Study on Phase Transformation Behavior in Continuous Cooling Process

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Abstract. The continuous cooling transition curve (CCT curve) system expresses the influence of cooling rate on the starting point of transition, transformation rate and microstructure are studied. In order to obtain desired microstructures and properties, the appropriate process parameters can be selected according to the continuous cooling transition curve. The CCT curve is very similar to the actual production conditions, so it is of great significance for studying the process of hot stamping of experimental steel.

1. Introduction

Conventional methods for plotting CCT curves include metallography, thermal expansion, and thermal analysis. Among them, the thermal expansion method is a commonly method for determining the phase transition temperature, which uses different principles of thermal expansion coefficient and specific volume of steel to determine the difference. The order of the specific volume of each phase is: austenite < ferrite < pearlite < bainite < martensite; and the coefficient of thermal expansion is in contrast. Therefore, in the phase transformation process of steel, the process of ferrite precipitation and austenite decomposition into pearlite, bainite or martensite are all accompanied by volume expansion[1].

The critical temperature of the experimental steel is determined by the thermal expansion method. By controlling temperatures and cooling rates, the expansion curves of the samples during heating and cooling as the continuous cooling time changes were measured on a thermal dilatometer, namely, the temperature-expansion curves.

2. Experimental equipment and experimental scheme

2.1 Experimental equipment and experimental materials

The experiment was carried out on the Formastor-FII thermal dilatometer instrument. The sample dimensions are shown in the Fig.1.
The microstructures observation were carried out on LEICA 2500-DM metallographic microscope. The morphology and distribution of microstructures were observed on FEI Quanta 600 scanning electron microscope.

2.2 Determination of critical temperatures.
According to Chinese nation code YB5127-1993: “Method for determination of critical point of steel (expansion method)” , the sample was heated to 500℃ at the heat rate of 10℃/s, then heated to 1000℃ at 0.05℃/s, holding for 10 minutes, and cooling to room temperature subsequently. For measuring the Ac$_3$ and Ac$_1$ temperatures, the heat process in the thermal expansion curve was record, as shown in Fig. 2(a). Another sample was heated to 1000℃ at the rate of 10℃/s, holding for 10 minutes, then the sample was cooled to 500℃ with the cooling rate of 0.05℃/s, the cooling process was also record to measure Ar$_1$ and Ar$_3$, then the sample was cooled to room temperature at 10℃/s, as shown in Fig. 2(b).

2.3 Effect of heating temperature on austenite grain size
The experimental scheme is shown in Fig. 3. The phase changer sample was heated to 950℃, 1000 ℃, 1050℃, 1100℃ at 10℃/s, and kept for 5 min, respectively. Then the samples were cooled to room temperature at a cooling rate of 50℃/s. The austenite morphologies and sizes were observed on a metallographic microscope in the thermocouple welding portion.
2.4 Determination of static CCT curve

On the thermal dilatometer instrument, specimens were heated to 1000℃ for the duration of 3 min at the heating rate of 10℃/s, and cooled to 950℃ at the cooling rate of 10℃/s, holding for 10s, then the specimens were subsequently cooled to room temperature with the cooling rates of 1℃/s, 5℃/s, 10℃/s, 20℃/s, 25℃/s, 30℃/s, 40℃/s, 60℃/s and 80℃/s, respectively, as is shown in Fig. 4.

By analyzing the relationship of dilatations, temperatures and the microstructures at room temperature, the phase transition temperature and transformation type at different cooling rates were obtained, and finally the static CCT curve is drawn[2].

3. Experimental results and analysis

3.1 Critical point temperature of experimental steel

The expansion curves of the experimental steel during heating and cooling process are as shown in Fig. 5. The critical temperatures are $A_{c1} = 746℃$, $A_{c3} = 921℃$, $A_{r1} = 729℃$, and $A_{r3} = 865℃$, respectively.

Fig.5 Temperature-expansion curve to identify the transformation temperature
(a)The determination of $A_{c1}$ and $A_{c3}$;  (b) The determination of $A_{r1}$ and $A_{r3}$

3.2 Effect of heating temperature on austenite grain size

The relationship between austenite grain size and temperature is shown in Fig.6. The grain size and morphology of austenite obtained at different heating temperatures are shown in Fig.7. With the temperature increasing, the grain size of austenite increases gradually, and the growth rate also increases. When the temperatures reach up to 1050℃, the growth rate of grains increase significantly[3].
Fig. 6 Relationship of austenite grain size and heating temperature

The heating temperature has a significant influence on the mechanical properties of the steel plate. The original grain sizes of austenite obtained in the low alloy high strength steel and the micro-alloyed steel are decreased as the heating temperature lowered. Due to the two processes occurred inside the heating steel sheet, solid solution of carbides and austenite grain growth.

The austenite grain size is related to the degree of solid solution of the residual carbide. When the carbide is completely dissolved in the austenite, the austenite grains begin to grow vigorously. If there is no second phase point that can prevent grain growth, the austenite grains will continue to grow as the holding time goes on. Furthermore, the higher isothermal temperature leads to the larger grain size[4]. In the experimental steel, Al and N element are combined to form a second phase particle which can prevent the growth of austenite grains during heating process.

Fig. 7 Austenite grain at different heating temperatures
(a) 950℃;  (b) 1000℃;  (c) 1050℃;  (d) 1100℃

The Ac$_3$ temperature was calculated to be 921℃. In order to make carbon and alloying elements dissolved and diffused sufficiently, the heating temperature was generally in the range of Ac$_3$+30~100℃. Considering the influence of the quenching temperature on the austenite grain size, the heating temperature was set to be 950℃, which also included the heating temperature range of hot stamping process.

3.3 Static CCT curve and microstructures
The static CCT curve of the experimental steel is plotted as shown in Figure 8, where the continuous transformation curve of untransformed austenite is composed of ferrite transformation zone, bainite
transformation zone, and martensite zone. The critical cooling rate of martensite was 25°C/s, the start transformation temperature of martensite (Ms) was 426°C, and the final transformation temperature of martensite was 290°C.

Fig. 8 CCT curves of the steel

The microstructures corresponding to Fig. 8 are shown in Fig. 9. It can be seen that ferrite and bainite phases obtained at the cooling rates of 1°C/s ~ 5°C/s, when the cooling rate increases to 10°C/s, the ferrite is disappeared, and the bainite and martensite exist. When the cooling rates reach up to 25°C/s, the microstructure is dominant by martensite phase. Moreover, the martensite lath size shows a decreasing trend as the cooling rate increasing.

Fig. 9 Microstructure under different cooling rates
(a) 1°C/s; (b) 5°C/s; (c) 10°C/s; (d) 20°C/s; (e) 25°C/s; (f) 30°C/s; (g) 40°C/s; (h) 60°C
4. Discussion

4.1 Effect of cooling rate on continuous cooling transformation of austenite

Fig. 10 shows the SEM microstructures obtained at different cooling rates. Combined with the static CCT curves, the microstructures in experimental steel include polygonal ferrite, granular bainite and a small amount of upper bainite obtained at the cooling rate of 1 °C/s. Granular bainite which was consisted of bainitic ferrite and M/A islands distributed on the bainitic ferrite matrix. The SEM morphology of upper bainite was observed to be composed by ferrite lath which grown parallel to the austenite grain boundary and the discontinuous and short rod shape cementite between ferrite lathes[5]. Generally speaking, the rate of growth of bainite was controlled by the diffusion of carbon. And the growth rate of upper bainite depended on the diffusion of carbon in austenite. When the cooling rate reached up to 5 °C/s, the content of ferrite and granular bainite decreases, but feathered bainite content increased and the martensite structure began to appear. When the cooling rate reached to 10-20 °C/s, the ferrite disappears and the structure was composed of feather-like upper bainite and martensite.

When the cooling rate is greater than 25 °C/s, full martensite structure is observed which gradually decreased as the cooling rate increased. When the high-temperature austenite was cooled rapidly and diffusive decomposition was inhibited, which lead carbon to have not enough time to diffuse, and the martensite formed by a shear mechanism, thus the original austenite grain boundary is retained. Therefore, if martensite formed, it shall satisfied two conditions: first, to avoid the transformation of austenite to pearlite or bainite, the cooling rate of supercooled austenite must be greater than the critical quenching speed; second, martensite transformation must be taken under the Ms temperature. In Fig. 8, it can be seen that Ms temperature of the experimental steel was 426 °C and the critical cooling rate was 25 °C/s.

![Fig. 10 Scanning morphology of tested steel with different cooling rates](image)

4.2 Thermodynamic study of martensitic transformation

The phase transform kinetics discusses the issue of phase transform rate, which is the relationship of phase transformation and time, and depends on the nucleation rate and growth rate of the new phase. Transformation thermodynamics researches the relationship of energy and phase transition from the angle of energy transformation and indicates that the stability of the phase state in the system...
determined by the level of free energy, and the most stable state exists in the lowest free energy. When the free energy of new phase is lower than that of old phase, the martensite transformation occurs[6,7].

As to the parent phase (P) and the martensite phase (M) during the martensite transformation, of which the chemical free energies ($G_C$) are a function of temperature and composition. The change rules between the chemical free energy and temperatures of the parent phase and the martensite phase are shown in Fig.11, and the energies are all increased with increasing temperature.

\[
\Delta G_C = G_C^M - G_C^P = 0
\]  

From equation (3-1), when $\Delta G_C < 0$, the free energy of the martensite phase is lower than that of the austenite phase, the transformation driving force formed then, but the martensite transformation does not occur at the equilibrium temperature.

The reasons are as followings: firstly, the martensitic transformation belongs to the shear type which needs to overcome the shear resistance and the matrix of the mother phase changed, which consumes the energy of parent phase; secondly, a large number of crystallographic defects such as faults and twins which lead to the increase of the system energy are generated during the martensitic transformation.; thirdly, the volume phases increased during the martensite transformation process, causing the squeezed austenite hardened and impede the martensite transformation. Therefore, the martensite transformation occurs by increasing the condensate supercooling, thereby the phase transformation driving force increased to overcome the phase change resistance caused by the interfacial energy and the elastic energy. Therefore, at Ms point of martensitic transformation, the variation of system free energy during the martensite form process is obtained by the formula as follows:

\[
\Delta G_C = \Delta G_C + \Delta G_e + \Delta G_s
\]  

Where, $\Delta G_C$ is the molar chemical free energy; $\Delta G_e$ is molar strain energy; $\Delta G_s$ is the molar interface energy.

Therefore, the martensitic transformation must occur when the degree of supercooling is large enough and the chemical driving force is equal to the sum of the strain energy and the surface energy.

5. Conclusion

(1) The critical temperature of the experimental steel was measured by a phase transformation measuring instrument to be $A_{c1} = 746^\circ C$, $A_{c3} = 921^\circ C$, $A_{r1} = 729^\circ C$, and $A_{r3} = 865^\circ C$. The phase diagram of the experimental steel was calculated by Thermo-Calc software. The results show that the temperatures of $A_{c1}$ and $A_{c3}$ measured by the experiment have little difference with the calculated
temperatures of $A_1$ and $A_3$. The $A_C$ temperature of the experimental steel is according with the heating temperature of hot stamping process.

(2) The effects of different heating temperatures on austenite grain size were investigated. As the heating temperature increased, the grain size of austenite and the growth rate increased. According to the $A_C$ temperature (921℃) and the effect of the heating temperature on the austenite grain size, the heating temperature of the experimental steel was set to 950℃.

(3) The static CCT and dynamic CCT curves of the experimental steel were measured. According to the static CCT curve, the critical cooling rate of the experimental steel is 25℃/s, which satisfies the cooling conditions of the hot stamping process. Observing from the microstructures graphs, as the cooling rate increased, the finer martensite laths formed.

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