Effects of cooling rate on the mechanical properties and precipitation behavior of carbides in H13 steel during quenching process

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Abstract

The effects of cooling rate (CR) on the mechanical properties and precipitation behavior of carbides in H13 steel during quenching process were investigated. The retained austenite tends to be more unstable with increasing CRs, while the martensite increases gradually, based on XRD analyses and EBSD results. The values of hardness are increased, and the elongation along with impact energy is decreased, respectively, at higher CRs. Tensile strength remains above 2.0 GPa. Work hardening rates increase considerably in three samples, suggesting that transformation-induced plasticity effect may take place during the tensile test. Moreover, an increase in yield strength is observed when CR exceeds 15 K s⁻¹, possibly due to a high volume fraction of martensite, decline in average grain size and precipitation of fine carbides. Types of the precipitates acquired were identified by electrolysis and XRD analyses. The results indicate the predominant existence of MC, M₆C and M₇C₃, which are confirmed by SEM-EDS analyses and FactSage thermodynamic calculations. The size, volume and distribution of the carbides were also scrutinized under SEM. It is found that the volume fraction and size of the precipitates both decrease with increasing CRs. Based on these experimental data, an optimum CR for the quenching process could be determined to achieve the desired distribution of carbides, which in turn leads to the enhanced mechanical behaviors.

1. Introduction

AISI H13 (4Cr5MoSiV1) is one of the most common hot-work die steels that display high resistance to wear, heat and thermo-mechanical fatigue [1]. Therefore, this material is widely used for high-temperature purposes (e.g., die casting molds, hot forging, hot extrusion and hot rolling), because it can withstand elevated temperatures and load levels [2]. Strength and toughness, as regulated by the carbides and martensite matrix [3, 4], are among the most critical factors that affect the mechanical properties of H13 steel [5].

The austenitization followed by quenching at various rates has long been in the spotlight for the heat treatment of steel, which plays an essential part for both microalloyed [6–9] and alloy steels [10–12]. Souza et al [6] demonstrated that the martensitic transformation kinetics in a Mo-Cr microalloying steel were not directly affected by the cooling process, but instead by its effect on the stability of the austenite phase. Yong et al [7] reported that a high-strength low-alloy steel, treated by air-cooling and tempering, exhibited the optimal combination of strength and toughness, when compared to that treated by oil-cooling or water-cooling and tempering. The vast majority of prior research has focused either on the phase transformation, or on the mechanical properties under different cooling rates (CRs), while much diminished attention has dedicated to the behavior of the precipitates, and its quantitative relationship with CRs during metal quenching. Luo et al[12] assessed the impact of different CRs on the precipitation behavior and transformation characteristics of carbides.
Table 1. Chemical compositions of the H13 steel.

| C  | Si  | Mn | Cr  | Mo | V  | Al | Cu | Ni | P  | S  | N  | O  |
|----|-----|----|-----|----|----|----|----|----|----|----|----|----|
| 0.39 | 0.98 | 0.38 | 5.10 | 1.39 | 1.0 | 0.05 | 0.05 | 0.08 | 0.0011 | 0.0006 | 0.004 | 0.0005 |

in M42 high-speed steel. They found a significant relationship between CR and the precipitation behavior of carbides. However, whether this model is applicable to H13 steel remains to be explored. Previous research has proposed that martensite is formed when the CR is above 1 K s\(^{-1}\). However, a more detailed analysis of the phase transformation and average grain size in a quantitative fashion leaves a lot to be desired.

The effects of quenching [13–19] on the mechanical features and microstructures of H13 steel have been widely explored, as the heat exposure markedly influences the mechanical properties of the final product as H13 steel dies. Kang et al [20] investigated the effects of thermal annealing parameters, such as CR and annealing temperature, on the precipitation behavior of carbides in H13 steel, by using an electron microscopy and a dilatometry. Their results showed that the Mo-rich carbides (e.g., M\(_2\)C and M\(_6\)C) were abruptly precipitated at 948–973 K, together with an annealing temperature of up to 1103 K and a low CR of <0.01 K s\(^{-1}\). Nilsson et al [21] investigated the effects of CRs and austenitization temperatures on the mechanical characteristics of H11 and H13 hot-work steels. Ning et al [22] reported that the precipitation-strengthening contribution of H13 steel ranges from 15% to 18% after quenching. Zhou et al [23] demonstrated that the addition of La in H13 steel could effectively delay the coarsening of austenite grain size, increase the amount of carbides and refine the lath bundles of martensite, which in turn results in the improvement of mechanical properties. Despite these advancements, several key topics, such as how the CR influences the precipitation behavior of H13 during quenching, as well as a thorough analysis of the phase transformation and precipitation change with increasing CRs, still remain to be undertaken.

The present study aims to determine the effects of different CRs on the mechanical properties, microstructures and precipitation behavior of carbides in H13 steel during quenching process. Based on our results, the mechanical properties of H13 steel could be enhanced by selecting the optimal CR for quenching process. Furthermore, a quantitative model describing the relationship among carbide precipitates, CRs and mechanical properties of H13 could be established.

2. Experimental procedures

The chemical compositions of the H13 steel are presented in table 1. H13 specimens were supplied by the Central Iron & Steel Research Institute (Beijing, China). The smelting process involved electric arc furnace melting, electroslag remelting, ladle refining, and vacuum degassing. The diameter of ingot was 220 mm, and the total weight was approximately 500 kg.

The forging process and heat treatment was conducted as follows.

Briefly, the electroslag remelted ingots were hot-forged at 1373 K to a metal bar with a diameter of 105 mm. The hot-forged ingots were annealed for 10 h at 1133 K, furnace-cooled to 773 K, and then air-cooled to room temperature. As shown in figure 1(a), a 105-mm wide by 60 mm thick sample was cut from the central parts of the ingot. After that, three sample bars were cut from the half radius of the sample, as shown in figure 1(b). The three bars were then preheated to 1113 K at 13 K min\(^{-1}\), held for 20 min, heated to 1303 K at 6 K min\(^{-1}\), and held for another 30 min. After cooling in the air (#1), oil (#2) and water (#3) to ambient temperature, the CRs of three samples were measured by thermocouples at approximately 1, 15, and 54 K s\(^{-1}\), respectively. An illustration of the quenching process is shown in figure 2.

In accordance with the Chinese standard GB/T 229-2007, the size of the impact specimens was set to 10 × 10 × 55 mm. The tensile specimens were ø8 × 50 mm in gauge length (fillet radius is specified as 6 mm and gripping end ø12 × 25 mm), according to the Chinese standard GB/T 228.1-2010. The impact value of each sample was acquired by the Charpy V-notch test method using a ZBC2452-B Pendulum Impact Tester (MTS Systems Corp., Shenzhen, China). Both tensile and yield strengths were evaluated using a CMT4105 Electronic universal Tester (MTS Systems Corp., Shenzhen, China). The hardness of samples was assessed by a Mitutoyo Wizzard HR 500 Rockwell Hardness Tester. For the assessment of mechanical properties, three samples were prepared for each condition and the average value of each group was figured out.

Electrolysis was conducted to extract the precipitates. The electrolyte consisted of 10% acetone, 1% tetramethylammonium chloride and methanol, and the current density (I) ranged from 0.04 to 0.06 A cm\(^{-2}\). After electrolysis, the residues were collected, rinsed with deionized water, and then air-dried. The precipitation powders were identified by x-ray diffraction (XRD; with Co K\(_x\) radiation, 45 mA, 35 kV, and 0.02° scan step) and Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) method. The phase compositions of
the steel samples were acquired and analyzed by XRD and the Eva Software which was based on the ICDD JCPDS Standards. Microstructure and precipitate distribution of the H13 steel samples were observed under a SU3500 scanning electron microscope (SEM; Hitachi, Tokyo, Japan) coupled with energy dispersive x-ray spectroscopy (EDS). Electron backscattered diffraction (EBSD) was conducted using a JSM-7001F SEM equipped with a Pegasus XM2 EBSD detector. A mixture of 5% glycerol, 10% perchloric acid and 85% alcohol was used for electrolytic polishing. Orientation Imaging Microscopy (OIM) data analysis was performed to assess the orientation and phase distribution of the samples. The FactSage thermochemical software version 7.2 and FSstel database (GTT Technologies, Aachen, Germany and Thermfact/CRCT, Montreal, QC, Canada) were used for thermodynamic calculations.

3. Results and discussion

3.1. Thermodynamic calculations
The phase distribution diagrams shown in figure 3 were constructed with the aid of FactSage according to the chemical compositions summarized in table 1. Figure 3(a) shows the equilibrium prediction. The phase composition could be $\gamma$ phase, $M_23C_6$, $M_2C$ or MC at 1303 K.
For the continuous cooling process, the Scheil-Gulliver model was employed to provide an alternative prediction of the phase distribution diagram (figure 3(b)). This model can be deployed to analyze the phase transformations of both annealed and quenched samples. It is found that the total amount of precipitates has reduced after continuous cooling, and the main phases are $\delta$ phase, $\gamma$ phase, M$_7$C$_3$ and MC.

3.2. Microstructures
SEM results suggest that the annealed steel is composed of ferrite as well as a significant load of carbides (figure 4(a)), which is reasonable to consider that the purpose of annealing H13 steel lies in spheroidizing the carbides as well as reducing the forging stress. After quenching at 1303 K, the martensite is existed, as revealed in figures 4(b)–(d). The volume and size of the precipitates appear to decrease with increasing CRs. X-ray diffraction analysis was also conducted on the three samples. The diffraction patterns are shown in figure 5.
Using the Eva software with the ICDD JCPDS cards, the amounts of retained austenite (represented by the (111) peak) and martensite (represented by the (110), (200), (211) and (220) peaks) were determined. The content of retained austenite (RA) is 3% and 1.6% in air- and oil-quenched samples, respectively. There is hardly no RA in water-quenched sample calculated by Eva software. These results indicate that the austenite has reformed during the austenitizing process, and reduces sharply as the quenching rate increases. It also shows that the faster the CR, the less stable the RA. This may be due to the fact that carbon has no enough time to diffuse from martensite to austenite, which makes RA among martensite laths carbon poor and unstable when the steel is cooled at a high rate [24].

To characterize the martensite morphology and average grain size (AGS), EBSD analysis was carried out. The IPF maps in figures 6(a), (d) and (g) based on the Euler angles of body-centered cubic martensite indicate that the prior-austenite grains are divided by various martensite packets. Each martensite packet is further subdivided into different martensite blocks and laths-containing blocks with generally parallel directions and different orientations. Figures 6(b), (e) and (h) show the orientation images of the martensite, in which the low angle grain boundaries (2°–5° and 5°–15°) and high angle grain boundaries (>15°) are marked with red, green and blue lines, respectively. The high angle grain boundaries (HAGBs) are typically regarded as martensitic blocks [25]. Based on OIM data analysis, the volume fractions of HAGBs are 62.0%, 62.2% and 66.0%, and the average grain size (AGS) of the high angle grains are 2.83, 2.59 and 2.55 μm, respectively (figures 6(c), (f) and (i)) from OIM data analysis. It can be inferred that the average size of martensite blocks decreases while the volume fraction of martensite increases with increasing CRs, when H13 is quenched at 1303 K.

3.3. Mechanical properties

The mechanical properties of H13 steel are displayed in table 2. After austenitizing and quenching, the strength improves considerably, but impact energy and elongation decrease sharply. Tensile strength reaches up to 2.2 GPa after quenching, and there is no significant change in the values of tensile strength. Yield strength can be increased by almost 200 MPa after water cooling compared with that after air and oil cooling, which can be attributed to the decrease in average grain size and increase of martensite. Based on the results in table 2, the values of elongation and transverse impact energy decrease sharply with increasing CRs. All the three as-quenched samples have a low impact energy, indicating that further tempering may be required to rectify this condition. Combined with XRD phase analysis, it can be seen that the increase in martensite leads to the increase in hardness at higher CRs.

The true stress-strain curves of the as-annealed and as-quenched H13 are shown in figure 7(a). It implies that the as-annealed sample has a large plastic strain, but the as-quenched samples show brittle features, especially water-quenched sample. This may be due to the high content of martensite. In order to illustrate the work hardening behavior of the as-quenched H13 steel, the work hardening (WH) rate curves are drawn in figure 7(b). It shows that WH rate increases after quenching compared with that after annealing. Three as-quenched samples show a similar work hardening behavior, in which the WH rates first increase sharply at strain of 0 ~ 0.02, and then fluctuate at strain of 0.02 ~ 0.06. This indicates that martensite deformation has occurred at first stage where the WH rate is dependent on the mass fraction of martensite. Considering that there are some RA in air-
and oil-quenched samples, it is possible that transformation-induced plasticity (TRIP) effect can take place during the deformation at second stage [4, 26, 27]. Water-quenched sample exhibits the highest WH rate, but decreases sharply after true strain of 0.04 due to the low content of RA. The ductility and toughness has improved greatly when the CR drops down (<54 K s⁻¹). The reason lies in the fact that the more RA in the steel, the more TRIP takes place during deformation, which keeps the steel a high strength and good toughness. Another reason lies in the fact that an appropriate number of precipitates may influence the mechanical properties of the steel [27]. Detailed precipitation behavior will be discussed in the next section. From the above analysis, it can be concluded that the oil-quenched sample has good work hardening ability and good combination of strength and toughness, and hardening of the as-quenched steel is more important for further tempering.

3.4. Evolution of the carbides
After analyzing electrolysis powders by XRD (figure 8), the as-annealed precipitates are identified predominantly as $M_7C_3$, $M_6C$ and VC. Their mass fractions are 43%, 33% and 24%, respectively, as indicated by the ICP-AES analysis. $M_7C_3$ has a hexagonal structure, and is mainly composed of Cr and Fe. $M_6C$ has a body-centered cubic

Table 2. Mechanical properties and Rockwell hardness of H13 steel under different conditions.

| Samples         | UTS (MPa) | YS (MPa) | Elongation (%) | Transverse impact energy (J) | HRC  |
|-----------------|-----------|----------|----------------|------------------------------|------|
| As-annealed     | 616 ± 14  | 312 ± 12 | 26.32 ± 2.11   | 30 ± 15.6                    | —    |
| Air-quenched    | 2176 ± 14 | 1241 ± 33| 5.15 ± 0.94    | 8.6 ± 0.6                    | 48.8 ± 1.2 |
| Oil-quenched    | 2206 ± 11 | 1290 ± 64| 3.36 ± 1.5     | 6.9 ± 1.4                    | 53.9 ± 0.6 |
| Water-quenched  | 2026 ± 50 | 1505 ± 23| 0.8 ± 0.2      | 4.9 ± 0.9                    | 56.8 ± 1.6 |

![Figure 6. IPF-colored EBSD maps, misorientation boundaries maps and grain size distributions of the H13 steel in the air (a)–(c), oil (d)–(f), and water (g)–(i) samples after 1303 K quenching. Misorientation boundaries are colored as follows: red = 2°–5°, green = 5–15°, and blue = >15°.](image)
structure, and is mainly consisted of Mo and Fe. VC has a face-centered structure. After quenching at 1303 K, the composition of the precipitates remains unchanged, while their mass fractions alter in each phase. The mass fractions of M7C3, M6C and VC are 20%, 37% and 43%, respectively. Evidently, the mass fraction of VC increases and that of M7C3 decreases, and the total mass of the precipitates is reduced by a fair margin after undergoing high-temperature quenching. The presence of these carbides is also confirmed by SEM observations, as shown in figures 9–11. The carbides in oil-quenched specimens are V-rich carbides (figure 9(a)), and the results of EDS are shown in figures 9(b)–(e). The Mo-rich carbides are also found in water-quenched specimens (figure 10). The mapping results demonstrate the presence of both types of carbides with large size, as shown in figure 11. Combined with the results from XRD analyses and thermodynamic calculations by FactSage, it can be concluded that Mo-rich carbides are indeed M6C, while V-rich carbides are MC.

3.5. Behavior of the carbides at different CRs

A sample was taken from 10 images, measuring 483.36 × 10 \( \mu \text{m}^2 \) each, for their field of view. Each precipitate was treated as a circle. By deploying image-pro software, the area and equivalent circle diameter (ECD) of each precipitate can be obtained. Size distribution of the precipitates in corresponding to different CRs during the quenching process is shown in figure 12.

It is observed that the carbide precipitates with <0.2 \( \mu \text{m} \) in size can form easily when the CR reaches approximately 54 K s\(^{-1}\). The carbides formed at a CR of 15 K s\(^{-1}\) have their size concentrated between 0.2 \( \mu \text{m} \) and 0.3 \( \mu \text{m} \). The numbers of carbides above 0.3 \( \mu \text{m} \) generally decrease with increasing CRs.

The volume fraction of the carbides can be calculated as follows [28]:

\[
f = \left( \frac{1.4 \pi}{6} \right) \cdot \left( \frac{ND^2}{A} \right)
\]

(1)
$N, f, A$ and $D$ are the total number of carbides, volume of carbides, total area of images, and mean diameter of all carbides, respectively.

Through nonlinear regression analyses of the experimental results, the association of CR with the average size and volume fraction of carbides can be represented by the following formulas [12]:

$$V = M \times e^{-N \times v_c}$$  \hspace{1cm} (2)

$$D = A \times v_c^{-B}$$  \hspace{1cm} (3)

Where $V, v_c, D$ are the volume fraction ($\%$) of the precipitates, the CR (K s$^{-1}$) and the average size ($\mu$m) of the precipitates, respectively. $M, N, A$ and $B$ are the constants derived from the nonlinear regression data (figure 13), which correspond to 2.7347, 0.00969, 0.33256 and 0.05238, respectively.

As can be seen from figure 13, the volume fraction and mean size of the precipitates are both reduced with increasing CRs. Combined with the results of mechanical properties, it can be logically concluded that small carbides are beneficial for improving the yield strength of H13 steel. On the contrary, the impact energy decreases when the CR elevates to nearly 54 K s$^{-1}$. Apart from martensite growth and austenite decomposition, another vital cause is the small amount of precipitates dissolving into the matrix, thus losing their pinning effect on the grain boundaries.

Figure 9. SEM micrograph (a) and EDS analysis (b)–(e) of V-rich carbides after oil quenching.
Based on all these findings, it is speculated that the precipitates have sufficient time to grow and nucleate when the CR is low. With increasing CRs, the nucleation of the carbides may be hindered. Therefore, the volume fraction of the precipitates is remarkably decreased with increasing CRs. During heat treating of H13 steel, the massive quantities of fine carbides are crucial for grain refinement, steel matrix strengthening and impact toughness enhancement. In other words, to produce a H13 steel with excellent strength and satisfactory
toughness, it is necessary to improve the volume fraction ($V$) and reduce the mean size ($D$) of carbides. Thus, an optimum CR can be obtained from oil cooling, which is chosen to enhance the precipitation behavior of carbides in H13 steel.

4. Conclusions

In this study, mechanical properties, microstructural evolution and behavior of the precipitates were investigated for the H13 steel under various quenching rates. The relationships among CR, precipitate size and volume fraction were elaborated. The results can be summarized as follows:

(1) The retained austenite is unstable after various cooling processes. Martensite increases and average grain size decreases gradually with increasing CRs.

(2) Hardness increases, elongation and impact energy decreases with increasing CRs. Tensile strength remains above 2.0 GPa after quenching at $1 \sim 54$ K s$^{-1}$ CR. Yield strength increases greatly when the CR is above 15 K s$^{-1}$. This may be due to the high volume fraction of martensite and fine grains with small carbides in the steel.
(3) Types of the precipitates are predominantly MC, M₆C and M₇C₃, in both as-annealed and as-quenched samples. Notably, their total amount decreases after heat treatment. MC and M₆C are the main precipitates obtained after austenitizing and quenching.

(4) The volume fraction and size of the precipitates are decreased with increasing CRs. An exponential relationship between CR and volume fraction of carbides is observed, \( V = 2.7347 \times e^{-0.00969 \times c_r} \), while the relationship between CR and the average size of carbides corresponds to a power function, \( D = 0.33256 \times c_r^{-0.05238} \). According to the regression coefficient results, an optimum CR can be selected for controlling the precipitation behavior of carbides in H13 steel.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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