.
2. From EBSD analysis of the effective grain size (the martensitic packets and the blocks) the part 2

exhibited the smallest average effective grain size. This can be attributed to the absence of a normalising treatment prior to carburisation. The normalising treatment results in slight grain growth as can be see for the part 1, which could have a negative effect on the fatigue properties.

3. The findings would suggest that the two-step quenching process (with an additional annealing step, followed by hardening and quenching) and no prior normalisation, as applied to the part 2, results in the most refined microstructure, with the smallest PAGS and effective grain size. However, this refinement in grain size appears to have no significant effect on the measured mechanical properties e.g. hardness, UTS or yield strength. Additionally, the refined microstructure may have a beneficial influence on the fracture toughness of the material, not investigated in this study.

Summary

Table 4: A comparison summary of the analysis conducted on the heat-treated 18CrNiMo7-6 forged parts

Heat-treatments	Part 1	Part 2	Part 3	Part 4
Avg. grain size of core in micron (from optical micrographs)	7.95 ± 3.60	9.85 ± 5.49	19.83 ± 9.05	17.81 ± 8.06
Avg. martensitic packet size of core in micron (from EBSD analysis)	3.46±1.81	2.79±1.09	3.23±1.60	3.54±2.0
Avg. UTS (Mpa)	1121.8	1112.1	1120.4	1096.8
Avg. Yield stress (MPa)	921.2	914.6	920.9	906.8
Average hardness of Case (HV)	670.89	680.82	676.82	693.93
Average hardness of Core (HV)	402.80	408.09	400.64	411.53

It is noteworthy that, the current work has provided a deep insight into the effect of tailored heat-treatment approaches on the final mechanical properties and microstructure development, as seen in the results summarized in Table 4. Whilst the two-step quenching process with no prior normalising heat-treatment provided slight refinement in the microstructure, the feasibility of this heat treatment must be assessed from the overall context of the total manufacturing route. It may be the case that the component with the least stages of heat-treatment, the part 4 in the current work, can meet the engineering requirements for a specific application. Hence, the current work has provided four different heat-treatment combinations that can be used to tailor the final properties of a given component to meet the specific end application requirements.

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