Analysis of Austempering Characteristics of Cr12MoV Die Steel

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Abstract—The characteristics of microstructure and properties of Cr12MoV die steel with austempering were investigated by scanning electron microscope, electron backscattered scattering detection, X-ray diffractometer and Rockwell hardness tester. The results indicated that the tempering process reduced retained austenite apparently, especially after direct quenching process which decreased the volume fraction of retained austenite from 24.8\% to 8.5\%. Prolonging the holding time of isothermal quenching from 3h to 5h promoted the transformation of lower bainite and reduced the content of residual austenite. The bainite formed in isothermal combined with the secondary hardening caused by austenite decomposition made the hardness of 55.9-56.6HRC similar to that of 57.2HRC by direct quenching and tempering.

1. Introduction
Cr12MoV is a high carbon and high chromium ledeburite steel. The microstructure contains a large amount of carbide and high carbon martensite, which makes the material obtain high hardness, wear resistance and hardenability. This steel is mainly used for various cold stamping dies and tools under complex shape and heavy working conditions, which is an important cold working die material [1-3]. However, it has a lot of eutectic carbides and retained austenite, whose distribution and content significantly affect the deformation of the material. The deformation of the material is mainly caused by several aspects [4-5]: the zonal, massive or network distribution of eutectic carbides leading to anisotropy and die deformation, the high content of retained austenite (Ar) causing volume shrinkage and instability and thermal stress during cold and hot working and quenching. Among them, the residual austenite and quenching stress can be improved by austempering [6-7]. This process can reduce the quenching stress and obtain the mixed structure of martensite (M) and lower bainite (B\textsubscript{L}). Many studies showed that [8-10], the strength and toughness of M / B\textsubscript{L} duplex structure is better than that of all martensite and all bainite.

In this paper, the effects of different vacuum gas quenching processes (direct gas quenching, lower bainite austempering, metastable austenite + lower bainite austempering) and tempering processes on the microstructure and properties of Cr12MoV were studied, which provides reference basis for the deformation control and later application of the material.

2. Materials and methods
The material used in this study is Cr12MoV die steel. Its chemical composition is listed in Table 1. The process test was carried out on the WZD-40 vacuum carburizing gas quenching multi-functional furnace in the cleaning heat treatment center of Beijing research institute of mechanical and electrical technology.
Co., Ltd., the effective size of the furnace was 600 × 400 × 400mm³, the limit pressure of the gas quenching was 1.5MPa, and the size of the specimen was 80 × 50 × 25mm³, hanging in the center of the furnace. The heat treatment processes were shown in Fig.1. The processes were divided into the following stages: preheating → heating and heat preservation → gas quenching → tempering. The following four kinds of gas quenching processes with 0.6 MPa pressure were adopted:

1) P1: directly gas quenched the specimen to room temperature.
2) P2: gas quenched the specimen to 553K for 3 hours and then gas quenched it to room temperature.
3) P3: gas quenched the specimen to 553K for 5 hours and then gas quenched it to room temperature.
4) P4: gas quenched the specimen to 833K for 10 minutes, gas quenched it to 553K for 5 hours and then gas quenched it to room temperature.

Finally, all specimens were tempered twice at 773K.

Table 1 Chemical composition of Cr12MoV die steel (wt.%)

|   | C     | Si    | Mn    | Cr    | Mo    | V     | P    | S    | Fe    |
|---|-------|-------|-------|-------|-------|-------|------|------|-------|
|   | 1.45~1.70 | <0.4  | <0.4  | 11~12.5 | 0.4~0.6 | 0.15~0.30 | ≤0.03 | ≤0.03 | Balance |

Fig. 1 Schematic diagram of heat treatment process

Then the specimens were ground and etched in 4% Nital solution or polished using 10% perchloric solution. An optical microscope (OM, Zeiss Axio Scope, A1, Carl Zeiss AG, Germany) and a scanning electron microscope (SEM, Zeiss GeminiSEM 500 field emission microscopy, Carl Zeiss AG, Germany) equipped with energy dispersive spectroscopy (EDS) and HKL electron backscattered scattering detection (EBSD) systems were used to evaluate the microstructures. The hardness was tested on the Rockwell hardness tester (HRC, TH300, TIME, China). X-ray diffractometer (XRD, Empyrean, PANalytical, Netherlands) combined with EBSD were used for phase analysis. The continuous cooling transformation (CCT) and time temperature transformation (TTT) curves were obtained by thermal expansion instrument (DIL805L, Baehr, Germany).

3. Results and discussion

3.1. Original microstructure and CCT/TTT characteristics

Fig. 2 shows the microstructure of original material (MOM), which is a high carbon and high chromium ledeburite steel, and the original structure contains 28% eutectic carbides. Eutectic carbides are carbides rich in Fe and Cr, i.e. (Fe, Cr) 7C3 (M7C3). The carbides were precipitated during molten steel crystallization and very stable, which is difficult to eliminate during quenching and tempering. Carbides of this steel showed three sizes: large banded eutectic carbides (LC) with the width of 5-20μm, medium dispersed granular carbides (MC) with diameter of 1-2μm, and small dispersed carbides (SC) with diameter of 0.2-0.5μm.
Combined with the CCT / TTT detection of thermal expansion instrument and the calculation of JMatPro software, the CCT and TTT curves of Cr12MoV were obtained as shown in Fig.3. The phase transformation characteristics of continuous cooling and isothermal cooling of the material at different cooling rates can be analyzed. The martensite can be obtained when the continuous cooling rate is greater than 0.5K/s, and the lower bainite can be obtained when the isothermal temperature is below 620K. Different properties can be obtained by adjusting the holding time of the lower bainite area and the content of lower bainite and martensite. By adjusting the holding time of the lower bainite region, the content of lower bainite and martensite can be improved to optimize the performance.

3.2. Effect of quenching and tempering processes on microstructure

In Fig. 4 and Fig. 5, it is obvious that after holding at 1293k for 30min, small dispersed carbides of Cr12MoV were obviously dissolved in austenite, and the content was reduced from about 9% in the original structure to 2.4% - 3%, while the other two types of large-size eutectic carbides were difficult to dissolve. The distribution of large-size carbide can be improved only by optimizing the forging process.
After 0.6 MPa gas quenching to room temperature, as shown in Fig. 6, it can be seen that M and Ar were formed at room temperature after quenching. After quenching by P2, P3 and P4 process (Fig. 7), a large number of lower bainite were generated. Carbides with an angle of about 60° with its main axis were distributed in the flake ferrite, which were mixed with martensite to form M / Bl duplex structure. At the same time, there were a large amount of Ar.

The quenched specimens were tempered twice at 500 °C for 2 hours, and the phase distribution differences before and after tempering were compared. As shown in Fig. 8, after tempering, volume fraction and the size of Ar decreased significantly. In addition, combined with BSD and XRD, the volume fraction of Ar was obtained. As shown in Fig. 9, it can be seen that with the isothermal holding time increasing from 3h to 5h, the relative transformation of lower bainite was relatively more sufficient, and the amount of Ar decreased from 29.5% to 26.2%, while the holding of metastable austenite zone had no significant effect on Ar. During tempering, in Ar some carbides precipitated, which reduced the carbon content of 8% - 9% Ar and increased the martensite start temperature (Ms). Martensite was formed in the subsequent tempering process, and then transformed into tempered sorbite. Therefore, it
can be seen from EBSD and XRD that tempering significantly reduced Ar. The transformation of austenite in the tempering process of process P1 was more sufficient, because the carbon content of retained austenite in process P1 was lower than that in other processes, and the Ms was relatively higher, which made the transformation easier. Therefore, 16.3% of Ar was transformed in the tempering process.

Fig.8 EBSD maps of Cr12MoV with quenching and tempering (red: Ar, blue: F, yellow: M₇C₃, green: Fe₃C, white: zero resolution)

Fig.9 Volume fraction of Ar with different processes

3.3. Effect of quenching and tempering processes on properties

At the same time, the transformation of Ar also significantly affected the properties. As shown in Fig. 10, the hardness of the specimen directly cooled to room temperature decreased from 64.9HRC to 57.2HRC after tempering, which was due to the lower secondary hardening effect of austenite during tempering than the softening effect caused by the transformation from martensite to tempered sorbite. The hardness of specimens with isothermal quenching was lower than that of process P1 after quenching, which was due to the lower hardness of BL than that of M significantly. However, during tempering, as shown in Fig. 11, in BL the carbon did not diffuse sufficiently during tempering, which induced the specimen to maintain the morphology of BL and high hardness. In addition, the secondary hardening effect formed by austenite increased the hardness and maintained the hardness close to that of the process P1. At the same time, the isothermal quenching process can significantly reduce the thermal stress caused by temperature gradient in the quenching process, especially the two-stage isothermal of metastable austenite zone + lower bainite zone in process P4. And the specific volume of lower bainite was smaller than that of martensite, which caused the smaller deformation of specimen with process P4 than that with process P1. Therefore, process P4 can obtain ideal microstructure and properties and reduce the deformation of the workpiece during heat treatment.
4. Conclusions
The austempering characteristics of Cr12MoV die steel were analysed. The main results are as follows:

(1) During quenching, small dispersed carbides were obviously dissolved in austenite, while the other large-size eutectic carbides were difficult to dissolve. The distribution of large-size carbide can be improved only by optimizing the forging process.

(2) Tempering process can apparently reduce retained austenite, and the decomposition of retained austenite in direct quenching process was more significant than that in isothermal quenching process. Prolonging the holding time of isothermal quenching can promote the transformation of lower bainite and reduce the content of residual austenite.

(3) The carbon diffusion of bainite formed in isothermal quenching was not obvious in the subsequent tempering process, which combined with the secondary hardening caused by austenite decomposition made the hardness similar to that of direct quenching and tempering. At the same time, it can effectively reduce the deformation of the workpiece.

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References
[1] Wang, J., Zhou, J., Zhu, S., Zhang, J. (2017) Friction properties of groove texture on Cr12MoV surface. J. Cent. South Univ., 24: 303–310.
[2] Wu, B., Liu, P., Duan, J., Deng, L. (2016) Xiaoyan Zeng, Xizhao Wang. Study on picosecond pulse laser ablation of Cr12MoV cold work mold steel. Mater. Des., 110: 549–557.
[3] Liu, C., Lu, D., Luo, J., Zou, J., Lu, L. (2016) Effects of pulse-plasma detonation treatment on microstructure and properties of Cr12MoV steel. T. Mater. Heat Treat., 37: 177–183.
[4] Long, B. (2019) Analysis of cracking failure of Cr12MoV die. MW Met. Form., 10: 18–20.
[5] Wu, X., Xu, N., Shi, J., Ge, C., Ma, K., Zang, Q. (2009) Control of eutectic carbides in Cr12MoV die steel and research progress in China. MW Met. Form., 9: 72–74.
[6] Lu, J. (2020) Austempering of H13 and Cr12MoV die steels. Heat Treat., 35: 42–44.
[7] He, W., Li, Z., Zhang, X., Wei, X., Li, J., Li, S. (2021) Effect of bainite isothermal quenching on
microstructure and thermal fatigue performance of H13 hot working die steel. T. Mater. Heat Treat., 42: 81–87.

[8] Wang, Q., Lu, J., Zou, Z. (2011) Heat treatment of martensite/bainite dual phase of Cr12MoV steel. Heat Treat. Met., 36: 63–65.

[9] Liu, D., Xu, H., Yang, K., Bai, B., Fang, H. (2004) Effect of bainite/martensite mixed microstructure on the strength and toughness of low carbon alloy steels. Acta Metall. Sin., 40: 882–886.

[10] Zhang, Q., Chu, Z., Chen, D., Jin, K. (2014) Austempering process and toughness of 5Cr4W5Mo2V hot-working die steel. T. Mater. Heat Treat., 35: 94–96.