Effect of Tempering Time on Microstructure and Properties of 65Si2CrV Spring Steel

Siming Huang\textsuperscript{1,a}, Liejun Li\textsuperscript{1,b}, Zhengwu Peng\textsuperscript{1,c}, Xianqiang Xing\textsuperscript{2,d}, Jixiang Gao\textsuperscript{3,e}, Bokun Wang\textsuperscript{1,f}

\textsuperscript{1}National Engineering Research Center of Near-net Shape Forming Technology for Metallic Materials, South China University of Technology, Guangzhou 510640, China
\textsuperscript{2}Guangzhou Osai Steel Wire Technology Co., Ltd., Guangzhou 511450, China
\textsuperscript{3}Guangdong Polytechnic Normal University, Guangzhou 510635, China
\textsuperscript{a}email: scutsiminghuang@163.com, \textsuperscript{b}email: liliejun@scut.edu.cn, \textsuperscript{c}email: pengzw@scut.edu.cn, \textsuperscript{d}email: orsa8899@163.com, \textsuperscript{e}email: gjx205@163.com \textsuperscript{f}email: 2654029688@qq.com

Abstract: The microstructure evolution and mechanical properties of 65Si2CrV spring steel at 420 °C with different tempering time were studied in this paper. The results show that the extension of tempering holding time will make the martensite recover more fully, so as to transform into massive ferrite, and the precipitated carbide increases at the same time. With the extension of tempering holding time, the strength of the sample decreases gradually. When the tempering time exceeds 300s, the ratio of length and width of carbide decreases gradually, spheroidization occurs and plasticity decreases sharply. Excellent comprehensive properties were obtained when holding for 300s. The tensile strength was 2083MPa, the elongation was 8.3%, and the product of strength and plasticity was 17.28 GPa ·%.

1. Introduction
Spring steel, as an important part of automobile shock absorption system, is required to have excellent mechanical properties because of its harsh service environment. In order to meet the needs of lightweight and energy saving, higher requirements for the strength of suspension spring materials are put forward, and the development of better mechanical properties of suspension spring materials and industrialization work is increasingly urgent\cite{1-3}.

65Si2CrV is an ultra-high strength hot formed spring steel developed in recent years. It is a tempered troostite structure with good plasticity and toughness and good elastic limit and yield limit obtained through certain quenching and tempering process. It is mainly used for automobile suspension spring, clutch spring and valve spring. The researches on the tempering process of hot formed spring steel mainly focuses on 60Si2CrV\cite{4,5}, 55SiCr\cite{6,7}, 51CrV4\cite{8,9} and other steel grades. For the study of tempering process of spring steel, Li Ying et al.\cite{10} found that the tensile strength of 60Si2CrVAT spring steel reached 1950-1970MPa and the elongation was 9-12% when the austenitizing temperature of 910°C was held for 60 min and then quenched at 410°C for 90 min. Zhu Jie et al.\cite{11} Studied the effects of different tempering temperature and time (tempering at 400-520 °C and holding for 30-120min) on the microstructure and properties of 60Si2CrVAT spring steel. The results show that the microstructure obtained by tempering the material at 400 °C to 520 °C is tempered troostite. With the increase of temperature and time, needle like ferrite changes to granular and massive. The mechanical properties
were the best (tensile strength 1993 MPa, elongation 9%) after oil cold quenching at 910 °C for 30 min and oil cold tempering at 430 °C for 60 min. In 55SiCrV spring steel, Meng Jian et al.[12] found that the best mechanical property matching (Rm=1815 MPa, Z=28%) can be obtained after quenching at 900 °C and tempering at 430 °C for 30 min. In Peng Erbao's[13] research, with the increase of tempering temperature, the carbon atoms in the martensite of 51CrV4 spring steel gradually desolvate, the matrix structure gradually recovers, the content of spherical cementite gradually increases, the characteristics of martensite lath gradually disappear, the strength and hardness gradually decrease, and the elongation gradually increase at the same time. At 480 ~ 520 °C, the strength and hardness of 51CrV4 steel decrease rapidly and the elongation increases greatly with the increase of tempering temperature. At 520 ~ 560 °C, the strength and hardness of 51CrV4 steel decreased slowly and the elongation increased slightly with the increase of tempering temperature. In contrast, there are few reports on the related research of 65Si2CrV, so it is very necessary to study the heat treatment process of 65Si2CrV spring steel.

In this paper, different tempering holding time are set to test, so as to obtain materials with different microstructure, and analyze the microstructure evolution and mechanical properties of 65Si2CrV in the tempering process, so as to provide guidance for optimizing the production process.

2. Materials and test methods

2.1 Material and heat treatment
The chemical composition of 65Si2CrV steel in this experiment is shown in Table 1. After pickling and phosphating, the experimental steel wire was cold drawn to φ 12.5 mm semi-finished steel wire is then heated to the austenitizing temperature of 940 °C in an electric heating tubular furnace for 5 minutes before quenching. The quenched sample is tempered in a lead bath furnace. The tempering temperature is set to 420 °C and the holding time at each tempering temperature is set to 50 s, 100 s, 150 s, 200 s, 300 s, 500 s and 700 s.

| C    | Si   | Cr  | Mn  | V   | Ni  | Cu | P  |
|------|------|-----|-----|-----|-----|----|----|
| 0.66 | 1.44 | 0.71| 0.71| 0.11| 0.02| 0.02| 0.008|

2.2 Mechanical properties
The sample after heat treatment shall be cut by wire-electrode cutting to φ 12.5 mm × 5 mm cylinder, polished smooth with sandpaper, and then polished. The microhardness of the polished sample under different heat treatment processes is tested by DHV-1000Z Vickers hardness tester. The load selected for the test is 1000 g and the loading time is 10 s. Select 10 test values for each sample, and then calculate the average value as the microhardness value of the tested sample.

Conduct tensile test at room temperature on WAW-600 600KN microcomputer controlled electro-hydraulic servo universal testing machine with constant strain rate of 1 mm / min. The tensile test shall comply with the standard GB / T 228.1-2010.

2.3 Characterization of microstructure
The sample after heat treatment is cut into a cylindrical sample with a thickness of 5 mm by wire-electrode cutting, and the surface of the sample is polished with 350 #, 800 #, 1000 #, 1200 # and 1500 # sandpaper before polishing. After the polished sample is etched with 3% nitric acid alcohol, the metallographic structure is observed under Leica DNI8A intelligent digital metallographic microscope. The SEM morphology was observed under Nova Nano SEM-430 ultra-high resolution field emission scanning electron microscope.

3. Results and discussion

3.1 Microstructure
Fig. 1 shows the metallographic structure of the sample in quenched state and tempering temperature at
420 °C for different tempering times. Compared with quenched samples, tempered at 420 °C, the microstructure is mainly tempered troostite. However, when the holding time continues to increase, the martensite begins to coarsen, and white massive ferrite appears, and its number increases with the extension of holding time. This is because some martensite structures have begun to recover after tempering at 420 °C; The change of metallographic structure is more obvious when the holding time exceeds 150s at 420 °C tempering temperature. The amount of white ferrite increased significantly with the extension of holding time, and the area also increased, and the shape gradually changed from point and sphere to fine strip and block. It can be seen that tempering at 420 °C and prolonging the holding time will make the martensite recover more fully and transform into massive ferrite.

Fig. 1 The metallographic structure of 65Si2CrV under different tempering holding time.
(a) quenched sample; (b) 420°C-50 s; (c) 420°C-100 s; (d) 420°C-150 s; (e) 420°C-200 s;
(f) 420°C-300 s; (g) 420°C-500 s; (h) 420°C-700 s.

Fig. 2 a-h are SEM images of the sample as quenched and tempered at 420 °C for different times. As shown in Fig. 2 b and c, compared with the quenched structure in Fig. 2 a, the microstructure of the sample is tempered martensite and almost no precipitated carbide when the tempering holding time is short; When the tempering holding time is extended to 150 s, carbides begin to precipitate, and continue to extend the holding time, and the number of precipitated carbides increases significantly, as shown in Fig. 2 d-h. When the tempering holding time is prolonged, the actual temperature of the sample exceeds
the decomposition starting temperature of martensite, the microstructure begins to decompose martensite, and the carbon element originally dissolved in martensite begins to desolve and form at the grain boundary ε- Carbides, thereby reducing the contribution of solution strengthening, thereby reducing hardness and strength. When the holding time is extended to 300 s, the actual temperature reaches 420 °C and precipitates from martensite along the grain boundary ε- Carbide dissolves into flake θ- Carbides are distributed in martensite in parallel, as shown in Fig. 2 f. As shown in Fig. 2 g-h, when the holding time is further extended, the fine flake carbides begin to aggregate and grow, the ratio of length to width of carbides gradually decreases and spheroidization occurs. At this time, in addition to the transformation of carbides, the tempered martensite structure will reduce the dislocation density with the extension of tempering holding time, and the dislocation strengthening effect will be weakened. At the same time, the aggregation and growth of carbides will be reduced, which not only reduces the precipitation strengthening effect, but also weakens the original fine grain strengthening.

Fig. 2 SEM morphology of 65Si2CrV samples under different tempering. 
(a) quenched sample; (b) 420°C-50 s; (c) 420°C-100 s; (d) 420°C-150 s.(e)420°C-200 s; (f) 420°C-300 s; (g) 420°C-500 s; (h) 420°C-700 s.

3.2 Mechanical property analysis
Fig. 3 and Table 2 show the mechanical properties of samples at 420 °C for different tempering times and the variation trend with holding time. It can be seen that the strength of the sample decreases
gradually with the extension of tempering and holding time. When the tempering holding time is 50 s, the tensile strength of the sample is the highest, 2240 MPa, while the plasticity is the lowest, and the elongation is only 2.5%. The elongation reaches the best when the tempering holding time is 300 s, which is 8.5%. At this time, the tensile strength and Vickers hardness are 2083 MPa and 591.6 HV respectively. However, when the tempering time is extended to 700 s, the strength decreases and the plasticity decreases sharply. The reason is that with the extension of tempering and holding time, the precipitation of spherical carbides increases and tends to aggregate along the grain boundary. The carbon content in the original martensite decreases, which reduces the solid solution strengthening of martensite. It can be seen from Fig. 3 that the product of strength and plasticity of the sample reaches the maximum value at 300 s tempering, which is 17.28 GPa ·%. Once the holding time exceeds 300 s, not only the strength and hardness will be further reduced, but also the plastic toughness will be reduced. Overall, excellent mechanical properties can be obtained by tempering at 420 ℃ for 300 s.

### Table 2 Comprehensive mechanical properties of samples under different tempering parameters

| Tempering holding time/s | Tensile Strength/MPa | Elongation/% | Hardness/HV |
|--------------------------|----------------------|--------------|-------------|
| 50                       | 2240                 | 2.5          | 636.5       |
| 100                      | 2180                 | 5            | 629.9       |
| 150                      | 2134                 | 7            | 605.5       |
| 200                      | 2106                 | 8            | 607.8       |
| 300                      | 2083                 | 8.3          | 591.6       |
| 500                      | 2000                 | 6.5          | 565.0       |
| 700                      | 1934                 | 6            | 550.5       |

### 3.3 Fracture morphology analysis

Fig. 4 is the tensile fracture diagram of tempering at 420 ℃ for different times. It can be seen from the figure that when the tempering and holding time is 50 s, there is an obvious fan-shaped pattern on the fracture, that is, cleavage fracture, which is in line with the poor plasticity of the material after tempering and holding for 50 s. When the tempering holding time is extended to 200 s, the fracture diagram is shown in Fig. 4b. At this time, the fracture morphology is mainly fracture dimples, and a large number of equiaxial dimples are distributed. At this time, ductile fracture occurs. Continue to extend the holding time to 300 s and 700 s, and the fracture morphology is shown in Fig. 4 c and d. At this time, the fracture
morphology is also dominated by dimples, but it is obvious that with the extension of holding time, the average diameter of dimples is larger, the depth of dimples is deeper, the cross-section fluctuation is large, and the plasticity of the material is better. In Fig. 4 c, when tempered at 420 ℃ for 300 s, the dimples have higher density and uniform distribution, which also confirms the above mechanical properties.

Fig. 4 SEM morphology of tensile fracture under different tempering holding time
(a) 420℃-50 s; (b) 420℃-200 s; (c) 420℃-300 s; (d) 420℃-700 s.

4. Conclusions
The microstructure evolution process and mechanical properties of 65Si2CrV spring steel under different tempering holding time are studied. The results are summarized as follows:
1. The extension of tempering holding time will make the martensite recover more fully and transform into massive ferrite.
2. With the extension of tempering holding time, the strength of the sample decreases gradually. When the tempering time exceeds 300s, the ratio of length and width of carbide decreases gradually, spheroidization occurs and plasticity decreases sharply.
3. Excellent comprehensive properties were obtained when holding for 300s. The tensile strength was 2083MPa, the elongation was 8.3%, and the product of strength and plasticity was 17.28 GPa ·%.

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