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Enhanced Axial Tension-tension Fatigue Resistance of a 51CrV4 Spring Steel by Cryogenic Treatment

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Abstract: *51CrV4 spring steel is widely used in heavy duty dump trucks ascribing to its superior mechanical properties. The fatigue life and strength of dump trucks are the main performance indicators that must be considered in the manufacturing process. Cryogenic treatment (CT) can improve the main performance of materials which has been proved by recently research. The effect of cryogenic treatment CT on the axial tensile fatigue strength of 51CrV4 spring steel was studied in this paper. The results showed that the axial tension-tension fatigue life of 51CrV4 spring steel after CT was significantly higher than conventional heat treatment (CHT) samples. The microstructure of 51CrV4 leaf spring material is mainly acicular bainite and thin strip martensite after CT. Compared with CHT, CT makes the microstructure of the material more compact. The introduction of cryogenic treatment (CT) before tempering makes the Ca element in the material aggregate, and the micro amount of Ca has the function of deoxidizing and desulphurizing and improving the morphology of sulfide, thus enhancing the fatigue life of the material.*

Keywords: *axial tension-tension fatigue, 51CrV4 spring steel, cryogenic treatment*

1.Introduction

51CrV4 plate spring steel has certain strength and good plastic toughness, which is widely used in the manufacture of automobile suspension steel plate [1]. Strength requirements are involved in the design of heavy vehicle suspensions, especially in terms of impact and fatigue [2]. Microstructural characteristics are one of the primary factors affecting a components fatigue life. There are two main approaches of leaf spring manufacturing for altering the microstructure of the spring material: chemical composition selection and heat treatments [3]. Besides the chemical composition and steelmaking process, heat treatments are normally the last step to achieve the desired steel properties [4]. Nowadays, heat treatment has been widely used to improve the properties of materials.

ZHOU et al [5] studied the ultrahigh cycle fatigue failure behavior and crack initiation mechanism of 51CrV4 high strength spring steel under different heat treatment conditions (quenching + medium tempering and annealing). The results showed that the microstructure of quenching & tempering samples was typical tempered troosite structure, and the carbide size was about 0.5μm, distributed evenly in the matrix. In addition, the fatigue cracks of quenched and tempered specimens originated from internal inclusions. LI et al [6] studied the effect of heat treatment on the microstructure and mechanical properties of 51CrV4 spring steel. The comprehensive properties of the spring steel are the best after oil-quenching at 860℃ for 40min and tempering at 450℃ for 90min.The fatigue fracture has transformed into plastic fracture, and the fatigue life increases from 6.3×10^4 times to 1.1×10^5 times, which meets the performance requirements of the spring steel. Xu et al [7] proposed that the microstructure of spring steel can be refined by microalloying and deformation heat treatment. Induction heat treatment can refine grain, reduce decarbonization, and improve the plasticity and toughness, elasticity and fatigue strength of steel. Kubit et al [8] showed that the specimens were quenched to 860℃ for 2.15min and next tempered to 480℃ for 15min. The experimental studies showed that this method visibly increased the fatigue strength limit, for QT (Quenching + tempering) by about 5.3%. Mechanical properties were also

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better in comparison to specimens without treatment. As mentioned above, it can refine the microstructure of 51CrV4 spring steel by proper adjustment of heat treatment parameters, which can improve the properties of the material. However, there are still many deficiencies after the heat treatment of steel [9]: 1) The internal structure of steel after heat treatment contains austenite about 10%~20%. Austenite is easily converted to martensite when subjected to external load or temperature change. Due to the different specific capacities of austenite and martensite, the material will expand irregularly, making it difficult to guarantee the dimensional accuracy of the material. 2) The grain inside the material is coarse and the carbide solution supersaturation occurs after heat treatment. 3) The specimen after heat treatment will inevitably have residual internal stress, which will have a negative impact on the fatigue strength and other properties of the material. With the release of residual internal stress, it is easy to cause deformation of the specimen.

Given the remaining defects of heat treatment, relevant researchers are constantly exploring new ways to enhance and improve the properties of materials. Cryogenic technology gradually entered the field of vision from the1980s and was introduced into the heat treatment process of materials. Cryogenic technology can promote the transformation of retained austenite into martensite, refine the grains and precipitate finely dispersed carbides. Different collocation can be carried out between quenching, tempering, cryogenic treatment and other processes, so as to improve the properties of the material [10-11]. In recent years, cryogenic treatment has been gradually applied to various fields. In particular, a large number of research achievements have been made in the cryogenic treatment of cemented carbide tool materials, high-speed steel, titanium alloys and aluminum alloys [12-24]. However, the cryogenic treatment technology of spring steel is not very mature, and some relevant studies are shown as follows:

V.R.M. Goncalves et al. [25] conducted 24 h of cryogenic treatment (Quenching + cryogenic treatment for 24 h + Tempering) on 51CrV4 spring steel, and found that its fracture toughness had no positive effect compared with conventional heat treatment, and the results were very similar. R.C. Özden1 et al. [11] studied the cryogenic treatment of spring steel after tempering. If deep cryogenic treatment is applied after the conventional heat treatment (quenching+ tempering), however, the coarse carbides form in the micro-structure and the improvement in the mechanical properties of the spring steel is limited.

This paper aims to study the fatigue performance and microstructure of 51CrV4 after three times of CT before tempering (quenching + tempering). In terms of macroscopic properties, the Rockwell hardness, impact toughness and axial alternating load fatigue tests were performed on the cryogenic and uncryogenic samples respectively. Moreover, the effect of ultra-low temperature treatment on the microstructure of 51CrV4 plate spring steel and its cryogenic strengthening mechanism were studied by means of scanning electron microscopy (SEM).

2. Materials and methods

A common hot-rolled steel plate, with a grade of 51CrV4 (International standard), was chosen as a target material, and its chemical composition is shown in Table 1.

Impact tests were performed according to ASTM E23 [26]. The Charpy impact test performed was The specimens' size and surface treatment method of the tensile test were the same as those of fatigue specimen subjected to axial alternating load, and tensile tests were conducted at a strain rate of 4.7×10^{-3} s^{-1} by a WDW-10D microcomputer controlled electronic universal testing machine manufactured by Jinan Test Gold Group Co., Ltd., Jinan, Shandong Province.

Table 1. Chemical composition of 51CrV4 spring steel studied（wt%）

C	Si	Mn	S	P	Cr	Ni	Cu	V
0.48	0.30	0.68	0.025	0.025	1.02	0.30	0.20	0.12

with standard Charpy V-Notch (CVN) specimens (Figure 1a) performed at room temperature. The fatigue samples were cut into the thin plate shown in Figure 1b according to ASTM E466 [27]. All prepared samples were divided into two groups of experimental groups. The experimental conditions of each group are shown in Table 2. Quenching and tempering parameters were defined according to the manufacturer recommendations. The CT process diagram was shown in Figure 2.

Table 2. Experimental conditions

Groups	Quenching (℃) +Time(min)	Oil (℃) +Time(min)	Cryogenic conditions	Tempering (℃) +Time(min)
CT	900+30	70+30	-196℃+1℃/min+ 24h+3times	480+60
CHT	900+30	70+30	-	480+60

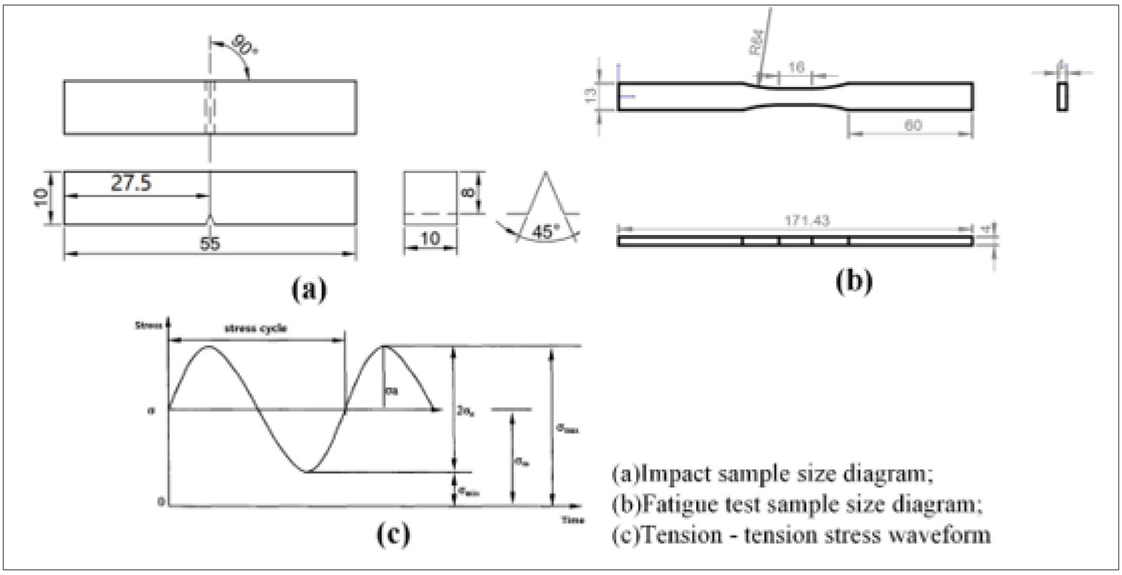

Figure 1. Experimental sample diagram and stress wavwform

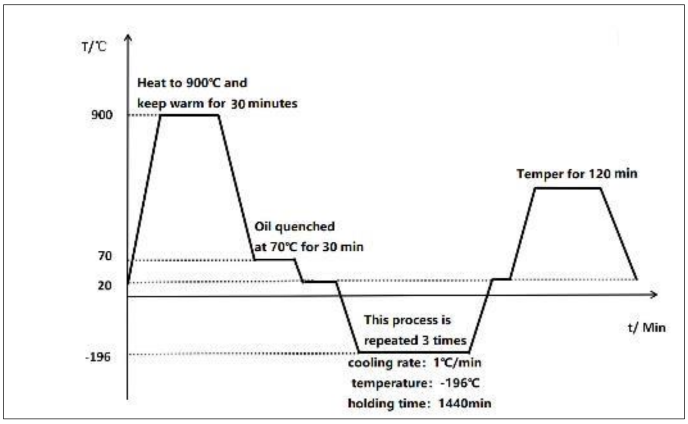

Figure 2. Heat treatment process flow chart

Fatigue tests were also performed at room temperature under controlled amplitude of alternating stress in axial loading and according to ASTM E466 using a High Frequency Fatigue Testing Machine of PLG- 100 type developed by Changchun Test Machinery Research Institute, Changchun City, Jilin Province, China. Fatigue testing employed alternating tension-tension loading with sinusoidal waveform with R (R is the ratio of the minimum stress S_{min} to the maximum stress S_{max}) equal to 0.1 at a frequency of 110 Hz (Figure 1c).

In general, only the S-N curve obtained when the R=-1 is called the basic S-N curve. The average stress corresponding to the basic S-N curve is 0, in fact, the average stress has a great influence on the fatigue life of the material [28]. when S_m >0, that is, the average stress behaves as tensile average stress, and the S-N curve moves down as the basic S-N curve, indicating a decrease in fatigue life under the same stress level, which is due to the adverse effect of tensile average stress on fatigue, resulting in a decrease in the fatigue performance of the materials. when S_m <0, that is, the average stress behaves as the compressive average stress, the S-N curve will shift upward relative to the basic S-N curve at this time, which indicates that the fatigue life increases under the same stress level, which proves that the compressive average stress is beneficial to fatigue. A large number of experiments show that the stress-life relationship is closer to reality in the modified average stress effect. Researchers have concluded the specific conversion formula for different metal materials [29]. (1) Goodman correction equation: $S_e = \frac{S_a}{1-\frac{S_m}{S_b}}$ (2-1); (2)Gerber correction equation: $S_e = \frac{S_a}{1-\left(\frac{S_m}{S_b}\right)^2}$ (2-2); (3)Soderberg correction equation: $S_e = \frac{S_a}{1-\frac{S_m}{S_y}}$ (2-3).

Annotation :

1) S_e means the stress amplitude when the stress ratio is equal to -1 and the average stress is equal to 0;

2) S_a means the stress amplitude at the stress ratio is unequal to -1 and the average stress is unequal to 0;

3) S_m means the average stress at the stress ratio is unequal to -1 and the average stress is unequal to 0;

4) S_b means the tensile strength of the material;

5) S_y means the yield strength of the material.

Goodman correction formula is generally accurate for brittle materials. The tensile strength generated is close to the real fracture stress, and the variation of fatigue life is more consistent with the test results [30]. Soderberg modified formula is for most engineering alloys and estimates fatigue life conservatively [31]. Gerber correction formula is mainly aimed at plastic materials, and the stress-life relationship obtained by this average stress is more accurate [32]. In this study, the main research object is spring plate with good plastic toughness, so the Gerber formula is used to correct the average stress.

Table 3. Axial alternating stress level

Stress level	S_a/MPa	S_m/MPa	R	S_{max}/MPa	S_{min}/MPa	f/Hz
1	299.38	365.94	0.1	665.32	66.56	110
2	270.00	330.31	0.1	600.31	60.31	110
3	243.75	298.13	0.1	541.88	54.38	110
4	217.50	263.72	0.1	451.22	45.12	110

Four levels of stress were set in this experiment (Table 3), and the effective number of samples in each heat treatment method was 12. Three samples were tested at each level of stress, and the stress increment was within 5% of the estimated fatigue limit. Statistical analysis of the results was performed according to the S-N data linearization technique where S (amplitude of alternating stress) is the independent variable (controlled) and N (number of cycles required to fail) is the dependent variable (random) according to ASTM E466.

In this test, when the number of cycles of the specimen at a certain stress level exceeded 10 million cycles, it was considered that the fatigue fracture of the specimen would not occur at the stress level, and the test would be terminated.

3. Results and discussions

3.1. Rockwell hardness

Table 4. Rockwell hardness test data

Experimental groups	sample	1#(HRC)	2#(HRC)	3#(HRC)	4#(HRC)	5#(HRC)	Average (HRC)	The final result (HRC)
CT	CT-1	42.2	41.8	42.0	43.1	41.5	42.12	42.61
	CT-2	44.4	44.4	44.5	40.5	41.7	43.10	
CHT	CHT-1	42.1	42.9	43.0	43.1	42.3	42.64	42.45
	CHT-2	42.5	42.1	43.0	42.0	41.7	42.26	

Rockwell hardness measurements were performed initially, primarily for evaluating the heat treatment parameters chosen. The average hardness of 51CrV4 plate spring steel sample after CT was 42.61HRC, which was 0.38% higher than that of the sample after CHT. All test data were shown in the Table 4. It was important to note that the average hardness of the sample after both treatments is within the standard range of the spring material: 40.5 HRC minimum and 48 HRC maximum [33]. The results of this experiment show that cryotreatment has mild effect on Rockwell hardness of 51CrV4 spring steel.

3.2. Impact toughness

Table 5. Results of impact tests

Samples	Quenching temperature	Cryogenic temperature	Cooling rate	Cold time	Cryogenic times	Tempering temperature	Impact toughness	standard deviation
CT samples	900℃+70℃ oil cooling	-196℃	1℃/min	1440min	3 times	480℃	48.72J/cm^2	0.1867
CHT samples	900℃+70℃ oil cooling	-	-	-	-	480℃	46.86J/cm^2	0.0469

Then the impact toughness test was carried out for the samples with two treated methods. Results obtained with Charpy impact tests are presented in Table 5. The final results showed that the impact toughness of 51CrV4 plate spring steel samples after CT was 3.97% higher than that of the samples after CHT. Through the above test, it can be confirmed that the effect of cryogenic treatment on the hardness and impact toughness of 51CrV4 spring steel is small. This conclusion is consistent with V.R.M. Goncalves et al [13].

The fracture of the impact toughness sample can be visually observed from Figure 3. By comparing the microstructure of the impact fracture of the two heat-treated samples, it could be found that the fracture of the samples after CT formed a large diameter dimple of the same axis, and the dimples were deep and the bottom was flat. In terms of fracture mechanisms, the fracture form with this feature belongs to ductile fracture. However, the dimples with large and deep diameters can absorb more impact energy when bearing impact load, thus showing better impact toughness in macroscopic performance [34]. It was worth noting that a large number of homogeneous isoaxial dimples were formed at the fracture of the impact sample after CT, but no second-phase particle precipitates at the dimples (Figure 3a). Compared with the microstructure of the impact sample after CT, a large number of homogeneous but smaller size of the dimples of the sample after CHT were formed, and the second phase particles with smaller size were precipitated in the center of the dimples (Figure 3b). Energy spectrum test was conducted on the precipitates of spherical second-phase particles, and the main components were shown in Figure 4.

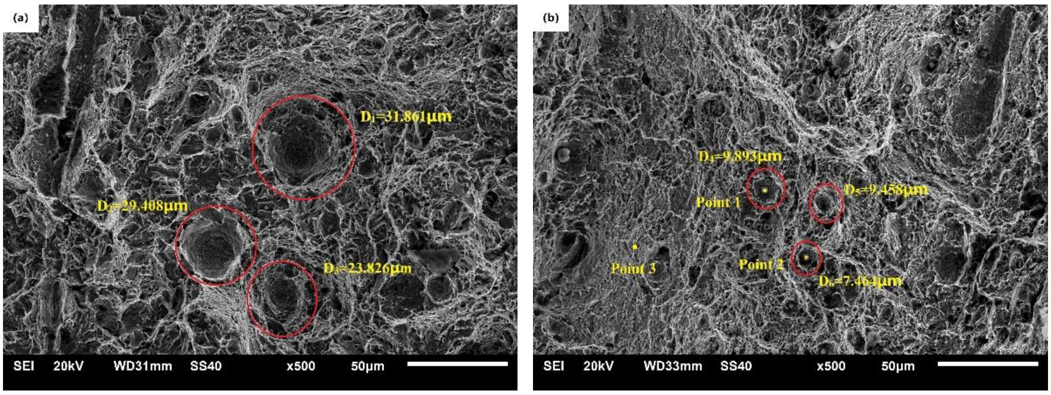

Figure 3. Impact fracture of samples after two kinds of heat treatment((a)CT;(b) CHT)

These spherical inclusions are rich in Al, Ca, S and Mg elements, which may be the Al_2O_3-MgO-CaO composite oxides. It was the presence of such inclusions that caused holes to occur in the material. When the material was subjected to a large impact load, stress concentration would occur near the inclusions, resulting in fracture of the sample [35].

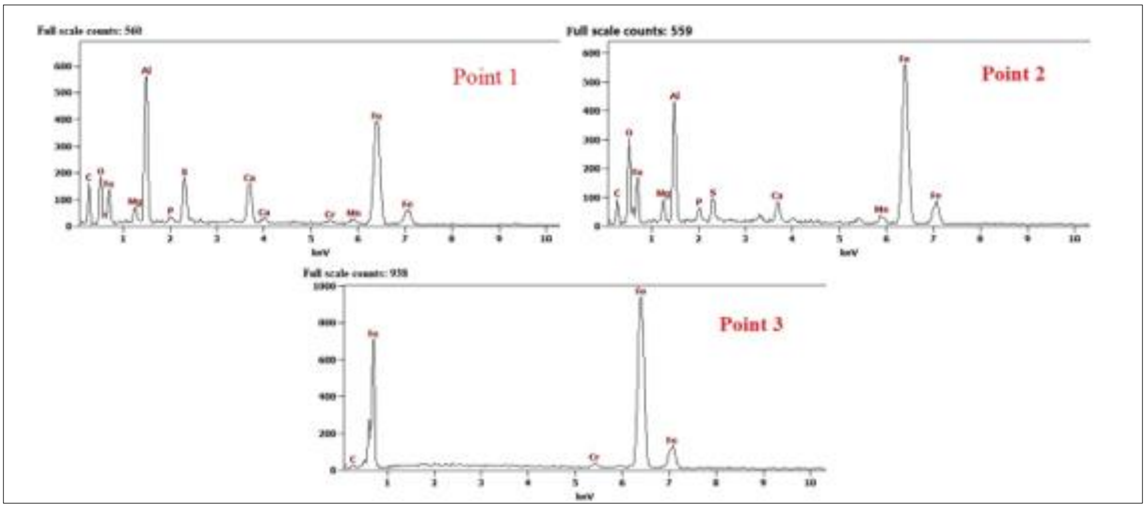

Figure 4. EDS analysis for fracture surface of Figure 5(b)

3.3. Tensile test

The tensile strength and yield limit of 51CrV4 plate spring steel after CT and CHT can be obtained by tensile test, so as to draw the σ - ε curve (Figure 5). As can be intuitively seen from Figure 5, the yield limit of cryogenic treated samples is about 1.22GPa, which is nearly 15.10% higher than that of CHT samples. It was necessary to point out how to obtain the yield limit of the material according to the stress-strain curve. Extended the nearly linear part of the stress-strain curve at the beginning and tried to intersect the X-axis, or figured out the equation of the line for the line part, and then shifted this line to the right by 0.2%. At this point, the y-coordinate of the intersection of the new line and the stress-strain curve was the yield limit [36].

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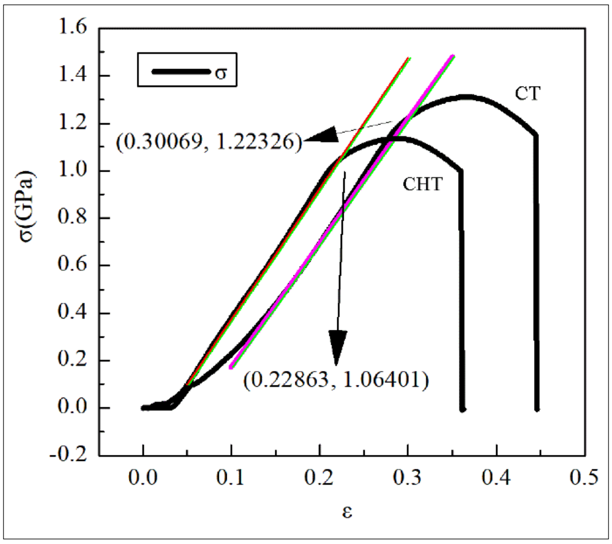

Figure 5. Stress-strain diagram after CT and CHT

The tensile strength of the specimen in this tensile test can be obtained from Figure 6. The tensile strength of CT sample is about 1.31GPa, which is 15.93% higher than that of CHT sample. Thus, the CT effectively increased the tensile strength and yield strength of 51CrV4 plate spring steel.

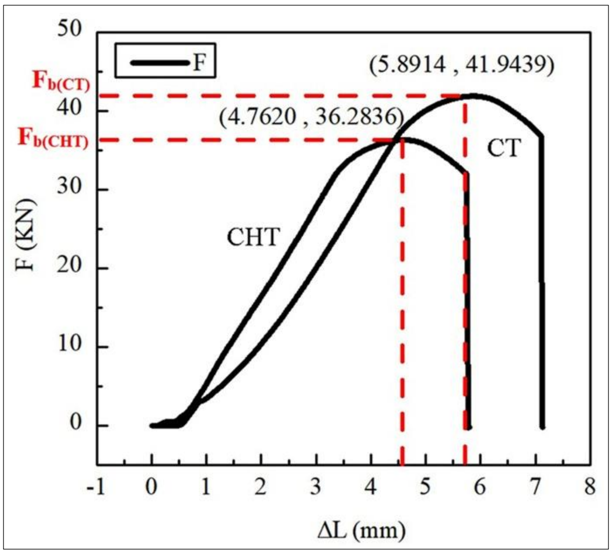

Figure 6. Force-the length of the increment diagram after CT and CHT

3.4. Axial tension-tension fatigue test

The experimental results showed that the fatigue life of 51CrV4 plate spring steel samples under axial alternating load can be improved by CT. The fatigue test data are shown in Table 6. At the fourth stage stress level, the amplitude of axial alternating stress is 217.5 MPa. The fatigue cycles of all specimens after two kinds of heat treatment reached 10^7 cycles, and the specimens were not broken. The S-N curve of this fatigue test was drawn according to the experimental data, as shown in Figure 7. As the number of fatigue cycles under the fourth level of stress is far greater than the number of fatigue cycles measured under the first three stress levels, reaching the specified fatigue limit, considering the beauty of the figure, the fatigue life under the fourth level stress level is not shown in the figure.

The fatigue fracture of general steel consists of three parts [37]: the area of fatigue source, the area of crack propagation and the area of failure. An obvious radial pattern can be observed from the crack

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source to the area of crack stable propagation, which are the traces left by material tearing during the crack growth process (Figure 9c). Then the area of failure is the roughest area of the whole fracture and its area is affected by the external load (Figure 9a,b). The larger the load, the larger the area of the fracture. There were studies showed that the larger the loading stress, the smaller the area of fatigue source [38-39], and this experiment reached a similar conclusion (Figure 8).

Table 6. Data for axial tension-tension fatigue test

Mean number of cycles/ cycles	number of cycles/ cycles	Stress amplitude/ MPa	The sample number (CT)	The sample number (CHT)	Stress amplitude/ MPa	number of cycles/ cycles	Mean number of cycles/ cycles
149,015.3	78,177	299.38	191122-1	19112201	299.38	110,782	101,822.0
	154,644		191122-2	19112202		61,772	
	214,225		191122-3	19112203		132,912	
236,863.7	237,161	270.00	191122-4	19112204	270.00	221,765	219,624.3
	233,461		191122-5	19112205		227,421	
	239,969		191122-6	19112206		209,687	
873,278.0	875,496	243.75	191122-7	19112207	243.75	372,899	610,859.0
	1,001,352（Not broken）		191122-8	19112208		717,357	
	742,986		191122-9	19112209		742,321	
10,000,000+	10,015,173（Not broken）	217.50	191122-10	19112210	217.50	10,000,874 (Not broken）	10,000,000+
	10,003,638（Not broken）		191122-11	19112211		10,000,518（Not broken）	
	10,001,913（Not broken）		191122-12	19112212		10,000,816（Not broken）	

The fracture morphology of the fatigue samples after the treated in CT and CHT was observed. The fatigue fracture morphology of the samples after CHT was discussed, firstly. Obviously, the morphology and size of spherical inclusions from different zones can be observed at the fracture surface by magnifying 1000 times under SEM from Figura 10, and the position and size of inclusion were random. Notably, the inclusions were where the crack propagation occurs.

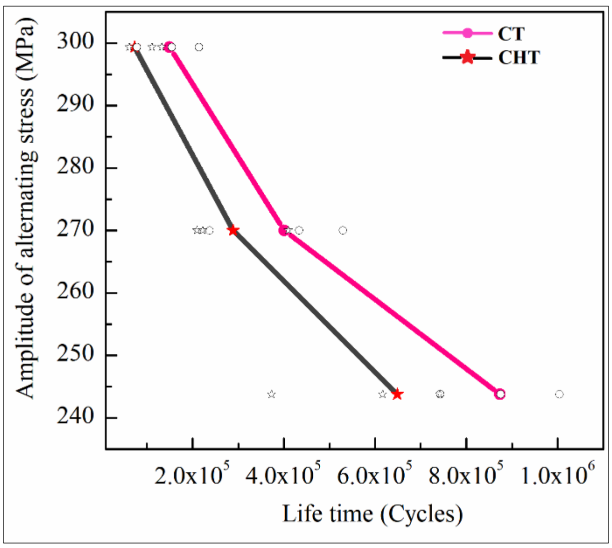

Figure 7. Results of fatigue tests after heat-treated in CT and CHT

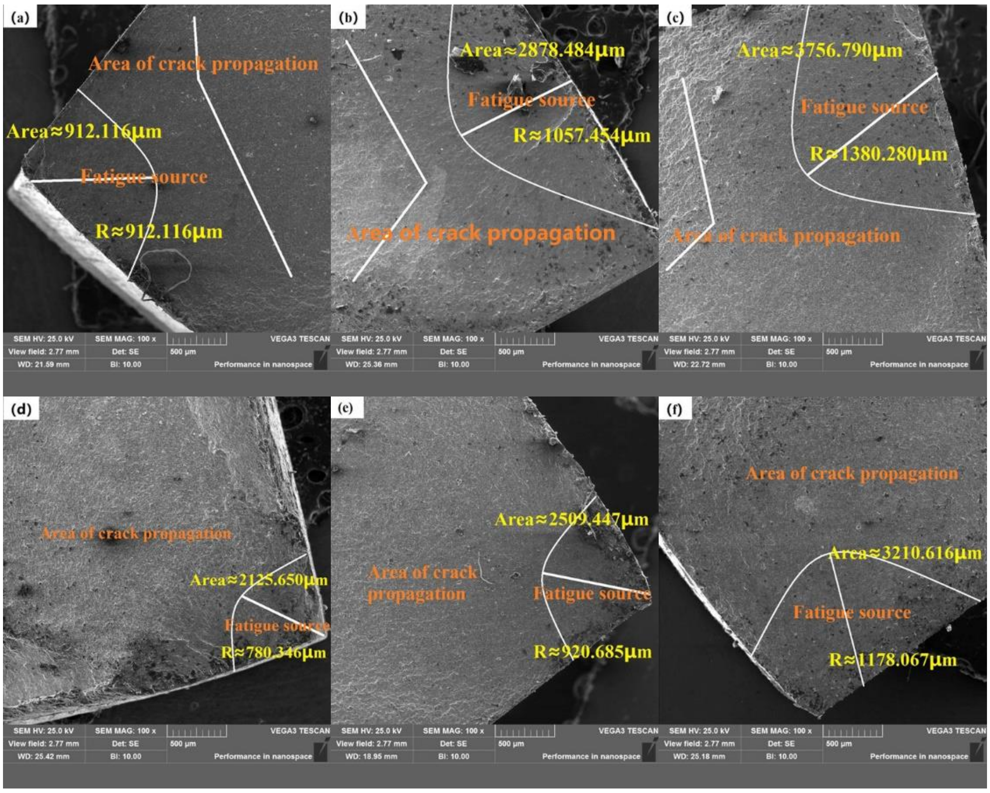

Figure 8. SEM images of fatigue crack source under different loads after CHT and CT (299.38MPa:(a), (d); 270.00MPa:(b), (e); 243.75MPa:(c), (f); CHT:(a)-(c); CT:(d)-(f))

EDS (Energy Dispersive Spectrometer) analysis indicated the presence of manganese sulfide inclusions according to Figure 10 and the graphical data presented in Figure 11. The results of EDS analysis showed that these inclusions mainly contain aluminum, calcium, magnesium, sulfur, silicon and manganese and so on, which were complex alumina or sulfide inclusions. May be Al_2O_3-MgO-CaO-($SiO2$), Al_2O_3-SiO_2-CaO and FeS or MnS. Because the thermal expansion coefficient of this kind of inclusions was different from that of the matrix, the plasticity was relatively low, and the deformation of the matrix was inconsistent with that of the matrix under the action of cyclic load. Finally, the radial tensile stress was generated in the frontal matrix near the inclusion, which made the fatigue crack generate and expanded in the matrix close to the inclusion [40].

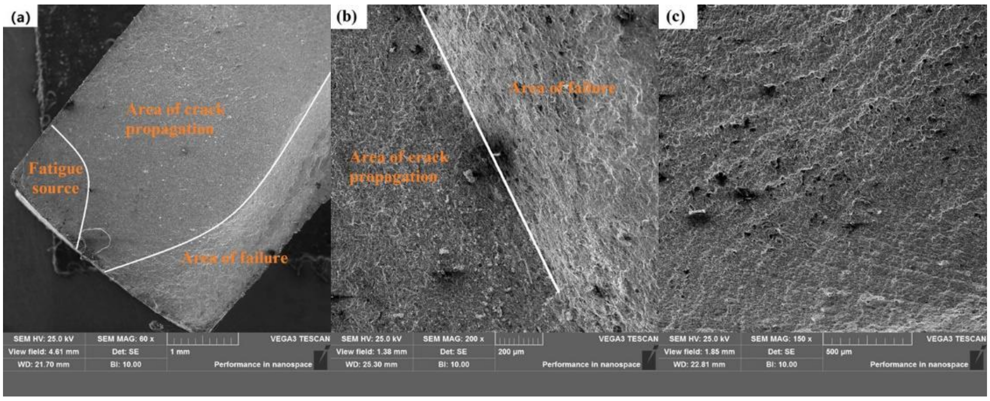

Figure 9. The morphology of three regions of fatigue fracture

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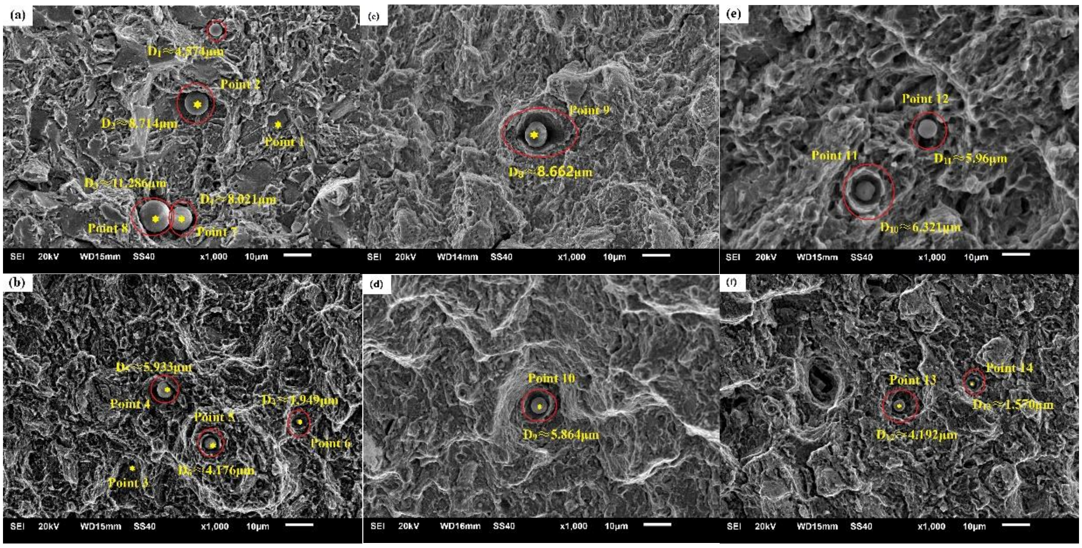
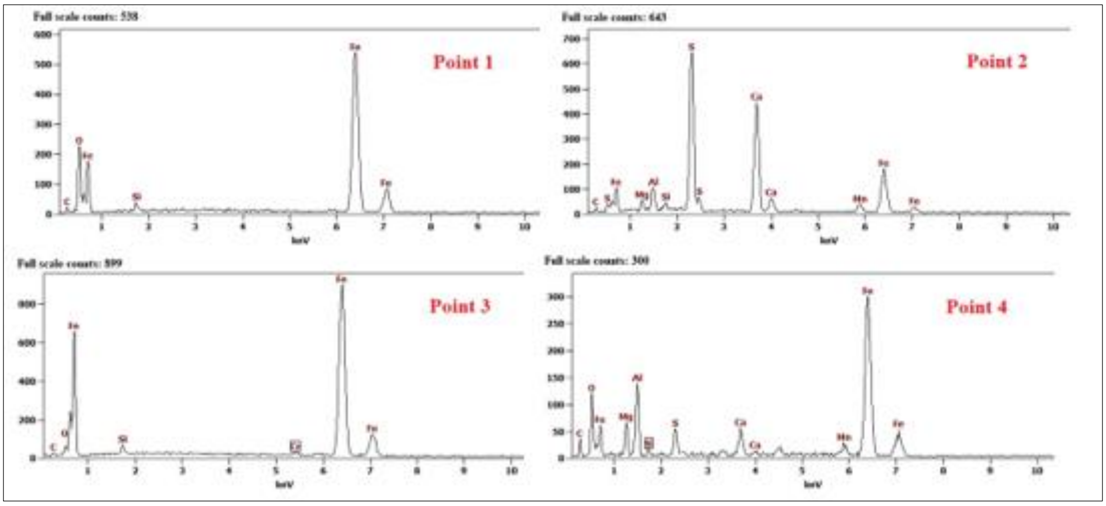


Figure 10. Morphology and sizes of inclusions in different zones.
CHT: (a), (c), e); CT: (b), (d), (f)

Inclusion is a typical microstructure defect in the high-strength steel, and it can decrease the fatigue property of spring steel. As shown in Figure 10a and b, the large-size inclusion can increase the fatigue risk, this is because the inclusion can cause serious stress concentration and make the fatigue life decrease. However, the distribution of inclusions is usually random, and the position of the bigger inclusion is difficult to predict. Thus, it is necessary to control the size and quantity of inclusions as a whole. By controlling the quantity and size of inclusions in the surface layer, the fatigue risk factor can be further decreased and fatigue life can be increased. However, it is very difficult to control the number and size of inclusions.

This experiment presented a phenomenon (Figure 10 and Figure 11): by comparing the composition of inclusions at the fracture of samples after CT and CHT, it was found that after CT, the sulfur content at the fracture of samples was lower than calcium content, while after CHT, the sulfur content at the fracture of samples was higher than calcium content. The results show that the introduction of CT before tempering can cause the aggregation of *Ca* element in the material, and trace *Ca* has the effect of deoxidizing and desulphurizing and improving the morphology of sulfide [41]. It is well known



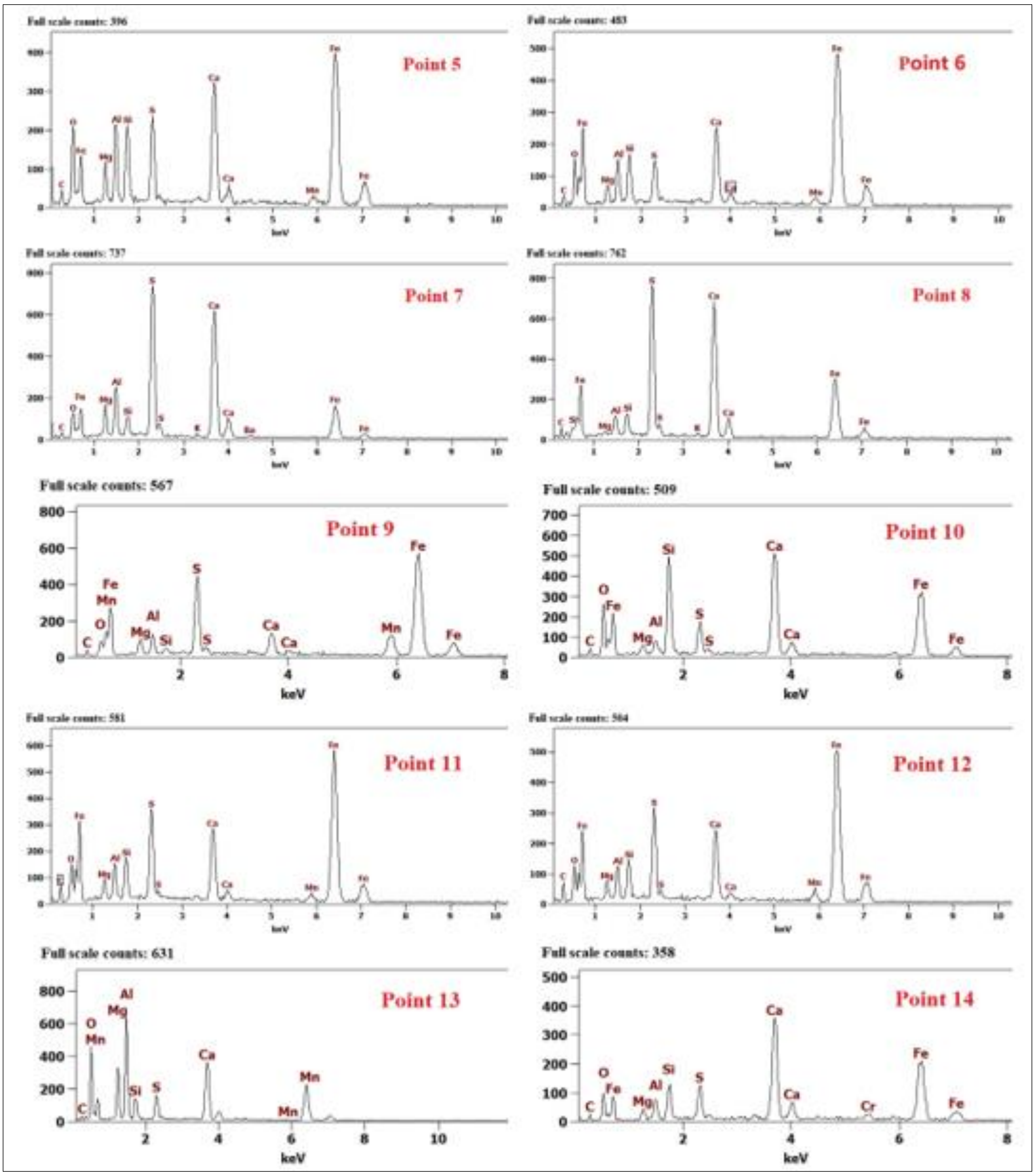


Figure 11. EDS analysis for fracture surface of 51CrV4.
(CHT: Point 1,2,7,8,9,11,12; CT: Point 3,4,5,6,10,13,14)

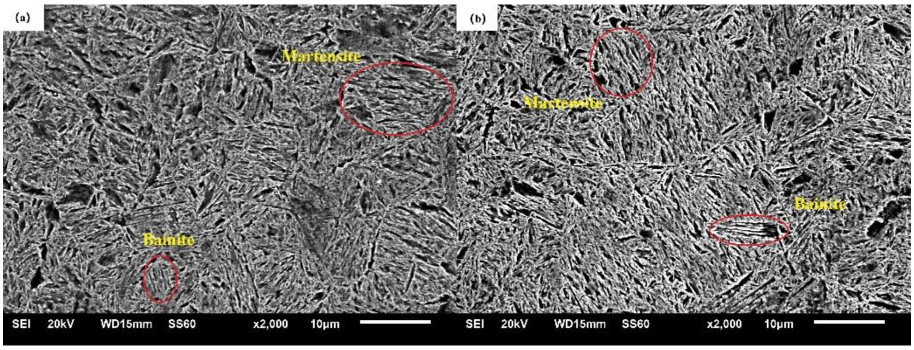


Figure 12. Microstructure of 51CrV4 after different heat treatment processes. (a) CHT process; (b) CT process

Figure 12 shows the SEM microstructure of 50CrVA leaf spring steel specimen taken near the fracture surface. The results show that the banded bainite and lath martensite can be observed in the microstructure of fatigue specimens whether or not CT. As a whole, bainite and martensite are evenly distributed. However, it is obvious that in the visible range, the microstructure in Figure 12b is denser, the microstructure gap is finer, and the arrangement of bainite and martensite is more orderly. Obviously, the microstructure of 51CrV4 leaf spring steel has been refined by CT. The dense structure distribution has a positive effect on the fatigue life of the material, which is also an important reason for the CT to improve the fatigue life of 51CrV4 leaf spring steel [14].

4.Conclusions

The effect of the introduction of CT technology on the fatigue life of 51CrV4 plate spring steel in CHT process was studied in this paper. In order to determine the feasibility of the process, the Rockwell hardness and impact toughness were tested firstly, and the fatigue test was carried out. The following conclusions can be drawn:

-in this experimental scheme, the effect of CT on the hardness and impact toughness of 51CrV4 leaf spring steel is not significant;

-the CT played a positive role in the fatigue life of 51CrV4. The *S-N* curve of 51CrV4 plate spring steel after CT is significantly higher than that after CHT. The CT greatly improved the fatigue life of 51CrV4 plate spring steel under axial alternating load;

-the fracture surface of the specimen was precipitated with varying size and quantity of inclusions under axial alternating load, and the main components of inclusions were *S*, *Ca* and *Mn*. The introduction of CT before tempering makes the *Ca* element in the material aggregate, and the micro amount of *Ca* has the function of deoxidizing and desulphurizing and improving the morphology of sulfide, thus enhancing the fatigue life of the material;

-in this test, after CT, 51CrV4 leaf spring steel reaches permanent fatigue when the amplitude of axial alternating stress is equal to 217.50 MPa and the average stress is equal to 119.625 Mpa;

-the results show that the microstructure of 51CrV4 leaf spring material is mainly acicular bainite and thin strip martensite before tempering. Compared with CHT, CT makes the microstructure of the material more compact.

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