Effect of hot stamping and quenching & partitioning process on microstructure and mechanical properties of ultra-high strength steel

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Abstract
In order to achieve the purpose of automobile lightweight, hot stamping and quenching & partitioning (HS-Q&P) process has become a hot research focus. This process includes a hot stamping followed by a controlled quenching and partitioning process. In this paper, the effect of different austenitizing temperatures and time, as well as different holding pressure and partitioning temperatures and time on the microstructure evolution and mechanical properties of the tested steel was systematically studied. The resulting microstructure was characterized by scanning electron microscope (SEM) and electron backscatter diffraction (EBSD), and the volume fraction of retained austenite and carbon concentration in austenite were measured by x-ray diffraction (XRD). The results show that, the microstructure of HS-Q&P steel was finer and smoother than that of Q&P steel. During the pressure holding and partitioning process, the carbon atoms diffuse from martensite to austenite, thereby improving the stability of the retained austenite. Because both retained austenite and ferrite can improve the product of strength and elongation (PSE), the PSE of two phase zone austenitized HS-Q&P steel is better than that of completely austenitized HS-Q&P steel. When the austenitizing temperature was 780 °C and the pressure holding time was 90 s, the HS-Q&P steel reached the best mechanical properties. Its tensile strength was 1350 MPa, elongation was 14.24%, and PSE was 19.22 GPa%.

1. Introduction
In order to comply with environmental legislation, and achieve the goals of vehicle energy saving and emission reduction, reducing the weight of vehicle is the best way[1]. It was estimated that a 10% reduction of weight will result in a 5% decrease in fuel consumption [2]. In this case, the research on the new technology of manufacturing automobile parts by high strength steel is very important.

The traditional quenching & partitioning (Q&P) process consists of austenitizing the steel sheet, followed by quenching it to a temperature called the quenching temperature ($T_q$), which is between the temperature of the martensite start temperature ($M_s$) and the martensite finish temperature ($M_f$). Then, the steel is heated from $T_q$ to the partitioning temperature ($T_p$) and hold for a given time, or perform the partitioning at the same temperature as $T_q$. The former was called the two-steps Q&P process, and the latter was called the one-step Q&P process. In the Q&P process, retained austenite is very important because it can produce a transformation induced plasticity (TRIP) effect [3]. This effect occurs during plastic deformation. It can make the car parts absorb the collision impact energy, improve the strength of the car body, and reduce the hurt to the passengers [4].

Hot stamping technology first appeared in 1973. It can solve the defects such as cracking and wrinkling of ultra-high strength steel formed during room temperature, and solve some problem such as large spring-back of formed parts. Since its emergence, it has been favored by global automobile manufacturers [5]. By 2007, the
global hot-stamped parts had exceeded 100 million. It is predicted that by 2020, the parts demand will exceed 1 billion [6].

Hot stamping can improve strength and formability of the auto parts [7], thereby reducing the weight of the car body. However, it will reduce the plasticity and toughness of the hot stamped steel. For the auto parts [8], it needs to have a certain degree of plasticity and toughness to absorb impact energy, thereby improving the safety of the automobile. Therefore, a new process that combines the Q&P process with HS process, that is, the hot stamping and quenching & partitioning (HS-Q&P) process, has attracted more and more attention from researchers. Liu [9] proved that there are fine-grain retained austenite and lath martensite in the HS-Q&P steel. Liu [10] proved that the product of strength and elongation (PSE) of the HS-Q&P steel have been greatly improves compared with the direct hot stamping process. Zhu [11] had proved that there is more retained austenite in the microstructure through HS-Q&P process compared with the direct hot stamping process. Combined with the previous research, this paper studies the mechanism of HS-Q&P process to improve the plasticity of materials. The HS-Q&P process can obtain a refined multiphase structure by deformation, phase transformation and carbon partitioning, this process can significantly improve plasticity of the material while ensuring strength.

2. Experimental

The tested steel was low-carbon silicon-manganese steel. Its delivery status is 1.2 mm thick annealed cold-rolled sheet, and its initial microstructures are ferrite and pearlite. The composition is shown in table 1.

The DIL 805 A thermal dilatometer was used to measure the phase transformation points of the test steel. The thermal expansion specimen was heated to 900 °C at 10 °C/s, then held for 5 min, and finally quenched to room temperature at a cooling rate of 50 °C/s. The measured austenite formation finish temperature (AČ3), austenite formation start temperature (AČ1), and martensite start temperature (MS3) is 840 °C, 710 °C and 330 °C, respectively. The hot stamping mold used in this experiment was a self-designed mold, which could achieve precise control of mold temperature within the range of 20 °C–500 °C.

The rectangular heat-treated specimens of 100 × 100 mm were cut from the cold-rolled sheet along the rolling direction. The specimens were heated to 780 °C and 930 °C, respectively, in an atmosphere furnace with a holding time of 5 min. After that, they were taken out of the furnace and placed on the stamping mold for hot stamping. Then they were quenched to a mold temperature of 300 °C with a holding pressure time of 5 min. Finally, they were water quenched to the room temperature. The processes were shown in figures 1(a) and (b), respectively. During the pressure holding process, the Q&P process was performed, the carbon partitioning time.

According to the ASTM E8 standard, the heat-treated specimens were wire-cut into standard tensile specimens with gauge length of 25 mm, width of 6 mm, and thickness of 1.2 mm. The standard tensile specimens
were tested at an extension rate of 1 mm min$^{-1}$ at room temperature by WDW-100E universal testing machine. The scanning electron microscopy (SEM) specimens were etched with 4% nital solution. The SEM observation was carried out using a field emission scanning electron microscope (FESEM) Hitachi SU3500. Electron backscatter diffraction (EBSD) technique was employed to characterize different microstructure such as ferrite, martensite and retained austenite. The specimens for EBSD were mechanically ground and then electro-polished using a 700 ml CH$_3$COOH + 200 ml HClO$_4$ solution at 16$^\circ$C and 31 V. All EBSD maps were obtained on a Zeiss SUPRA 55 equipped with a conventional EBSD system by a spatial step size of 0.02$\mu$m [12]. The EBSD was operated at 20 kV with specimen tilted by 70$^\circ$. The x-ray diffraction (XRD) specimens were investigated using an Ultima IV diffractometer with Cu K$_\alpha$ radiation at 50 kV and 300 mA, the scanning range are from 40$^\circ$–100$^\circ$, and the scanning step is 0.05$^\circ$ [13]. The volume fraction of retained austenite was determined by the Rietveld full pattern fitting method [14]. Combined with equation (1), the volume fraction of retained austenite was calculated [15].

$$f = \frac{1.4I_\gamma}{1.4I_\gamma + I_\alpha}$$

Where $f$ is the volume fraction of retained austenite, $I_\gamma$ is the integrated intensity of the austenite peaks, and $I_\alpha$ is the integrated intensity of the ferrite peaks. In addition, the carbon concentration in austenite was determined by equations (2) and (3) according to XRD analysis [16]:

$$a = \frac{\lambda}{2 \sin \theta} \sqrt{h^2 + k^2 + l^2}$$

$$a = 0.3556 + 0.00453X_C + 0.000095X_{Mn} + 0.00056X_{Al} + 0.0006X_{Cr} - 0.0002X_{Ni}$$

where $a$ is the lattice constant of the austenite (nm), $\lambda$ is the incident wavelength (nm), $hkl$ is the crystal plane indices, and $2\theta$ is the diffraction angle. $X_C$, $X_{Mn}$, $X_{Al}$, $X_{Cr}$ and $X_{Ni}$ present the weight percent of carbon, manganese, aluminum, chromium and nickel in the austenite, respectively (%).

3. Results and discussion

3.1. Effect of austenitizing temperature on the microstructure of test steel

Figure 2 shows the SEM images of the tested steel at different austenitizing temperatures. At 780$^\circ$C, the microstructure was comprised of ferrite, martensite, and retained austenite. It can be seen from figure 2(c) that the martensite boundary was blurred, which was caused by interface migration. The red frame in figure 2(c) indicated a narrow pit with a length of 0.5$\mu$m at the grain boundary. The low austenitizing temperature led to incomplete dissolution of carbides, resulting in undissolved carbides falling off during the stamping process, whereby the pits were formed.

When the austenitizing temperature was higher, the carbon atoms had a higher driving force for a longer distance in the austenite. During the pressure holding process, the carbon partitioning would cause the austenite interface to move to the martensite side. As the pressure holding time increased, the austenite would be homogenized. At the same time, carbides originally soluble in pearlite would also dissolve, resulted in an increase in carbon concentration in austenite. Moreover, at 930$^\circ$C, the austenite obtained sufficient undercooling and transformed into martensite during the stamping process.

Compared with figures 2(c) and (d), at 930$^\circ$C, the martensite was mainly lath-like, and its boundary was sharper and clearer, but some were block structure with a size of 2–3.5$\mu$m shown in figure 2(e). It can be seen that there were two corrosion reactions. According to the computer simulation results, when the tested steel was heated to 900$^\circ$C, its structure was totally austenite, so the block structure was not pro-eutectoid ferrite [17], but the different types of martensite [18]. The different carbon concentration caused this differentiated corrosion reaction in the SEM images. The stamped martensite $M_1$ was formed during the carbon partitioning process [19]. The carbon concentration in martensite $M_1$ is relatively low, so $M_1$ was corroded deeper. While the quenched martensite $M_2$ was formed in the water quenching. During the partitioning process, carbon atoms were diffused from the stamped martensite $M_1$ to the retained austenite. The carbon-rich austenite was transformed into quenched martensite $M_2$ during water quenching. Compared to $M_1$, the quenched martensite $M_2$ had a higher carbon concentration, so $M_2$ was more resistant to corrosion. Therefore, in the SEM images, martensite presented two different morphologies.

Figures 2(e) and (f) show the SEM images of the tested steel without hot stamping (HS) process. It can be seen from figures 2(a) and (e) that when the austenitizing temperature was 780$^\circ$C, the surface of martensite had more obvious wrinkles in the steel without HS process. The boundaries between martensite and ferrite were clearer and sharper, and some boundaries were relatively straight. In the steel with HS process, the grain surface was relatively smooth. It can be seen from figures 2(b) and (f) that when the austenitizing temperature was 930$^\circ$C,
there were local coarse martensite in the steel without HS process. While in the steel with HS process, the martensite surface was relatively smooth, and the martensite morphology was lath. This is because in the HS process, the tissue was subject to constant external pressure, the TRIP effect occurred, and the grain surface was relatively smooth, the wrinkles in the images were almost invisible. In addition, after HS process, the martensite and austenite were re
refined, and more fragmented martensite appeared. Hence, according to Hall-Petch relationship [20] (4):

\[ \sigma = \sigma^0 + Kd^{-1/2} \]  

where \( \sigma \) is the yield strength, \( \sigma^0 \) is the resistance due to lattice friction, \( d \) is the mean grain size, and \( K \) is a constant. Therefore, the refinement of the martensite and austenite grains size increases its yield strength.

Besides, compared by the direct quenching process, the width of the martensite lath in figure 2 is more extensive by HS-Q&P process, and the martensite boundary is more curved, which is also caused by the interface migration during the carbon partitioning process [21].

Figure 2. SEM images of tested steel at different austenitizing temperatures. (a) 780 °C, 3000 \times, (b) 930 °C, 3000 \times, (c) 780 °C, 8000 \times, (d) 930 °C, 8000 \times, (e) 780 °C, 3000 \times, (f) 930 °C, 3000 \times. In the figures, (a)–(d) show the tested steel with hot stamping process, (e) and (f) show the tested steel without hot stamping process.
Figure 3 shows the processed SEM images of figures 2(a) and (b) processed by the image processing software. In figure 3(a), the martensite and a small amount of austenite were showed in red and the ferrite was showed in yellow. By calculation, at 780 °C, the ratio of the red areas to the yellow areas was 52.4:47.6. Through computer simulation, the ratio of austenite to ferrite was 48.5:51.5 at 780 °C. The ferrite in the tested steel was reduced by 3.9% compared with the simulation results, indicated that some ferrite may be transformed into austenite during the holding process in the two phase zone. The specific mechanism of this transformation needed further experimental verification. In figure 3(b), the red areas were quenched martensite M2, the yellow areas were stamped martensite M1. Calculated by software, at 930 °C, the ratio of the red areas to the yellow areas was 73.9:26.1.

3.2. Morphology and content of retained austenite

Figure 4 shows the EBSD maps of different pressure holding time when the austenitizing temperature was 780 °C. Figures 4(a) and (c) were the phase color maps of 60 s and 120 s. In the figure, the grains difference on both sides of the thick black line is 10°–20°, that of the thin black line is 3°–10°, and that of the thin gray line is less than 3°[22].

It can be found that most lines are thick black lines, indicated that the grains difference was enormous. Nevertheless, there were still a few thin black lines. These areas were considered as substructures. The black block areas in the figure were the unrecognized area. These areas were caused by the volume change during the transformation of austenite to martensite, which resulted in slight unevenness of the phase interface. The green areas in phase color maps were body-centered cubic lattices (martensite and ferrite), and the red areas were face-centered cubic lattices (austenite).

Compared figure 4(b) with figure 2(c), the grains of the same color had the same orientation. The single blue color block was ferrite, and the colorful areas were martensite island. Most of the austenite was distributed inside the martensite island, and the rest distributed at the boundaries between martensite and ferrite. And these austenites appeared as discontinuous networks and their thickness was relatively thin. Therefore, it could be considered that the morphology of austenite was film-like. Compared figures 4(b) and (d), as the pressure holding time increased, the grains showed a clear tendency to break. The primary grains would break into many small grains with different phases. In figure 4(d), some adjacent grains were similar in color, indicated that their orientation difference was small, because they came from the same original austenite grain. Some fine grains had different orientations, because they were the stamped martensite M1 and the quenched martensite M2, respectively. The difference in carbon concentration and formation process caused a large phase orientation difference.

Figure 5 shows the XRD patterns at different austenitizing temperatures. After calculation, the proportion of retained austenite at 780 °C was 3.98%, and at 930 °C, that was 6.05%. At 930 °C, the microstructure had been completely austenitized, and the initial amount of austenite was relatively high, so more austenites were more likely to be retained at room temperature. At 780 °C, the initial amount of austenit was relatively small, and the retained austenite was less at room temperature. But, when 780 °C, the degree of carbon homogenization in the austenites was lower, resulted in some austenite being rich in carbon, and the rest poor in carbon, and this high-carbon retained austenites could obtain better room temperature stability. At 930 °C, although the degree of carbon homogenization was high, the carbon concentration in the austenite was not high, such austenite have
lower stability at room temperature. Therefore, the retained austenite was less treated at 780 °C than that at 930 °C, but did not decrease proportionally. At 930 °C, the average carbon concentration in retained austenite was 1.04–1.17%, and at 780 °C, the average carbon concentration in retained austenite increased significantly, ranged from 1.13–1.23%.

3.3. Effect of austenitizing temperature on mechanical properties

Figure 6 shows the mechanical properties of the tested steel at different austenitizing temperatures. At 780 °C, with the pressure holding time increased, the tensile strength of tested steel decreased from 1430 MPa at 30 s to 1280 MPa at 120 s. The reason for the decrease was that as the holding time increased, the carbon partitioning effect increased, the carbon concentration in martensite decreased, resulted in the strength of martensite decreased. Through the measurement, the elongation was 12.60%, 12.12%, 14.24% and 14.40% under pressure holding time of 30 s, 60 s, 90 s and 120 s, respectively. The elongation of 90 s was increased by 17.49% compared with that of 30 s. The elongation increase was caused by two reasons: (i) Carbon partitioning improved the
stability of retained austenite \([23, 24]\), which is a plasticity contribution phase \([25]\); (ii) As the holding time increased, the degree of grains fracture increased, resulted in fine-grain strengthening \([26]\). When the pressure holding time was 90 s, the PSE \([19]\) reached the maximum value of 19.22%. While at 120 s, although the elongation reached its highest, the PSE showed a small decline due to the sharp drop in tensile strength.

When the temperature was 930 °C, the change of the tensile strength was same as that at 780 °C. With the holding time increased, the tensile strength decreased. Through the measurement, the elongation was 10.04%, 9.72%, 11.04% and 11.16% under pressure holding time of 30 s, 60 s, 90 s, and 120 s, respectively. The plasticity did not change obviously, but showed an increasing trend. The reason is that the carbon concentration in austenite was not high. Even if the austenite was homogenized under 930 °C, the stability of the retained austenite would not change significantly. The mechanical properties have a very close relationship with the element partitioning, especially in the carbon partitioning process, and these reasons caused the PSE first decreased and then increased and finally decreased again of 30–120 s.

When the austenitizing temperature was 780 °C, there was more ferrite in the room temperature, so the tested steel had a higher elongation. At this temperature, the total amount of austenite was small, and the total amount of martensite formed by austenite was also small, resulted in a decrease in the tensile strength of the tested steel. However, as mentioned earlier, during the stamping process, the microstructure was refined, according to the Hall-Petch relationship, the strength of tested steel increased with the refinement of the grains. And partial austenite was transformed into martensite through the TRIP effect in this process. During the Q&P process, partial austenite was more stable due to the carbon partitioning. These reasons improved HS-Q&P steel’s strength \([27]\).

In terms of martensite, at 930 °C, the temperature was already in the completely austenite zone. When the tested steel was water quenched to room temperature, there was more martensite in the structure, which is the main contributor to the strength. In terms of austenite, when it was subjected to external force during hot stamping, the TRIP effect would be produced, which could increased the plasticity of the steel, while the stamped martensite \(M_1\) also appeared. In terms of ferrite, it had been demonstrated that no ferrite was produced at 930 °C.
in section 3.1, and no ferrite structure was found in the SEM images. At 780 °C, after hot stamping process, there was still a large amount of ferrite in the structure. As the softer phase, the ferrite phase has lower strength and better plasticity. At both temperatures, a trace amount of bainite was found in the microstructure, but the content was less, which had little effect on the mechanical properties.

Compared with the 930 °C steel, the 780 °C steel had an average tensile strength reduction of 15.15%, an average elongation increase of 27.17%, and an average PSE increase of 7.9%.

4. Conclusion

(1) The initial microstructures of the tested steel are ferrite and pearlite. After HS-Q&P process, the pearlite disappeared, and martensite and austenite appeared. At completely austenitizing temperature, martensite had two structures: lath-shaped quenched martensite M2 and stamped martensite M1. As the austenitizing temperature increased, more retained austenite remained.

(2) As the austenitizing temperature increased, the tensile strength of the tested steel increased, but the elongation decreased, resulting in a decrease in PSE. Therefore, the mechanical properties of the two phase zone austenitized HS-Q&P steel was better than that of completely austenitized HS-Q&P steel.

(3) As the pressure holding and partitioning time increased, the tensile strength of the tested steel decreased, and the elongation first decreased and then increased. The results showed that Q&P process could indeed improve the plasticity of hot stamping steel. When the austenitizing temperature was 780 °C and the pressure holding time was 90 s, the HS-Q&P steel reached the best mechanical properties, with a tensile strength of 1350 MPa, an elongation of 14.24%, and a PSE of 19.22 GPa.

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Conflicts of interest

The authors declare no conflict of interest.

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