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The effect of tempering temperature on the microstructure and mechanical properties of ultrahigh strength 23CoNi steel plate

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Abstract: The effect of tempering temperature on microstructure and mechanical properties of 23CoNi steel plate has been studied. Tempering in the temperature range of 100-460℃, the ultimate tensile strength σ_b tends to change along a curve which has the shape of the letter "n", the peak of the σ_b occurs in the range of 460-475℃, beyond the peak, σ_b deceasing as the temperature increasing. Tempering in the temperature range of 100-200℃, the impact property α_{ku} increases as the temperature goes up. After 200℃, α_{ku} goes down as the temperature increases, when the tempering temperature goes higher than 460℃, α_{ku} goes up again tempering at higher than 485℃, the acicular alloying carbides M_2C precipitate from matrix. The orientation relationship between M_2C and matrix coincides with follows: $(011)_\alpha // (001)_{M_2C}$, $[100]_\alpha // [100]_{M_2C}$. We found out a new order phase B2 which dispersing in the matrix and the size is very small. This paper discusses the reason of the formation of this new order phase and the sequence of effects to mechanical properties. With the increasing of the tempering temperature, both M_2C and new order phase growing up. As the tempering temperature goes beyond 470℃, the amount of retained austenite tends to go up, higher than 510℃, the growing up of reverted austenite is accelerative. It is identified that retained austenite is coming from the reverse transformation. Tempered at higher than 550℃, we can easily find the reversion austenite which contains much more Nickel than matrix does and emerged in the bulk form under TEM.

Key words: 23CoNi steel; M_2C; order-phase; reversion austenite; tempering temperature

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1 Introduction

23CoNi (0.23C-14Co-2Ni-3Cr-1Mo) steel is an ultrahigh strength steel with high alloy. It exhibits high strength, good toughness and excellent corrosion resistance. The tensile strength of 23CoNi is above 1930 MPa and fracture toughness tends to higher than 110 MPa $\sqrt{m}$. It is not only used to make critical aircraft parts, such as landing gear, joints etc., but also is used for bulletproof material. It is a kind of secondary hardening alloy steel, strengthened by the precipitation of a fine dispersion of M_2C carbides. We used plate steel to study the effect of tempering on the microstructure and mechanical properties. Micro-

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structure was studied by transmission electron microscopy (TEM). Although we studied M_2C carbides elsewhere and published our achievements in prior paper[1-3], this time we examined this carbide again. Besides the M_2C, we found out a new order phase B2, and a lot of reverted austenite formed after middle or higher temperature tempered.

2 Experimental procedures

23CoNi steel was VIM/VAR melted and hot rolled to 6mm plate. The composition of the steel is shown in Table 1.

Table 1 Composition of steel

Element	C	Si	Mn	Al	Ti	S	P	Co	Ni	Cr	Mo	Fe
Mass fraction/%	0.24	<0.1	<0.1	0.010	<0.01	0.001	0.0054	13.12	11.26	2.90	1.17	Bal.

The heat treatment procedures were as follows: homogenized at 900℃, air cool, tempered at 680℃, air cool, austenitized at 885℃ in vacuum furnace, quenched under argon to room temperature, and immediately transferred to −70℃ for 1h, tempered at various temperatures holding for 6h at each temperature. The tensile specimen is plate. The impact specimen is 5 mm×5 mm and U-notch. The volume fraction of retained austenite was determined by X-ray diffraction. Microstructures were examined by Transmission Electron Microscopy (TEM).

3 Results

3.1 The effect of tempering on mechanical properties

Specimens were austenitized at 885℃, quenched in argon to room temperature, and immediately transferred to −70℃ for 1 h, and then tempered at different temperatures for 6 h. A range of temperatures resulted in substantial changes in mechanical properties is shown in Fig.1. From this figure, tempering under 300℃, the curve of $\sigma_{0.2}$ declines while temperature goes up; while tempering from 300℃ to 490℃, $\sigma_{0.2}$ increases , when tempering at 490℃ the curve reaches the peak. Beyound 490℃, $\sigma_{0.2}$ decreases again and gets the lowest value: 1304 MPa at 600℃. As to tensile strength, σ_b, and HRC tempering below 200℃, they decline rapidly. The reason is that the strain in quenched martensite decreases when steel tempered at low temperature. The curve shows that there is no change for σ_b and HRC as tempering between 200℃ and 300℃. Tempering over 300℃, σ_b and HRC climb up and σ_b achieves peak value which is about 2126 MPa between 460℃and 475℃. Tempering after 475℃, σ_b declines slowly until 510℃. Beyond this point, σ_b drops quickly as temperature goes up and it gets only 1379 MPa at 600℃. HRC reaches peak at 475℃ and decreases as temperature goes up. Above 510℃, HRC decreases quickly to 44.2HRC at 600℃. The trend of changing of HRC is the same as σ_b. With temperature goes up, there is no notable change for δ_5. However, δ_5 increases when tempering above 510℃ and the peak value is 17.1% at 600℃. a_{ku} gradually goes up as tempering from 100℃ to 200℃ and declines over 200℃. Tempering at 460℃, a_{ku} gets the lowest value of 49.1 J/cm². a_{ku} shows increasing as tempering in the range from 460℃ to 600℃ and the peak appears at

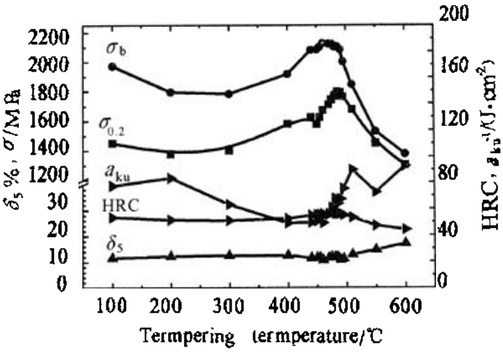

Fig.1 Variation of mechanical properties with tempering temperature for 6h

600℃.

3.2 Effect of tempering on microstructures

Due to the steel is used after tempered at higher than 482℃, the microstructures of the steel tempered at high temperatures have been intensively studied.

3.2.1 485℃

Tempering at 485℃, the fine, dispersed, needle-shaped M_2C precipitates are obtained and they distribute along the dislocation lines. These particles are coherency with matrix. The relationship between M_2C and matrix is as follows: $(011)_\alpha // (001)_{M_2C}$, $[100]_\alpha // [100]_{M_2C}$. The electron diffraction patterns from martensite are a little bit distortion, showing the strain between M_2C carbide and matrix is strong. Fig. 2 shows that extra spots of superlattice appear on the reciprocal plane$(100)_\alpha$. We believe that a kind of order phase B2 caused such extra spots of superlattice and the lattice parameter of B2 is very close to that of matrix. The dark-field images show that the phase B2 possesses fine, dispersed, and spot shape, the diameter is about 2—3 nm and coherency with matrix. The relationship between order phase B2 and matrix is $(100)_M // (100)_{B2}$, $<100>_M // <100>_{B2}$. Fig. 2 shows the image of M_2C carbide and order phase B2. After tempering at 485℃, the reverted austenite formed and gradually increasing.

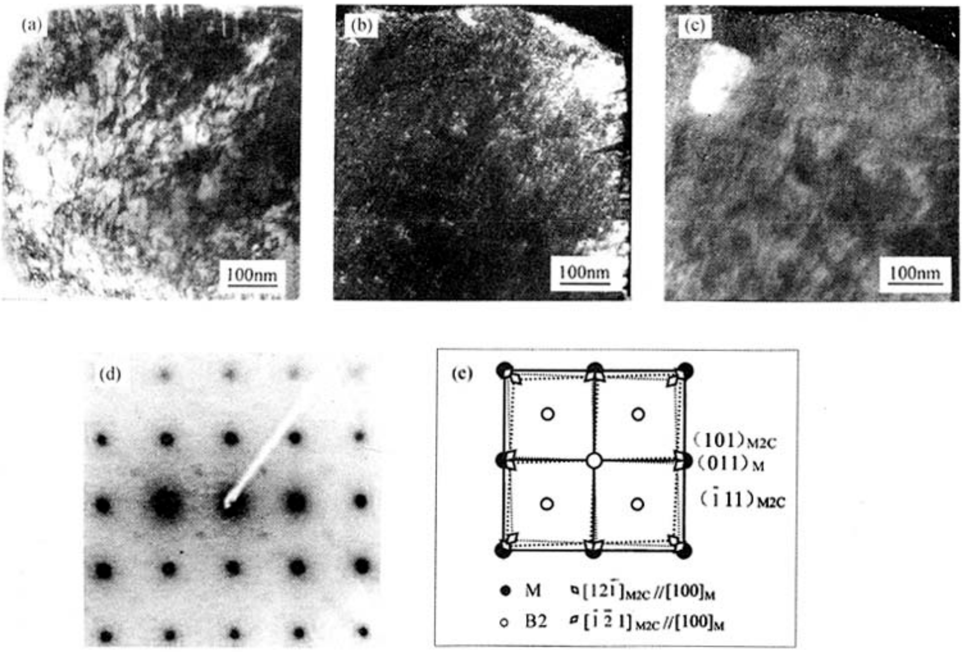

Fig. 2 Images of M_2C carbide and order phase B2 after tempering at 485℃

(a)—Bright-field image; (b)—The $(2\bar{1}0)$ dark-field of M_2C carbide; (c)—The (100) dark-field of order phase; (d)—Diffraction patterns; (e)—Indexing of the pattern

3.2.2 550℃

Tempering at 550℃, results in growing of M_2C carbides. The size of M_2C carbide is about 30nm in length and 4-5 nm in width (Fig. 3). After tempered at this temperature, the diffraction spots of (100) from the order phase are clearer than that of tempered at 485℃. The dark—field images of the order phase are distinctly obtained and shown in Fig.4. There are two kinds of shape of the order phase B2. One shows rod-shape, the longitudinal direction lying along the $[100]_a$ or $[010]_a$, the length is about 10nm and the width is about 2nm. The other shape is like square which consists of 4 small parallel rods, the side of the square is about 10 nm. In such condition the reverted austenite is easily observed, some of such austenite is about 440nm in length and 24-40 nm in width, and the others are 80 nm×80 nm square (see Fig.5). The Ni content in austenite is about 31.67%, much higher than that in martensite (10.28%). In contrast, 11.22% Co is in martensite and 7.31% is in austenite.

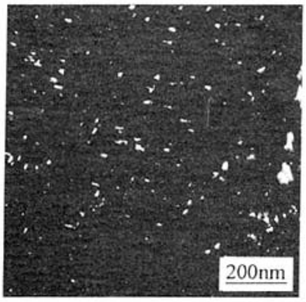

Fig.3 The $(2\bar{1}0)$ dark-field of M_2C carbide

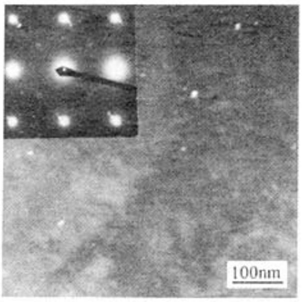

Fig.4 The (100) dark-field of order phase

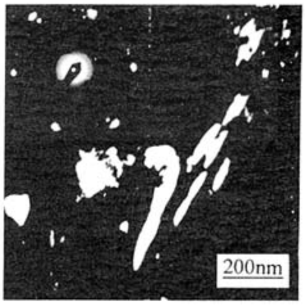

Fig.5 The (200) dark-field of austenite

3.2.3 600℃

Tempered at 600℃, M_2C carbide shows small rod-shaped and the length is of 30-50 nm, the diameter is of 5-10 nm. Fig.6 shows the moiré fringes that parallel to long axis of carbide with spacing of 1-2 nm. Fig.7 shows the order phase with 10nm in size. Fig.8 shows the austenite in block-shaped. Now in austenite Ni content is only about 25.54% which is lower than that of tempering at 550℃. Co is 10.54% in martensite, closes to that of tempered at 550℃. The decreasing volume percentage of Ni in reverted austenite may be attributed to the increased volume of this austenite. Although there are some errors in the analysis of EDS, we would conclude that the amount of Ni and Co is different in reverted austenite and martensite

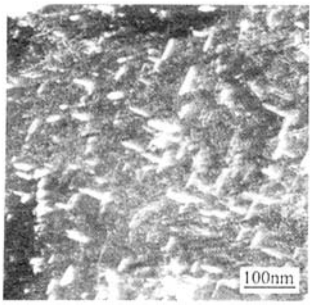

Fig.6 The (101) dark-field of M_2C carbide

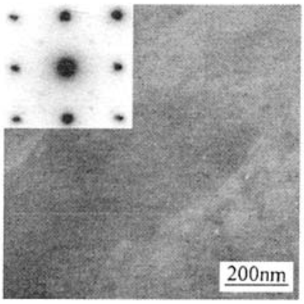

Fig.7 The (100) dark-field of order phase

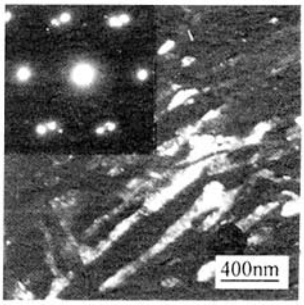

Fig.8 The (200) dark-field of austenite

3.3 The effect of temper temperature on the amount of austenite

Fig.9 shows the change of the volume percentage of austenite after tempered at various temperatures. Due to the difficulty to separate the retained austenite from the reverted austenite by XRD, Fig.9 gives out the total content of austenite. Tempering over 100℃, the volume fraction of austenite decreases slightly. There is a little change for the volume fraction of austenite as tempered from 440℃ to 482℃. The volume fraction shows an increase between 482℃ and 510℃, and increasing rapidly as tempering beyond 510℃. The main reason is that the reverted austenite increasing.

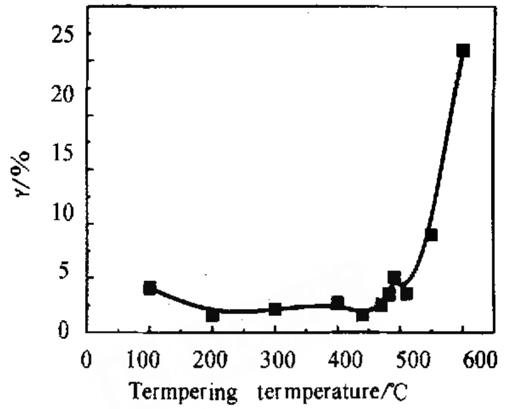

Fig. 9 Variation of volume fraction of austenite with tempering temperature for 6 h

4 Discussion

4.1 Order phase

Some evidence[4] and the cards of X-ray show that there are three kinds of order phases B2: FeCo, NiAl, and FeAl. Their lattice parameters are close to that of matrix in this alloy, $d_{FeCo}=0.28504$ nm, $d_{FeAl}=0.289$ nm, $d_{NiAl}=0.288$ nm. From the phase diagrams of Fe—Co, Ni—Al, and Fe—Al[5], all these 3 phases are existence from 480℃ to 600℃. With Cr and Mo gathering in matrix[3], M_2C phase is easy to form and its content is $(Cr_{0.35}Fe_{0.52}Mo_{0.13})_2C$. The formation of M_2C phase makes the nickel and cobalt content increase in matrix. As tempered at higher enough temperatures, Co, Ni and Al would get enough energy for moving. When the atomic number of Fe—Co, Ni—Al and Fe—Al gathers and reaches about 40%—60% in some regions, these elements may be arranged in order. i. e. FeCo, NiAl and FeAl order phases are formed. It is not sure that the extra spots of superlattice on the reciprocal plane$(100)_\alpha^*$ are caused by one, two or three of these order phases, because these order phases are too small to be judged by EDS. Very few reports about FeCo and FeAl phases exist but as strengthening phase NiAl does exist in some steel. PH13—8Mo steel is strengthened by the precipitation of NiAl particles[6]. Al was added in some secondary hardening steel to form NiAl phase[7], so M_2C and NiAl phase co—existed in some steels. Hence authors tend to consider that the order phase is NiAl particle in this studied steel. In PH13—8Mo steel[6], the ageing—peak and temper brittleness occurred simultaneously at about 500℃. If B2—type NiAl order phase exists in 23CoNi steel, it might be harmful to the toughness of this steel to be tempered at 485℃.

4.2 The formation of reverted austenite

The analytical result of reverted austenite shows that there is higher percentage of Ni in austenite. It proves that the formation of reverted austenite is the result of the gathering of Ni from matrix[3]. At the Fe—rich dual—phase region in Fe—Ni phase diagram, the reaction is:

$$\alpha_2 \longrightarrow \alpha' + A_{RR}$$

where α_2 is martensite, α' is low Ni content bcc phase, ARR is high Ni content fcc phase (reverted austenite)[5]. During tempering, the formation of M_2C carbide, $(Cr_{0.35}Fe_{0.52}Mo_{0.13})_2C$[3], makes the nickel content increasing and Cr, Mo content decline in matrix. The fluctuation of the metal content ensures the nucleation of reverted austenite. From the energy point of view, during tempered at middle or high temperatures Ni atom gets enough energy for long or short range migrating and clustering. It's easier for reverted austenite to nucleate at interfaces, the reason is that the formation of the new boundaries causes the reduction of the original boundaries, and the strain energy decreasing simultaneously. Thus this nucleation leads ΔG reducing as the drop of ΔG_S and ΔG_E. ($\Delta G=-\Delta G_v+\Delta G_S+\Delta G_E$, where ΔG is the total changing of free energy in the system, ΔG_v is the energy of phases changing, ΔG_S is the surface energy, ΔG_E is the increment of strain energy). So it is easier to get the condition of $\Delta G < 0$ in the whole system.

5 Conclusions

(1) When tempering at 490℃, the $\sigma_{0.2}$ of 23CoNi steel reaches the peak. The peak of the σ_b appears in the range of 460－475℃. Tempering at 460℃, a_{ku} possesses the lowest value.

(2) When tempering at 485℃, we not only found out that M_2C precipitated from the matrix, but also found out the order phase B2. As the tempering temperature went up, both M_2C and order phase grew up.

(3) Tempering at higher than 485℃, the volume content of austenite increases as the temperature goes up and most of them come from the reverted austenite. Tempering over 550℃, the square－shaped reverted austenite appears often. The Ni content in austenite is much higher than that in martensite, but Co content is lower than that in martensite.

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