Effect of Tempering Temperature on the Microstructure and Mechanical Properties of Granular Bainite in 2.25Cr-1Mo-0.25V Steel*

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Abstract

The variation of normalized granular bainite microstructure and mechanical properties with tempering temperature in 2.25Cr-1Mo-0.25V steel for hydrogenation reactors was investigated using nanoindentation, OM, SEM, TEM, XRD, EPMA and other techniques. The results indicate that after normalizing, 2.25Cr-1Mo-0.25V steel develops a granular bainite microstructure consisting of bainitic ferrite, martensite, and retained austenite islands (M-A islands). Nanoindentation measurements reveal that due to carbon enrichment in the M-A islands, their hardness is significantly higher than that of bainitic ferrite. During tempering, the combined effects of M-A island decomposition and softening of bainitic ferrite cause the impact energy of 2.25Cr-1Mo-0.25V steel at -18 ℃ to first increase and then decrease with increasing tempering temperature. In addition to the softening effect from recovery and recrystallization of the ferrite matrix, the degree of tempering transformation of the hard-phase M-A islands in the granular bainite microstructure, as well as the morphology, size, and distribution of precipitated carbides, are key factors influencing the impact toughness of 2.25Cr-1Mo-0.25V steel.

Full Text

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Abstract Using a nanoindenter, OM, SEM, TEM, XRD, EPMA, and other equipment, the changes with tempering temperature in the normalized granular bainite microstructure and mechanical properties of 2.25Cr-1Mo-0.25V steel for hydrogenation reactors were investigated. The results show that, after normalizing, 2.25Cr-1Mo-0.25V steel obtains a granular bainite microstructure composed of bainitic ferrite, martensite, and retained austenite islands (M-A islands). Nanoindentation measurements show that, because C is enriched in the M-A islands, their hardness is significantly higher than that of the bainitic ferrite. During tempering, the combined effects of M-A island decomposition and bainitic ferrite matrix softening cause the impact energy of 2.25Cr-1Mo-0.25V steel at −18 ℃ to first increase and then decrease with increasing tempering temperature. In addition to the recovery, recrystallization, and softening effects of the ferrite matrix, the degree of tempering transformation of the hard-phase M-A islands in the granular bainite microstructure, as well as the morphology, size, and distribution of precipitated carbides, are key factors affecting the impact toughness of 2.25Cr-1Mo-0.25V steel.

Keywords 2.25Cr-1Mo-0.25V steel; granular bainite; impact toughness; martensite-austenite island

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EFFECTS OF TEMPERING TEMPERATURE ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF GRANULAR BAINITE IN 2.25Cr-1Mo-0.25V STEEL

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ABSTRACT 2.25Cr-1Mo-0.25V steel is the most popular material used for pressure-vessel applied at elevated-temperature in hydrogen environment. For higher process efficiencies in future coal-conversion plants, chemical processing plants, and petrochemical-refining plants, much thicker cross-section component are necessary for constructing much larger pressure-vessel for these plants. Because of the thick cross-section, the cooling rate in the central region of the component is insufficient to obtain low bainite during quenching treatment, and a large amount of granular bainite appears in the central region. Previous studies have shown that good impact toughness can be achieved by appropriate tempering for 2.25Cr-1Mo-0.25V steel with low bainite microstructure. However, the impact toughness of 2.25Cr-1Mo-0.25V steel with granular bainite after tempering always cannot satisfy the demanding requirement due to the unclear understanding of the evolution of microstructure and mechanical properties during tempering. In this work, the influence of tempering on the microstructure and mechanical properties of 2.25Cr-1Mo-0.25V steel with granular bainite microstructure was investigated by OM, XRD, SEM, TEM and EPMA. The results show that the normalized 2.25Cr-1Mo-0.25V steel with granular bainite microstructure is composed of bainite ferrite and island of martensite and austenite (M-A island). Nanoindentation test indicates that M-A island is much harder than that of metrix bainite ferrite, because of the high concentration of carbon in M-A is-

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lands. The synergistic effect of the decomposition of M-A islands and softening of bainite ferrite determined that Charpy absorbed energy at −18 ℃ increases first and then decreases with the increase of tempering temperature. The degree of decomposition of M-A islands and the morphology, size and distribution of carbides in granular bainite, coupled with the softening effect of bainite ferrite recrystallization are the key factors determining low-temperature impact toughness of 2.25Cr-1Mo-0.25V steel.

KEY WORDS 2.25Cr-1Mo-0.25V steel, granular bainite, impact toughness, M-A island

Low-carbon Cr-Mo steel has relatively high creep resistance, excellent oxidation resistance and hydrogen embrittlement resistance, good processability, and favorable economy, and has been widely used in deep-processing fields for energy sources, such as petroleum cracking and coal liquefaction. Because of tight global petroleum demand and the continuous rise in crude-oil prices, it is necessary to continuously improve the efficiency of petroleum refining. The heavy-oil or extra-heavy-oil cracking technologies that have consequently emerged require materials for hydrogenation equipment to be able to adapt to more demanding service environments. Therefore, 2.25Cr-1Mo-0.25V steel has gradually been selected to replace traditional Cr-Mo steels[1–3]. In practical engineering applications, the heat-treatment process for 2.25Cr-1Mo-0.25V steel forgings is usually quenching plus high-temperature tempering; after heat treatment, mixed microstructures such as tempered upper bainite, lower bainite, and granular bainite are obtained. In general, when the quenching rate is relatively high, a microstructure dominated by lower bainite is obtained after quenching. After appropriate tempering, 2.25Cr-1Mo-0.25V steel exhibits good strength–toughness matching[4]. As the size and power of hydrogenation units continue to increase, the wall thickness of 2.25Cr-1Mo-0.25V steel forgings increases substantially. Owing to the limited quenching-cooling capacity in actual production, when the wall thickness increases to a certain extent, the core of the forging is difficult to through-quench, and a microstructure dominated by granular bainite is often obtained. Thus, the quenched microstructure of thick-walled 2.25Cr-1Mo-0.25V steel forgings gradually changes from the surface layer to the center. Owing to the gradient variation in the as-quenched microstructure of large-section thick forgings, after the traditional tempering heat-treatment process used in actual production, problems often arise in which the impact toughness at the center of the forging is poor and unstable. Therefore, on the basis that the quenched microstructure is dominated by granular bainite, studying the effect of the tempering process on the microstructure and mechanical properties of 2.25Cr-1Mo-0.25V steel has important theoretical and practical value.

The evolution of microstructure during tempering of Cr-Mo low-alloy steels, especially the second phase and mechanical properties, has long attracted attention from researchers at home and abroad. Studies[4–7] show that tempering-

process parameters directly affect the microstructure and mechanical properties of Cr-Mo low-alloy steels. Tao Peng et al.[3] studied the transformation process of the second phase in 2.25Cr-1Mo-0.25V steel welds during high-temperature tempering. The results showed that the second phase undergoes a complex change process during tempering. Li Zhenjiang et al.[5] studied the effect of different tempering temperatures on the microstructure and impact properties of G18CrMo2-6 steel, and considered that the type, morphology, size, and distribution of the second phase in the tempered microstructure are key factors affecting its impact properties. Qu Yuebo et al.[8] studied the effect of retained austenite content in 2.25Cr-1Mo steel welds on impact properties, and found that the tempering heat-treatment process affects the retained-austenite content and thereby significantly affects impact toughness. The above studies mainly used mixed microstructures of upper bainite, lower bainite, and granular bainite as the original microstructure, whereas no reports have been found on the effect of tempering transformation on mechanical properties in Cr-Mo low-alloy steel with granular bainite as the original microstructure. At the same time, most of the above studies focused on microstructural evolution during the tempering process, while neglecting the influence of the nonuniformity of composition and microstructure in the quenched (or normalized) original microstructure on the microstructure and properties during tempering. Klueh and Nasreldin[9] studied the relationship between the microstructure and mechanical properties of 3Cr-1.5Mo steel, and found that the state of the microstructure before tempering has an important influence on microstructural evolution and mechanical properties during tempering; among these, the strength and toughness of granular bainite are more significantly affected by tempering-process parameters than those of lower bainite. Luo et al.[10] studied the influence of the evolution during tempering of martensite/austenite (M-A) islands in the normalized microstructure of low-alloy Cr-Mo steel on impact properties, and considered that the optimal impact toughness can be obtained by reasonably controlling the tempering temperature to adjust the degree of transformation of M-A islands. Zhang et al.[11] comparatively studied the influence of tempering temperature on carbide evolution when 2.25Cr-1Mo-0.25V steel was tempered at 700 ℃ with quenched and normalized states, respectively, as the original microstructures. The results showed that different original microstructures have a large influence on the evolution behavior of carbides during tempering holding. Therefore, to obtain suitable mechanical properties in 2.25Cr-1Mo-0.25V steel after tempering, it is necessary to clarify the microstructural characteristics of the quenched (or normalized) state and, on this basis, analyze the changes in mechanical properties caused by microstructural evolution during tempering.

In this work, granular bainite obtained by normalizing and air cooling 2.25Cr-1Mo-0.25V steel was used as the initial microstructure for tempering treatment. The evolution of granular bainite during tempering was investigated, and the effects of the decomposition transformation of M-A islands in granular bainite, softening of the bainite ferrite matrix, and carbide precipitation behavior at different tempering temperatures on the mechanical properties of 2.25Cr-1Mo-

0.25V steel were analyzed, providing theoretical guidance for formulating appropriate tempering heat-treatment processes for practical large low-alloy Cr-Mo steel castings and forgings.

1 Experimental Methods

The material used in the experiment was taken from a wrought thick plate for a large hydrogenation reactor in actual production. The measured chemical composition is shown in Table 1. Table 1 also lists the chemical-composition range for 2.25Cr-1Mo-0.25V steel in the ASME standard. It can be seen that the chemical composition of the material used in the experiment meets the ASME standard requirements. The wrought thick plate was cut into specimen blocks of 60 mm × 40 mm × 13 mm. First, the specimen blocks were subjected to 940 ℃

was air-cooled after normalizing and holding for 2 h, and was then subjected to tempering treatments under different conditions. The tempering temperatures were 500, 630, 680, 700, 720, and 750 °C, respectively; the holding time was 4 h, followed by air cooling after removal from the furnace.

After electrolytic polishing of the normalized specimen, nanoindentation tests were carried out using a Nanoindenter XP nanoindenter. To obtain reliable data for each phase, 120 continuous nanoindentation tests were performed on the specimen. The 120 indentation points formed a 10 × 12 rectangular array with a spacing of 10 μm, and the applied load was 3000 μN. After tempering heat treatment, metallographic specimens were prepared for microstructural analysis. After grinding and polishing, the metallographic specimens were etched either with 4% (volume fraction) nitric acid alcohol solution or by thermal tint etching technology[⁀12] (the polished specimens were pre-etched in 4% nitric acid alcohol for 15 s and then placed in a heat-treatment furnace in an air atmosphere, held at 260 °C for 2.5 h; with this technique, different phases can be observed under an optical microscope as different colors, with bainitic ferrite appearing bluish purple, martensite brown, and retained austenite beige). After metallographic etching, microstructural observations were performed using an S-3400N scanning electron microscope (SEM) and a LEXT-OLS4100 confocal laser scanning microscope (CLSM). An EPMA-1610 electron probe microanalyzer (EPMA) was used to analyze the distribution of alloying elements in the microstructure of the normalized specimen.

A D/Max-2500PC X-ray diffractometer (XRD) was used to measure the retained austenite content in specimens under different heat-treatment conditions. The retained austenite (200) peak and martensite (200) peak were selected to calculate and determine their relative contents. Transmission electron microscopy specimens were prepared using a Tenupol-5 twin-jet electrolytic thinning apparatus. The preparation procedure was as follows: the thin-foil specimen was first thinned to about 50 μm, and discs with a diameter of 3 mm were punched

from the specimen. The discs were then thinned into thin-foil specimens using a twin-jet thinning apparatus. The twin-jet electrolyte was an anhydrous ethanol + 10% (volume fraction) perchloric acid solution; the temperature was −25 to −20 °C, and the current was 30–50 mA. A Tecmai G^2 20 transmission electron microscope (TEM) was used to observe the morphology and distribution of precipitated phases in the specimens; at the same time, the energy-dispersive spectroscopy (EDS) system attached to the TEM was used to analyze the chemical compositions of carbides. A Linseis rapid quenching dilatometer was used to measure the phase transformation points of the material.

The mechanical properties of 2.25Cr-1Mo-0.25V steel under different heat-treatment conditions were tested. Tensile tests were conducted in accordance with GB/T228.1-2010, using standard tensile specimens with a diameter of 5 mm; the test equipment was an AG-100KNG tensile testing machine. Impact tests were conducted in accordance with GB/T2651-2008, using standard V-notch impact specimens of 10 mm × 10 mm × 55 mm. The test temperature was −18 °C, and the test equipment was a BKP450. Tensile properties were tested twice and the average value was taken; impact toughness was tested four times. After the impact fracture surfaces were cleaned, an S-3400 SEM was used to observe the fracture surfaces. According to the metallographic technique for fracture cross sections, CLSM was used to observe the relationship between crack propagation and microstructure.

2 Experimental Results and Analysis

2.1 Microstructural characteristics of granular bainite

Measurement of the equilibrium phase transformation points of 2.25Cr-1Mo-0.25V steel by the thermal expansion method showed that the starting temperature of austenitization was 776 °C and the finishing temperature was 887 °C. Therefore, the austenitizing temperature in this experiment was selected as 940 °C. Figure 1 shows 2.25Cr-1Mo-0.25V steel after holding at 940 °C.

Table 1 Chemical compositions of the steel 2.25Cr-1Mo-0.25V in ASME standard and this work

(mass fraction / %)

Material	C	Cr	Mo	V	Ni	Mn	Si	P	S	Fe
Standard	≤ 0.17	2.0–2.5	0.9–1.1	0.25–0.35	≤ 0.25	0.3–0.6	≤ 0.10	≤ 0.015	≤ 0.010	Bal.
Experimental	0.14	2.46	1.00	0.28	0.15	0.59	0.05	0.006	0.002	Bal.

Fig. 1 OM (a) and SEM (b) images of normalized 2.25Cr-1Mo-0.25V steel (M-A—martensite-austenite)

OM (a) and SEM (b) images of normalized 2.25Cr-1Mo-0.25V steel. Labels in the images include M-A island, stringy M-A island, blocky M-A island, and 10 μm scale bars.

Figure 1: OM (a) and SEM (b) images of normalized 2.25Cr-1Mo-0.25V steel. Labels in the images include M-A island, stringy M-A island, blocky M-A island, and 10 μm scale bars.

After holding for 2 h, the OM and SEM images of the microstructure air-cooled to room temperature are shown. Because the steel contains Mn, Cr, Mo and other elements that enhance hardenability, a granular bainite microstructure is obtained after air cooling, with no proeutectoid ferrite or pearlite appearing. Granular bainite is composed of a ferrite matrix and island-like constituents. Electron diffraction analysis indicates that these island-like constituents are mostly a mixture of martensite/austenite, with some single martensite or austenite also present; they are therefore referred to as M-A islands[^13-^16]. When the island constituent is a mixture, the austenite is generally distributed at the edges of the islands, as indicated by the arrow in Fig. 1a. This is attributed to the diffusion of C during continuous cooling transformation: the C concentration gradient can stabilize the retained austenite located at the martensite boundaries. In addition, the volume constraint also plays an important role in stabilizing the retained austenite at martensite boundaries[^13]. Using a TAS-type image analyzer, the volume fraction of M-A islands in the as-normalized condition was measured to be 22%. XRD measurements showed that the retained austenite content was 5.3% (volume fraction). According to the morphology and distribution of M-A in the granular bainite microstructure obtained by air cooling, the M-A islands in granular bainite can be divided into two types: one is large blocky M-A islands, mainly distributed along grain boundaries; the other is elongated M-A islands, distributed within the ferrite matrix or at lath boundaries. References [13] and [14] considered that blocky mixed M-A islands and some single martensite islands are distributed on the original austenite grain boundaries, whereas elongated M-A islands distributed between bainitic laths are mostly retained austenite. The cause of this difference is mainly the difference in C diffusion during the phase transformation process and the volume constraint effect.

Most scholars[^5,^10,^13,^14] have inferred, based on the formation mechanism of M-A islands, that M-A islands are hard and brittle constituents, but quantitative data have not been provided. In this work, a nanoindenter was used to measure the nanohardness of the bainitic ferrite and large blocky M-A islands in two types of microstructures. Figure 2 shows the nanoindentation load–displacement curves. The curves indicate that the hardness of the ferrite matrix is 3.8 GPa, while that of the large blocky M-A islands is 5.8 GPa; the hardness of the M-A islands is significantly higher than that of the matrix. EPMA was used to characterize the distribution of alloying elements in the granular bainite microstructure. The results show that substitutional alloying elements such as Cr

Fig. 2

Figure 2: Fig. 2

Fig. 3

Figure 3: Fig. 3

and Mo exhibit no obvious distribution difference between the ferrite matrix and the M-A islands, whereas the C concentration in the M-A islands is markedly higher than that in the matrix (Fig. 3). Therefore, the martensite in the M-A islands often appears as twinned martensite, as shown in Fig. 4. Meanwhile, Fig. 3 also shows that there are pronounced differences in C content among different M-A islands.

Fig. 2 Representative load–displacement curves recorded during nanoindentations of bainite ferrite and M-A island

Fig. 3 Distribution of carbon element in granular bainite obtained by EPMA

Fig. 4 Bright-field (a) and dark-field (b) TEM images of martensite in the M-A island

2.2 Effect of Tempering Temperature on the Mechanical Properties of Granular Bainite

After 2.25Cr-1Mo-0.25V steel was held at 940 °C for 2 h and air-cooled, its microstructure was typical granular bainite. At this time, its tensile strength σ_b was 1163 MPa, yield strength σ_s was 925 MPa, and brittle fracture occurred during impact testing at −18 °C, with an impact energy of only 7 J. Figure 5 shows the tensile properties of 2.25Cr-1Mo-0.25V steel after tempering at different temperatures for 4 h. It can be seen that, when tempered below 630 °C, both σ_b and σ_s show a slow decreasing trend. When the tempering temperature is increased to the range of 630–750 °C, σ_b and σ_s decrease rapidly. When the tempering temperature is increased to 750 °C, σ_b and σ_s decrease to 695 and 543 MPa, respectively, but still exceed the requirements of the ASME specification for the mechanical-property indices of 2.25Cr-1Mo-0.25V steel used in large hydrogenation reactors ($\sigma_b \geq 585$ MPa; $\sigma_s \geq 415$ MPa). The elongation of the material exhibits a trend opposite to that of strength as the tempering temperature increases. At 500 °C tempering, the elongation is the lowest, 11.9%; after tempering at 750 °C, the elongation reaches a peak value of 25.0%.

Figure 6 shows the impact toughness at −18 °C of 2.25Cr-1Mo-0.25V steel

Fig. 4

Figure 4: Fig. 4

Figure 5

Figure 5: Figure 5

Figure 6

Figure 6: Figure 6

after holding at 940 °C for 2 h, air cooling after normalizing, and tempering at different temperatures. It can be seen that, when tempered between 500 and 720 °C, the impact toughness improves to a certain extent with increasing tempering temperature. When tempered below 680 °C, the impact toughness increases only slightly with increasing tempering temperature, and the overall impact energy is relatively low. After tempering at 720 °C, the average impact energy reaches a peak value of 254 J, and the impact energies of the four specimens are relatively stable. When the tempering temperature is further increased to 750 °C, the impact toughness of the specimens is slightly lower than that of the 720 °C-tempered specimens, and the stability of impact energy decreases. After tempering at 700–750 °C, the impact energies of the individual impact specimens at −18 °C fluctuate between 127 and 270 J, but the toughness is relatively good. The ASME specification requires that, for 2.25Cr-1Mo-0.25V steel used in large hydrogenation reactors, the average impact energy at −18 °C be $ $54 J and the minimum impact energy of specimens tested at −18 °C be $ $48 J. From Figures 5 and 6, it can be seen that 2.25Cr-1Mo-0.25V steel with a granular bainite microstructure obtained by normalizing and air cooling can simultaneously meet the requirements for strength and impact toughness after tempering at 700–750 °C; among these conditions, the specimen tempered at 720 °C exhibits the best overall mechanical properties.

Figure 5 Effect of tempering temperature on the mechanical properties of 2.25Cr-1Mo-0.25V steel

Figure 6 Effect of tempering temperature on the impact toughness of 2.25Cr-1Mo-0.25V steel

2.3 Effect of Tempering Temperature on the Microstructure

The SEM and TEM images of the microstructures of 2.25Cr-1Mo-0.25V steel after tempering at different temperatures are shown in Figures 7 and 8, respectively. Figure 8a is a TEM image of the ferrite matrix after normalizing. It can be seen that the ferrite matrix contains a clear lath structure and has a very high dislocation density; no second-phase particles precipitate on the dislocation lines, indicating that normalizing at 940 °C can essentially dissolve the carbides and ensure that alloying elements such as Cr, Mo, and V are sufficiently dissolved in the ferrite, thereby enhancing the solid-solution strengthening effect and the dispersion strengthening by carbides after tempering. When tempered at 500 °C, SEM observation shows that the microstructure still retains the features of

Fig. 7 SEM images of normalized 2.25Cr-1Mo-0.25V steel tempered at 500 °C (a), 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f)

Figure 7: Fig. 7 SEM images of normalized 2.25Cr-1Mo-0.25V steel tempered at 500 °C (a), 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f)

the normalized structure (Figure 7a). XRD measurements indicate that, after tempering at 500 °C, the retained austenite in the M-A islands has completely transformed. This may be due to stress release during tempering and the precipitation of fine carbides, causing the retained austenite in the M-A islands to transform into martensite and fine carbides[ˆ10]. In the microstructures tempered at 630 and 680 °C for 4 h, a large amount of island-like phase is still present. The carbides precipitated in the microstructure are mainly distributed on the ferrite matrix in needle-like and lath-like morphologies, as shown in Figures 7b and 7c. When tempered at 630 °C, TEM observation reveals a large number of extremely fine needle-like carbides in the specimen matrix and on dislocation lines (Figure 8b). When the tempering temperature is increased to 680 °C, the precipitated carbides coarsen and are mainly distributed in lath-like form within the ferrite matrix (Figure 8c). When tempered below 680 °C, the dislocation density in the ferrite matrix remains relatively high. According to Refs. [15–18], when CrMoV steel is tempered at relatively low temperatures, precipitates such as MC and M_3C in the bainitic ferrite matrix pin dislocations, hindering recovery and recrystallization of the ferrite matrix at lower tempering temperatures. Therefore, the granular bainite microstructure obtained by normalizing 2.25Cr-1Mo-0.25V steel has relatively high tempering resistance. As can be seen from Figures 7d and 7e, after tempering at 700 and 720 °C, the island-like phases in the microstructure have essentially decomposed into carbides and ferrite; carbides precipitated in the bainitic ferrite matrix and at grain boundaries have also essentially spheroidized. In addition, the tempering process is accompanied by the gradual disappearance of dislocation cells and intragranular dislocations in the ferrite, reducing the dislocation density of the matrix. Under TEM, it can still be observed after tempering at 700 °C that···

Fig. 7 SEM images of normalized 2.25Cr-1Mo-0.25V steel tempered at 500 °C (a), 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f)

At individual positions along the grain boundaries of the tempered specimen, incompletely dissolved island-like constituents can be observed. Within these island-like constituents, elongated carbides can be seen (Fig. 8d). When the tempering temperature is 750 °C, the lath structure of the ferrite matrix has completely disappeared. At this time, the carbides are mainly distributed along grain boundaries and exhibit pronounced coarsening. From SAED patterns and EDS analysis, it is known that the elongated or granular second phases distributed at the grain boundaries are mostly $M_{23}C_6$-type carbides. The EDS analysis results show that the metallic elements in the carbides are mainly Fe and Cr, while the contents of Mo and V are relatively low.

Fig. 8 TEM images of 2.25Cr-1Mo-0.25V steel normalized (a) and tempered at 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f) (Insets show the corresponding SAED patterns)

Figure 8: Fig. 8 TEM images of 2.25Cr-1Mo-0.25V steel normalized (a) and tempered at 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f) (Insets show the corresponding SAED patterns)

From the above, it can be seen that when 2.25Cr-1Mo-0.25V steel is tempered in the range of 500–750 °C, if the tempering temperature is below 630 °C, the granular bainitic ferrite matrix still retains, to a certain extent, the characteristics of the normalized state. A large amount of island-like constituents and needle-like carbides are present in the microstructure and distributed on the ferrite matrix. As the tempering temperature increases, the ferrite boundaries tend to become straight, the lath structure gradually disappears, and the island-like constituents decompose into ferrite and lath-like carbides. The needle-like or lath-like carbides on the ferrite matrix aggregate, grow, and spheroidize. When the tempering temperature rises to 750 °C, the ferrite has undergone equiaxation, and the carbides are clearly aggregated and coarsened toward the grain boundaries. Therefore, after tempering at 500–750 °C, with increasing tempering temperature, the matrix is gradually softened, and the tensile strength and yield strength show an overall decreasing trend.

2.4 Analysis of impact fracture after tempering at different temperatures

Figures 9 and 10 respectively show the SEM images of the impact fractures and CLSM images of the side sections of the impact fractures of normalized 2.25Cr-1Mo-0.25V steel tempered at different temperatures and tested at −18 °C. It can be seen that the specimens tempered at 500 and 630 °C exhibit cleavage fractures or quasi-cleavage fractures. The cleavage fracture surfaces contain unit cleavage facets of different sizes (unit cleavage) (Fig. 9a and b). The unit cleavage facets of the specimen tempered at 630 °C are smaller, indicating that the unit crack path is shorter. When microcracks propagate along the cleavage facets, obstruction causes the propagation direction to change, requiring more energy consumption. At the same time, the cleavage-fracture region also contains fine tearing ridges.

Fig. 8 TEM images of 2.25Cr-1Mo-0.25V steel normalized (a) and tempered at 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e) and 750 °C (f) (Insets show the corresponding SAED patterns)

Toughness tear ridges (indicated by arrows in Fig. 9b) are favorable for improving toughness; therefore, the impact toughness of the specimen tempered at 630 °C is higher than that tempered at 500 °C. From the CLSM images of the side section of the impact fracture surface (Fig. 10a and b), it can be seen that near the fracture surface, microcracks are formed by separation of M–A

islands from the ferrite matrix (as indicated by arrows in Fig. 10a and b). The resulting microcracks are relatively straight, and no obvious deformation occurs in the surrounding ferrite matrix. These microcracks can act as paths for rapid crack propagation. After tempering at 680 and 700 °C, a certain proportion of dimples appears on the impact fracture surface of the specimen (inset in Fig. 9d). Some microcracks can still be observed in the CLSM images of the side section of the fracture surface (Fig. 10c and d), but at this time the ferrite near the microcracks has undergone obvious plastic deformation, and some cracks exhibit branching (indicated by arrows in Fig. 10c). It can also be observed that some microcracks initiate around incompletely decomposed M–A islands. When tempered at 720 °C, the impact fracture surface has relatively large and deep dimples with a uniform distribution, and the specimen shows ductile fracture (Fig. 9e). At this time, the M–A islands in the microstructure have completely decomposed into ferrite and dispersed granular carbides, and the ferrite matrix consumes energy through extensive plastic deformation during crack formation and propagation (as indicated by the arrows in Fig. 10e, where the cracks near the fracture surface show obvious deformation). At this time, the specimen has the highest impact energy. When the tempering temperature is 750 °C, the impact specimen still exhibits ductile fracture (Fig. 9f), and in the microstructure the···

Fig. 9 Fracture surface morphologies of 2.25Cr-1Mo-0.25V steel tempered at 500 ℃ (a), 630 ℃ (b), 680 ℃ (c), 700 ℃ (d), 720 ℃ (e) and 750 ℃ (f) after Charpy impact test at −18 ℃ (Arrows in Fig. 9b indicate ductile fracture bands and inset in Fig. 9d indicates a region of dimples)

The lath structure disappears, and the ferrite develops toward polygonal and equiaxed morphologies. Recovery softening occurs in the ferrite matrix, making its ability to impede crack propagation relatively weak.

After 2.25Cr-1Mo-0.25V steel is held at 940 ℃ for 2 h and then air-cooled, a granular bainite microstructure is obtained. After tempering at different temperatures, because the degree of transformation and decomposition of M-A islands, as well as the size and morphology of the carbides transformed from them, differ, the impact energy exhibits obvious differences. From the experimental results, it can be inferred that, under the same normalizing process, the key factors affecting the tempered impact toughness of 2.25Cr-1Mo-0.25V steel are the recovery softening effect of the matrix microstructure, the degree of tempering transformation of M-A islands, and the morphology and distribution of carbides[19-23]. The microstructural observations show that, when tempering is performed at relatively low temperatures, the M-A islands in granular bainite have relatively high stability, and needle-like dispersed carbides precipitate in the ferrite matrix. As the tempering temperature increases, the M-A islands in granular bainite gradually decompose into ferrite and lamellar carbides; the needle-like carbides in the matrix coarsen by aggregation and spheroidize. With a further increase in tempering temperature, for lamellar carbides, differences in interfacial curvature lead to nonuniform chemical potentials, and the chemical

Fig. 10

Figure 9: Fig. 10

potential difference provides the driving force for C diffusion, thereby causing spheroidization of the carbides with planar interfaces[5,23]. At the same time, the pinning effect of carbides on the ferrite lath boundaries is weakened, making the ferrite lath boundaries tend to become planar, and the ferrite matrix develops toward polygonal and equiaxed morphologies. Therefore, with increasing tempering temperature, recovery softening occurs in the ferrite matrix, and the carbides gradually transform from needle-like or lamellar to granular, with a tendency to aggregate and grow.

When tempering below 630 ℃, on the one hand, the microstructure still contains a large amount of micron-scale undecomposed M-A islands. At this time, the M-A islands are equivalent to hard particles embedded in the bainitic ferrite matrix. When the specimen is subjected to an external load, the M-A constituents hinder the motion of dislocations in ferrite, causing dislocation pile-up; the piled-up dislocations in turn exert stress on the M-A islands. In addition, because the M-A islands and ferrite have different deformation capacities, incompatible deformation between them leads to stress generation; the piled-up dislocations and···

Fig. 10 CLSM images of the cross-sectional area of the Charpy impact specimens fractured at −18 °C for the 2.25Cr-1Mo-0.25V steel normalized and tempered at 500 °C (a), 630 °C (b), 680 °C (c), 700 °C (d), 720 °C (e), and 750 °C (f).
(Arrows indicate the corresponding microcracks.)

The interaction of stresses causes the M–A islands to fracture or separate from the matrix, or causes microcracks to form in the surrounding ferrite as a result of stress concentration. Under the further action of the external load, those microcracks that satisfy the Griffith criterion propagate into the ferrite, causing quasi-cleavage fracture and severely deteriorating the impact toughness

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. On the other hand, fine acicular carbides precipitated from the matrix have a strong pinning effect on dislocations; that is, the softening effect of the matrix is weak. At the same time, during tempering at relatively low temperatures, relatively high residual stresses still remain at or around the interfaces between the M–A islands or carbides and the ferrite matrix. This also makes the vicinity of these interfaces, during subsequent impact, more likely to reach the threshold critical fracture stress, thereby inducing crack initiation and resulting in poor impact toughness

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When tempered at 680 °C, a considerable proportion of the island-like constituents has decomposed into lath-like or granular carbides. The occurrence of quasi-cleavage fracture is related not only to the incompletely decomposed island-like constituents but also to lath-like carbides. This is because, under stress, when dislocation motion in the matrix encounters these elongated carbides, it is impeded, producing dislocation pile-ups and forming cracks at the ferrite/carbide interfaces. Meanwhile, since the lath-like carbides are brittle phases with poor plasticity, under impact loading, when stress concentration reaches a certain level, the carbides themselves may crack and become cracks, or cracking may occur at the interfaces. Whether cracks form at the ferrite/carbide interfaces or within the carbides themselves, they all become fracture origins for subsequent quasi-cleavage fracture. Therefore, the material tempered at 680 °C exhibits quasi-cleavage fracture, and the impact energy of the specimen is relatively low.

When tempered in the range of 680–720 °C, the M–A islands have essentially decomposed into ferrite and carbides. The fine acicular carbides in the bainitic ferrite matrix have coarsened and spheroidized, weakening their pinning effect on dislocations and causing a certain degree of recovery and recrystallization softening in the microstructure, which leads to a decrease in material strength. As the tempering temperature increases, the acicular or lath-like carbides become granular and are dispersed in the ferrite matrix.

dispersed granular carbides reduce stress concentration and markedly increase the critical fracture stress for crack initiation at the carbide–ferrite matrix interface, so that the work of microcrack initiation during impact is increased and stress concentration is reduced, thereby improving impact toughness. As the tempering temperature continues to rise, carbide coarsening and the disappearance of the lath structure in the ferrite matrix increase the effective grain size, reducing the ability to impede crack propagation during impact, and the impact properties decrease to some extent

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. Therefore, provided that the material strength requirements are met, the tempering temperature for 2.25Cr-1Mo-0.25V steel should be selected within the temperature range in which the M-A islands have completely decomposed and spheroidized carbides have precipitated on the ferrite matrix, so as to obtain a favorable combination of strength and toughness.

3 Conclusions

(1) After normalizing, 2.25Cr-1Mo-0.25V steel exhibits a granular bainite microstructure composed of bainitic ferrite and M-A islands. Owing to carbon enrichment, the M-A islands have a hardness significantly higher than that of the matrix.

(2) After normalizing, the tensile strength and yield strength of 2.25Cr-1Mo-0.25V steel decrease gradually with increasing tempering temperature. At 750 °C the strength is the lowest, but it still satisfies the specification requirements. The tempering temperature has a pronounced effect on impact toughness. When the tempering temperature is below 700 °C, the impact toughness is relatively poor; at a tempering temperature of 720 °C, the impact energy reaches a peak value of 254 J.

(3) The granular bainite obtained after normalizing 2.25Cr-1Mo-0.25V steel has very high tempering stability. After tempering at 500–630 °C, the strength is relatively high and the impact toughness is poor. This is due to the strong pinning effect on dislocations caused by the large amount of undecomposed brittle M-A islands in the granular bainite and the fine acicular carbides precipitated within the ferrite matrix. When the tempering temperature is 700–720 °C, the carbides in the ferrite matrix spheroidize, the blocky M-A islands are essentially decomposed into ferrite and carbides, and the carbide morphology changes from rod-like or lamellar to granular, transforming from an inhomogeneous to a uniformly dispersed distribution; as a result, the impact toughness of the specimens improves. With a further increase in tempering temperature, carbide coarsening through aggregation at grain boundaries and obvious coarsening of the ferrite matrix are the main reasons for the decrease in impact toughness. In addition to the recovery and softening effect of the bainitic matrix, differences in the transformation of M-A islands and in the nature, size, and distribution of carbides in the microstructure are the principal factors affecting impact toughness.

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