Microstructural Evolution of Carbon Steels in Hot Stamping Processes

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Hot stamping is a very promising method for producing ultrahigh-strength automotive components. The heating and cooling conditions in the conventional hot stamping process are quite limited. To improve the productivity of the process and/or the mechanical properties of the product, optimizing the heating and cooling conditions is an important and urgent research subject.

In this study, the influence of the heating rate, heating temperature, and cooling conditions on the microstructure and mechanical properties of 0.22%C–2.5%Mn steel was investigated. Samples were heated at two different heating rates (10°C/s and 200°C/s) to various temperatures and then either cooled in air or quenched using water sprays. The sample heated at 200°C/s to 800°C and then immediately quenched in water exhibited a very fine martensite microstructure having a grain size with an average diameter of 4 μm and a hardness value around 550 Hv. The sample heated above Ac3 and subsequently cooled in air exhibited a full martensite microstructure and a hardness of 450 Hv. A model that predicted the precipitation of cementite during cooling was developed to clarify the cause of the difference in hardness of the martensite in the samples that were quenched in water and cooled in air. The calculations showed that the hardness of samples cooled in air was lower mainly because of the decrease in the solid solution hardening of C owing to the precipitation of cementite below the Ms temperature. Drawing on the results obtained, hot stamping technology is discussed and favorable operational conditions proposed.

KEY WORDS: hot stamping; model; rapid heating; cementite precipitation; solid solution hardening; ultrahigh-strength steel.

1. Introduction

Ultrahigh-strength steel components have been produced and incorporated into cars to reduce the weight of automotive bodies. Hot stamping technology is an attractive method for producing ultrahigh-strength automotive components with high gauge accuracy.1–15) Figure 1 shows a schematic diagram of a conventional hot stamping process, in which a sample is heated to Ac3 in a furnace and delivered to a press. After stamping, the stamped component is held in the dies until the martensite transformation completes. This die quenching simultaneously delivers ultrahigh strength and good shape fixability. However, a marked disadvantage of the conventional hot stamping process is its low productivity, mainly because of the time-consuming die quenching and laser cutting that needs to be performed after stamping.
Recently, the authors proposed a new, highly productive hot stamping technology. Figure 2 shows a schematic diagram of this new process, in which rapid heating is recommended instead of furnace heating. The samples are quenched in a shape-fixing device after stamping so that the time-consuming die quenching stage is avoided. Multiple stamping enables trimming and piercing in the stamping process to be carried out and reduces or omits the need for any laser cutting. Since multiple stamping and the transportation to a quenching facility cause a higher drop in temperature of the sample compared with the conventional process, this new process is recommended for more hardenable steels with a higher Mn content.

In this study, the influence of the thermal history, namely the heating rate, heating temperature, and cooling conditions, on the microstructure and mechanical properties of 0.22%C–2.5%Mn steels, a promising material for this highly productive hot stamping technology, was investigated. The aim of this study was to understand the metallurgy of the hot stamping processes and to determine the proper heating and cooling conditions for producing high-quality products with high productivity.

2. Experimental Procedure

2.1. Material

The main chemical composition of the steel used for hot stamping was 0.22%C–1.2%Mn–0.002%B. This steel exhibits a tensile strength of 1 500 MPa if the hot stamped component is cooled at a rate faster than 30°C/s.

Table 1 shows the chemical composition of the steel used in the experiment. The Mn content was increased to 2.5% to improve the hardenability so that no transformation apart from the martensite transformation would occur during the hot stamping process. The steel was melted in a vacuum furnace and cast into a 50 kg ingot. The ingot was reheated at 1 150°C for 60 minutes and then hot rolled into a 3 mm thick hot band at a finishing temperature of 930°C. The hot band was placed directly into an electrical furnace at 650°C, left for 60 minutes at that temperature, and then allowed to cool to room temperature at the natural rate of the furnace. The microstructure of the hot band consisted of ferrite and pearlite. After pickling, the hot band was cold rolled to form a 1.4 mm thick sheet. Figure 3 shows the microstructure of the steel before the heat treatment.

2.2. Heat Treatment

Figure 4 shows the heat treatment facility, which consists of a direct current heating device and water sprays. Figure 5 shows the thermal history of the sample heat treatment. Two heating rates were employed. The heating rate of 10°C/s is similar to the conventional heating rate in an electrical furnace. The heating rate of 200°C/s was envisaged as suitable for the highly productive hot stamping process shown in Fig. 2. The heating temperature varied from 650 to 900°C in 25°C increments. Some overheating could not be avoided when using the heating rate of 200°C/s. Immediately after reach-
ing the desired heating temperature, the samples were cooled either using water sprays or in air.

2.3. Microstructural Observations and Microhardness Measurements

The microstructural observations were carried out using optical microscopy (OM), secondary electron microscopy (SEM), and transmission electron microscopy (TEM). Some orientation imaging microscopy (OIM) functions of the electron backscatter diffraction patterns (EBSP), such as the image quality (IQ) and kernel average misorientation (KAM), were used to evaluate the ratio of martensite to the other microconstituents, such as ferrite and bainite. The hardness was measured on the micro-Vickers scale using a 500 g weight.

3. Experimental Results and Discussion

3.1. Evolution of the Microstructure and Hardness of Samples Heated to Various Temperatures at 10°C/s and 200°C/s and Subsequently Quenched Using a Water Spray

Figure 6 shows the hardness of samples heated to various temperatures using heating rates of 10°C/s and 200°C/s and subsequently quenched using water sprays. The hardness curve shifted to higher temperatures by around 8°C on increasing the heating rate from 10°C/s to 200°C/s. Figure 7 shows the microstructures of samples heated to 717°C at 10°C/s and to 727°C at 200°C/s. The formation of austenite had just begun in these samples. The sample heated at 10°C/s showed a partially recrystallized matrix, whereas the number of recrystallized grains was very low in the sample heated at 200°C/s. A previous study showed that the hardness curve shifted markedly to higher temperatures if the sample is fully recrystallized using a preheat treatment before any continuous heating occurs. However, the same study also showed that the hardness curve of a recrystallized sample shifts toward higher temperatures with increasing heating rate, because an increasing heating rate shortens the time for any formation of austenite. In the case of recrystallized samples, the shift in the hardness curve toward higher temperatures was around 25°C on increasing the heating rate from 10°C/s to 200°C/s. The observed slight shift in the hardness curve from the different heating rates is assumed to be attributable to the effect of the heating rate and the microstructure before austenite formation could occur, which would cancel each other out.

Figures 8 and 9 show the fraction of martensite in sam-
ples heated at 10°C/s and 200°C/s and the relationship between the hardness and the fraction of martensite. It was confirmed that the shift in hardness corresponded to the fraction of martensite and that the hardness value was determined by the fraction of martensite present in the sample.

Figure 10 shows the evolution of austenite grain size in the water-quenched samples with a microstructure containing almost 100% martensite heated at 10°C/s and 200°C/s. The grain size of the martensite in the samples heated just above Ac3 was around 4 μm, and increased with increasing heating temperature. The evolution of the hardness from the temperature rise in Fig. 6 corresponded well with the grain growth behavior. This result suggests that we should avoid setting unnecessarily high heating temperatures in order to maintain a fine microstructure and high sample strength.

It was also recognized that the samples heated at 200°C/s showed a smaller grain size than the samples heated at 10°C/s to the same heating temperature. The main reason for this is inferred to be that less time was available for grain growth in the samples heated at 200°C/s than in those heated at 10°C/s.

Figure 6 also shows that the maximum hardness of the samples heated at 200°C/s was slightly higher than that of the samples heated at 10°C/s. Since the grain size of the martensite was nearly the same in both samples exhibiting the maximum hardness value, as shown in Fig. 10, the slightly lower maximum hardness of the sample heated at 10°C/s occurs because of the very small amount of remnant ferrite, as shown in Fig. 8. The possible reason why the specimen heated at 10°C/s to 800°C showed the maximum hardness even though a small amount of ferrite remained may be explained by the consideration the grain size. The grain size of the specimen heated to 800°C was a half of that of the specimen heated to 850°C which revealed a 100% martensite microstructure. It is inferred that the maximum hardness of the specimen heated to 800°C was reflected by a strong effect of the grain refinement.

The investigation into the influence of the heating rate on the microstructure and the observed hardness showed that rapid heating had no detrimental effect on the microstructure and the mechanical properties, and also that it contributed to a marked improvement in productivity in the heating process.

### 3.2. Influence of Cooling Conditions on the Evolution of the Microstructure and the Hardness

Figures 11 and 12 show the influence of the cooling conditions on the hardness of specimens heated to various temperatures at heating rates of 10°C/s and 200°C/s, respectively. In the diagrams, the calculated values were also given. The calculation method is mentioned later. A markedly large difference in the hardness curves is observed between two cooling conditions, namely water quenching and air cooling. Especially a large difference is observed in the intercritical temperature range. To clarify the cause of this difference, the evolution of the microstructure during the heating is studied.

Figures 13 and 14 show the fraction of martensite of the specimens heated to various temperatures at 10°C/s and 200°C/s and quenched by water and cooled in air, respectively. These figures clearly show that γ→α transformation markedly proceeded during the air cooling due to the migration of ferrite remaining at intercritical temperatures into austenite.

To understand the evolution of the hardness given in Figs. 11 and 12, the relationship between the evolution of microstructure and hardness is discussed. We assumed that the hardness of a specimen can be calculated by Eq. (1).

\[
H_v = x \times H_{v_{max}} + x_{\alpha} \times H_{v_{\alpha}} + (1 - x - x_{\alpha}) \times H_{v_{min}} \quad \text{(1)}
\]

Here, \(x\) is the fraction of martensite, \(x_{\alpha}\) is the fraction of
transformed ferrite, $H_{\text{v,max}}$ is the maximum hardness of the specimens with full martensite under each combination of the heating and cooling conditions, $H_{\text{v,\alpha}}$ is the hardness of ferrite transformed during air cooling and is 200 Hv determined experimentally. $H_{\text{v,min}}$ is the lowest hardness of the specimens shown in the figures under each combination of the heating and cooling conditions. The specimens with the lowest hardness are free of martensite.

Figure 15 shows the image quality diagrams (a, c) and orientation maps (b, d) of the inverse pole figures showing the crystal orientations parallel to ND of the specimens heated at 200°C/s to 761°C (a, b) and 811°C (c, d) and cooled in air. The dark areas are martensite. The microstructure of the specimen heated to 811°C mostly consists of martensite and transformed ferrite while that of the specimen heated to 761°C consists of martensite, transformed ferrite and recovered ferrite (the fraction of the various phases in these pictures does not coincide with the fraction given in Figs. 13 and 14 because the microstructure is heterogeneous and the selected area of the pictures does not represent the average microstructure). The transformed ferrite is observed mainly in the area next to martensite. In the recovered ferrite, recrystallized grains were hardly recognized. The recrystallization was inferred to be inhibited by the formation of austenite which occurred at a relatively low temperature because of a high amount of Mn added to the steels. The significant retardation of recrystallization during an intercritical annealing has often been reported. This result justifies that Eq. (1), does not contain the term with the fraction of recrystallized ferrite. The calculation results reveal a good agreement with the experimental ones.

In a previous study using 3% Mn steel, there was almost no ferrite transformation during air cooling if the heating temperature exceeded the Ac3 temperature, but in the present case using 2.5% Mn steel, some austenite transformed into ferrite, even when the heating temperature exceeded Ac3. This was a consequence of the decreasing hardenability with decreasing Mn content. The sample heated to 930°C and cooled in air was fully transformed into martensite. The inferred reason is that the hardenability of the sample increased because of the grain growth of austen-
In a higher heating temperature range, both the water-quenched and the air-cooled samples exhibited 100% martensite microstructures, but had a marked difference in hardness. To investigate the cause of this difference in hardness, detailed observations of the microstructure were carried out using SEM.

**Figure 16** shows the microstructures of samples heated to 930°C at 200°C/s and subsequently water quenched or air cooled. In the sample cooled in air, many fine cementite particles were precipitated, but such precipitates were not observed in the sample quenched using water sprays. This fact strongly indicates that the tempering of martensite plays a significant role during the slow cooling in air. An additional experiment was carried out to examine the relationship between the decrease in hardness and the precipitation of cementite.

**Figure 17** shows a schematic diagram of the experimental conditions used. The samples were heated to 900°C at 200°C/s and cooled in air until a preset temperature from 500 to 200°C at increments of 50°C was reached, and then quenched in water. **Figure 18** shows the observed temperature curve of the sample cooled in air. The cooling rate decreased markedly from 370°C to 340°C. This marked decrease in the cooling rate is caused by the development of latent heat from the martensite transformation. This result, and those from simultaneous dilatation measurements, confirmed that no transformation other than the martensite transformation occurred.

**Figure 19** shows the relationship between the hardness of the samples and the onset temperature for water quenching. As can be seen in Fig. 19, the hardness of the samples quenched from a temperature above the Ms temperature exhibited a high constant value. A marked decrease in hardness was observed if the onset temperature before quenching was below the Ms temperature.

**Figure 20** shows the microstructure of samples that were quenched in water from 500°C and 250°C. It can be seen that the sample quenched in water from a temperature below the Ms temperature exhibited finely dispersed cementite, whereas virtually no cementite was observed in samples quenched from a temperature above the Ms temperature. This observation indicates that cementite only precipitates in the martensite matrix.

### 3.3. Model for Predicting Cementite Precipitation during Cooling below the Ms Temperature

A model for predicting the precipitation of cementite during cooling was developed to study the behavior of the hardness drop during air cooling. **Figure 21** shows a flow chart of the computer program for the model. The chemical composition of the steel and the cooling curve were input data for the model. The Ms temperature used in the calculations...
was calculated using the empirical formula: 
\[ \text{Ms} \, (^\circ \text{C}) = 539 - 423\% \text{C} - 30.4\% \text{Mn} \].

The time step used in the calculations was set to 0.1 s. The calculation of cementite precipitated with each time increment was carried out if the temperature \( T(t) \) was between the Ms temperature and 200 \(^\circ\text{C}\). The fraction of martensite was determined according to Eq. (2), as proposed by Koistinen.\(^{20}\)

\[ f = 1 - \exp\left(-0.01\, [\text{Ms} - T(t)]\right) \]  \quad (2)

Based on our experimental results, the precipitation of cementite was assumed to occur only in the martensite. The initial solute carbon content in martensite was assumed to be a value added to the steel. The precipitation of cementite was assumed to occur based on a nucleation and growth mechanism. The nucleation rate was formulated using Eq. (3), according to classical nucleation theory.\(^{21}\)

\[ I_{\text{com}} = N_0 \frac{KT}{h} \exp(-\frac{Q^c}{RT}) \exp(-\frac{\Delta G_{\text{a} \rightarrow \text{f}}}{kT}) \]  \quad (3)

Here, \( N_0 = (9 \times 10^9) \) is a constant used as a fitting parameter. The terms \( R, k, \) and \( h \) are the universal gas constant, the Boltzmann coefficient, and Planck’s constant, respectively. The term \( Q^c = (8.39 \times 10^4 \text{ J/mol})^{22}\) is the activation energy of the diffusion of carbon in ferrite, and \( \Delta G_{\text{a} \rightarrow \text{f}} \) is the driving force for the nucleation of cementite, and is given by Eq. (4).

\[ \Delta G_{\text{a} \rightarrow \text{f}} = \frac{16\pi}{3} \frac{\sigma_{\text{com}}^3}{(\Delta G_{\text{a} \rightarrow \text{f}} / V_{\text{Fe,C}})^2} \]  \quad (4)

Here, \( \sigma_{\text{com}} = (0.7 \text{ J/m}^2) \) is the interfacial energy of cementite and \( V_{\text{Fe,C}} \) is the mole volume of cementite. The term \( \Delta G_{\text{a} \rightarrow \text{f}} \) is the difference in Gibbs free energy between ferrite and cementite, and is given by Eq. (5).

\[ \Delta G_{\text{a} \rightarrow \text{f}} = -\frac{1}{4} RT \ln\left(\frac{C}{C_{\alpha}}\right) \]  \quad (5)

The term \( C \) is the amount of solute carbon in the martensite and \( C_{\alpha} \) is the equilibrium carbon concentration in ferrite at the interface between ferrite and cementite calculated under para-equilibrium conditions using the Thermo-calc software.

The growth of cementite was assumed to be controlled by the diffusion of carbon. The flux of carbon into cementite from ferrite was calculated by solving a diffusion equation with the equilibrium carbon content in ferrite at the interface between ferrite and cementite as a boundary condition. The growth rate was then described by Eq. (6).\(^{23}\)

\[ \frac{dr}{dt} = \frac{D_C^f (C - C_{\alpha})^2}{(C_{\text{com}} - C_{\alpha})(C_{\text{com}} - C_{\alpha})} \]  \quad (6)

Here, \( D_C^f \) is the diffusion coefficient of carbon in ferrite\(^{24}\) and \( C_{\text{com}} \) is the carbon concentration in cementite.

The cementite was assumed to be disc-shaped, with a diameter of \( d = 2r \) and a thickness of 0.2 \( d \).\(^{25}\) The precipitation of cementite is calculated for each time step as shown in Fig. 22. The number and size of the cementite nuclei were calculated in the martensite that was newly transformed in each time step \( \Delta t \). In the following time step, these nuclei grow, new nuclei nucleate, and the martensite transformation proceeds simultaneously. This calculation process proceeded for each time step until the onset time for quenching. The output data of the calculations were the number, size, and fraction of cementite and the amount of carbon in solution.

The strength of the steel was then expressed by Eq. (8).
Here, $\sigma_0$ is the basic strength, $\sigma_{\rho}$ is the strength attributed to the dislocation density, $\sigma_d$ is the strength attributed to grain and lath boundaries, $\sigma_s$ is the solid solution strength, and $\sigma_p$ is the precipitation strength. Given that there is an approximate relationship between the Vickers hardness, $H_v$, and the strength $\sigma$ (MPa) of $H_v = 0.3 \sigma$, the difference in hardness, $\Delta H_v = H_{va} - H_{vb}$, between samples quenched from a temperature above and a temperature below the Ms temperature is expressed by Eq. (9).

$$\Delta H_v = H_{va} - H_{vb} = 0.3\{(\sigma_0^a - \sigma_0^b) + (\sigma_{\rho}^a - \sigma_{\rho}^b) + (\sigma_d^a - \sigma_d^b) + (\sigma_s^a - \sigma_s^b)\} \quad (9)$$

For temperatures below the Ms temperature, the decrease in dislocation density owing to recovery is restricted, and the grain and lath size of the martensite do not change much, so the term $(\sigma_0^a - \sigma_0^b) + (\sigma_d^a - \sigma_d^b)$ can be set to zero, and Eq. (9) can be substituted by Eq. (10).

$$\Delta H_v = H_{va} - H_{vb} = 0.3\{(\sigma_{\rho}^a - \sigma_{\rho}^b) + (\sigma_s^a - \sigma_s^b)\} \quad (10)$$

Here, $\sigma_{\rho}$ can be expressed by $\sigma_{\rho} = A C^{1/2}$, where the value of $A (=140)$ is determined from fitting the experimental results. The term $\sigma_s$ was calculated using the Ashby–Orowan formula.26

Figure 23 shows the calculated and measured differences in hardness in strength. An example of an application of the model is now given. Conventional steel used in hot stamping contains 0.22% carbon. If the steel is water quenched, then a hardness of 500 Hv is usually obtained, but hot stamped components produced by die quenching exhibit a hardness of around 450 Hv. Figure 24 shows the cooling curve for a die quenching process.27 In a practical operation, a die temperature of around 150°C is normally found, because of the conduction of heat from the heated plates. By inputting the cooling curve for the case of a die temperature of 150°C, calculations using the model result in a decrease in hardness of around 50 Hv. This result explains the hardness of die quenched components being around 450 Hv, and supports the statement of Nishihata et al.28 that auto tempering significantly affects the hardness of hot stamped components. This analysis indicates the importance of controlling the cooling rate below the Ms temperature to strengthen hot stamped components and to minimize fluctuations in the strength.

4. Concluding Remarks

We investigated the influence of heating and cooling conditions on the microstructure and mechanical properties of 0.22%C–2.5%Mn steels. Samples were heated at either 10°C/s or 200°C/s. The cooling conditions used were quenching in water or cooling in air. The following results were obtained.

(1) The water-quenched samples exhibited a maximum hardness of 550–570 MPa. Good agreement was obtained between the measurements and the calculations. The calculations showed that the value of $\sigma_{\rho}$ is negligible, and that the decrease in hardness was chiefly attributable to the decrease...
hardness of the samples cooled in air was significantly lower than that of the water-quenched samples. The reason for this is that the ferrite existing at the intercritical temperature migrated into the austenite during air cooling, and the fraction of martensite was therefore markedly reduced.

3) Samples heated to high temperatures exhibited a full martensite microstructure, but the hardness was much lower than that in water-quenched samples. The difference in hardness was around 80 Hv.

4) To understand the cause of this difference, a model for predicting the precipitation of cementite in martensite was developed. The calculation results show that the difference in hardness can be attributed to the decrease of carbon in solution and indicate the importance of controlling the cooling rate below the Ms temperature to strengthen hot stamped components and minimize fluctuations in the strength.

5) The results of our study strongly suggest that the steel samples investigated are promising candidates for a highly productive new hot stamping technology, as discussed in the Introduction.

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