Effects of Cyclic Heat Treatment on Microstructure and Mechanical Properties of Ti-V Microalloyed Steel (Postprint)

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Abstract

The effects of cyclic heat treatment on the microstructure and mechanical properties of Ti-V microalloyed steel were investigated. Based on microstructural observation results, the grain sizes of the experimental steel at various cycle numbers were statistically analyzed and calculated, and combined with SEM secondary electron images, the microstructural evolution behavior of the experimental steel during cyclic heat treatment was elucidated, thereby explaining the grain refinement mechanism. TEM experiments were conducted on the experimental steel to observe the variation behavior of precipitate particle size and composition with cycle number; room-temperature tensile strength and impact toughness tests were also performed on the experimental steel at different cycle numbers. The results show that the average grain size of Ti-V microalloyed steel continuously decreases with increasing cycle number (up to the 7th cycle); influenced by the average size of complex carbides, the tensile strength Rm gradually decreases with increasing cycle number (starting from the 1st cycle); and the impact absorbed energy AKU, due to the combined influence of average grain size and proeutectoid ferrite proportion, first increases and then decreases with increasing cycle number, reaching its maximum at the 3rd cycle.

Full Text

Preamble

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Effect of Cyclic Heat Treatment on Microstructure and Mechanical Properties of Ti-V Microalloyed Steel

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Abstract

The effect of cyclic heat treatment on the microstructure and mechanical properties of Ti-V microalloyed steel was investigated. The metallographic microstructure of the steels after each heat treatment cycle was examined and the corresponding grain size was statistically determined. The cyclically heat-treated steels were also examined by means of SEM and TEM to reveal the microstructural evolution and the variations in average size and composition of precipitates with increasing cycle number, while their tensile strength and toughness were measured.

The results show that the average grain size of the steel decreases continuously with increasing cycle number (up to 7 cycles). The tensile strength Rm gradually decreases with increasing cycle number (starting from the first cycle) due to the influence of the average size of complex carbides. The impact absorption energy AKU initially increases and then decreases with cycle number, reaching its maximum at the third cycle, as a result of the combined influence of average grain size and the proportion of proeutectoid ferrite.

KEY WORDS: metallic materials, cyclic heat treatment, Ti-V microalloyed steel, grain refinement, complex carbides, mechanical properties

Introduction

Quenched and tempered steels contain fine, dispersed ferrite-cementite mixtures, which provide a good combination of yield strength and toughness. If the prior austenite grains are finer, both the yield strength and toughness of quenched and tempered steel will be higher. An effective method for prior austenite refinement is temperature cycling, which involves repeated heating and cooling of the steel. Various microstructural transformations and their effects on process kinetics and material properties fully demonstrate the potential of cyclic heat treatment technology. Therefore, cyclic heat treatment based on quenching and tempering can significantly improve the comprehensive mechanical properties of materials. The advantage of cyclic heat treatment is that it can achieve grain refinement through heat treatment alone without thermomechanical processing, reducing experimental difficulties and costs.

Based on microalloying elements, two technical routes are considered capable of achieving the required mechanical properties: austenite grain refinement and precipitation strengthening. For Ti-V microalloyed steel without deformation history, it is essential to carefully consider the effect of quenching and tempering treatment on the type, quantity, and size of precipitate particles. Selecting appropriate cycling temperatures and times ensures sufficient dissolution of alloying elements to guarantee dispersed precipitation effects after tempering, while avoiding the reduction in dislocation pinning effectiveness caused by precipitate particle coarsening. Previous research on microalloyed steel has mainly focused on the precipitation process of carbonitrides, dynamic recrystallization behavior, and microstructural evolution during deformation. This paper investigates cyclic heat treatment of Ti-V microalloyed steel.

Traditional cyclic quenching and tempering processes employ pre-treatment + continuous quenching and tempering. This method is relatively complex, primarily due to the alternating heat treatment temperatures, which increases the required equipment and number of practical operations. The cyclic quenching and tempering process employed in this study uses pre-treatment + continuous quenching + final tempering. This method significantly reduces the required equipment and number of practical operations compared with traditional processes and is more time-efficient. This paper investigates the microstructural evolution and grain refinement mechanism of Ti-V microalloyed steel during cyclic heat treatment and analyzes the variation patterns and underlying reasons for mechanical properties with cycle number.

Experimental Methods

The main chemical composition of the experimental Ti-V microalloyed steel is listed in Table 1 . The original as-cast material had a tensile strength Rm 700 MPa and impact absorption energy 120 J. Metallographic specimens with dimensions of 10 mm × 10 mm × 15 mm were cut from the ingot by wire electrical discharge machining and used to observe grain size and corresponding microstructure after different numbers of heat treatment cycles. For mechanical property evaluation, additional tensile and impact specimen blanks were cut and, after different numbers of heat treatment cycles, were finish-machined into standard R7 tensile specimens (gauge length 25 mm, d_0 = 5 mm, $L_0 = 5d_0$) and standard Charpy U-notch impact specimens (10 mm × 10 mm × 55 mm) according to GB/T228-2010 and GB/T229-2007, respectively.

The designed cyclic heat treatment process route is shown in Figure 1 [Figure 1: see original paper], with cycle numbers of 0, 1, 2, 3, 4, and 7. The entire process consists of three stages: pre-treatment, continuous quenching, and final tempering. The pre-treatment involves high-temperature solution treatment (960°C, 30 min) + short-term normalizing (960°C, 5 min). The subsequent cycling process includes the same number of short-term continuous quenching treatments (870°C, 5 min) as the current cycle number, followed by a final tempering (570°C, 40 min). Except for air cooling (AC) during normalizing in the pre-treatment, oil cooling (OC) is used for all other cooling processes.

The heat-treated metallographic specimens were ground and polished. Some were etched with saturated picric acid solution in a 60°C constant temperature water bath to reveal grain boundaries, while the remaining specimens were etched with 4% nital to reveal the microstructure. Grain boundaries and microstructures were observed using a ZEISS AxioCamMR5 optical microscope. Grain size was calculated and statistically analyzed using Image-Pro Plus software. To investigate the grain refinement mechanism, etched specimens were examined using a ZEISS ULTRA 55 thermal field-emission scanning electron microscope (SEM) to observe microstructural characteristics at each cycle number. TEM observations were performed on specimens from each cycle number using a JEM-2000FX instrument at an accelerating voltage of 160 kV, supplemented with energy-dispersive spectroscopy (EDS) for precipitate composition analysis using a NORAN-VOYAGER system. To study the variation of mechanical properties with cycle number, room temperature tensile and impact toughness tests were conducted using a CMT4105 universal testing machine and a ZBC2452-B pendulum impact tester, respectively.

2.1 Grain Refinement and Mechanism

The grain morphologies of pre-treated specimens and specimens subjected to different numbers of cycles are shown in Figures 2 [Figure 2: see original paper] and 3 [Figure 3: see original paper]. After solution treatment, the steel grains were relatively coarse (Fig. 2a). The control of austenite grain size in microalloyed steel is primarily based on Zener's "second-phase particle grain boundary pinning model" and its various modifications. This phenomenon occurs because, during prolonged high-temperature solution treatment, some precipitate particles may coarsen through Ostwald ripening, reducing their grain boundary pinning capability. Unpinned isolated grains then begin to consume surrounding fine grains and grow rapidly (as seen in Fig. 2a, where coarse grains are surrounded by fine grains), representing abnormal grain growth. With extended holding time, the coarse grains become larger while the fine grains become smaller, resulting in significant grain size inhomogeneity.

After subsequent normalizing of the solution-treated specimen (completion of pre-treatment), the steel grain size was approximately 9.4 m, significantly reduced compared with before (Fig. 2b). This is because normalizing is a diffusional transformation that refines the microstructure. After the first cycle, the grain size further decreased to 7.3 m. With continued cycling, the grain size decreased approximately linearly, reaching 4.6 m after 7 cycles (Figs. 3a-e). The overall variation trend of average grain size with cycle number is shown in Figure 4 [Figure 4: see original paper].

It is noteworthy that since the experimental steel has no deformation history, the final tempering (570°C, 40 min) does not affect the grain size obtained after continuous quenching (i.e., final tempering does not cause recrystallization of the steel). Therefore, to investigate the grain refinement mechanism, SEM examination was performed on continuously quenched specimens from different cycle numbers to observe their microstructural evolution, as shown in Figure 5 [Figure 5: see original paper].

0 cycles (pre-treated condition): Since the pre-treatment ends with normalizing and the experimental steel is hypoeutectoid, the microstructure consists of proeutectoid ferrite (black regions in the figure) and lamellar pearlite (Fig. 5a). The austenite phase is carbon-enriched relative to ferrite. The purpose of normalizing in this work is twofold: first, to restrict the nucleation and growth region of austenite within the pearlite region (carbon in the normalized microstructure exists mainly as cementite lamellae in pearlite), thereby refining austenite grains; second, compared with the diffusionless transformation during quenching, the diffusional transformation during normalizing allows some diffusion of alloying elements, improving microstructural homogeneity.

1 cycle: As shown in Fig. 5b, dissolution of cementite lamellae occurred during the quenching holding period of the first cycle. The heating temperature was set just above AC_3 and the holding time limited to 5 min to prevent excessively rapid dissolution of cementite lamellae, maintaining a relatively low solid solution carbon concentration in the original pearlite region and preventing rapid austenite grain growth. Meanwhile, the incompletely dissolved cementite lamellae could also hinder austenite grain growth.

2 cycles: During the second cycle, new austenite began to nucleate and grow extensively, but this nucleation and growth was confined to the original pearlite region. A real lamellar pearlite structure is never perfect: defect-enriched high-curvature regions such as kinks, striations, voids, cracks, and terminations commonly exist in cementite lamellae. These high-curvature regions have high chemical potential, causing cementite lamellae to dissolve preferentially at these defects. Undoubtedly, cementite lamellae terminations (connecting to pearlite boundaries), being the most abundant defects, dissolve first during quenching holding, making this region relatively carbon-enriched. Additionally, with pearlite boundaries serving as nucleation sites, austenite nucleation and growth are favored at these locations, as evidenced by the growth positions of lath martensite shown in Fig. 5c. Naturally, other internal defect locations also provide possible sites for austenite nucleation and growth.

3 cycles: In the third cycle, more new austenite nucleated and grew within the original pearlite region. However, as the number of austenite grains in the region increased, martensite packets with different orientations (such as Orientation 1 and Orientation 2 in Fig. 5d) that grew rapidly upon cooling collided and intersected, causing fragmentation at the roots of martensite packets and generating numerous structural defects. As high-energy regions, these structural defects provide more nucleation sites for the next cycle, promoting further refinement of austenite grains within the original pearlite region.

4-7 cycles: Before the fourth cycle, grain refinement was concentrated in the original pearlite region, representing localized refinement. Starting from the fourth cycle, some austenite grains began to grow from the original pearlite boundaries into the ferrite matrix, as can be seen from the growth pattern of martensite needles (MN) in Fig. 5e. This led to overall grain refinement of the material and caused a continuous decrease in the proportion of ferrite phase in the steel. Compared with the pre-treated specimen (Fig. 5a), the proportion of ferrite phase became extremely small after 7 cycles (Fig. 5f).

It is also noteworthy that due to pinning by precipitate particles, a "dual-phase structure" consisting of abnormally grown coarse grains and gradually consumed fine grains exists in microalloyed steel (Fig. 2a). Combined with continuous nucleation and growth of fine grains during cyclic heat treatment (Fig. 3), this results in a certain degree of grain size inhomogeneity. Here, RD (relative differences) is introduced to characterize the relative difference in grain size of the steel. The calculation method for RD is given by Equations (1) and (2), where GS b represents the average size of abnormally grown grains, and σ is the standard deviation of all austenite grain sizes measured when calculating GS (average grain size). If very few measured grain sizes satisfy Equation (1), the maximum value is taken as GS b .

$$\text{abnormal} = \text{abnormal} -$$

$$\mathrm{RD} = \frac{\mathrm{GS}_{\text{abnormal}} - \mathrm{GS}}{\sigma}$$

From Equation (2), it is clear that an RD value closer to 0 indicates better grain size uniformity. Table 2 presents the RD values of the steel after different numbers of cycles. The results show that a significant degree of grain size inhomogeneity exists after cyclic heat treatment (all RD values are close to or exceed 1). However, the RD value does not show a clear trend with increasing cycle number; except for relatively smaller RD values at 0 and 7 cycles, the RD values for 1-4 cycles remain essentially at the same level.

2.2 Precipitate Analysis

As shown in Table 1, the precipitate particles potentially present in the original steel are Ti(C, N) and V(C, N). The formation of precipitate particles occurs in three stages: Stage I forms in the liquid phase and during solidification; Stage II forms in austenite during solution treatment or thermomechanical processing; and Stage III forms during the austenite-to-ferrite transformation. According to the solubility product curves proposed by Nagata et al. and considering the precipitation sequence and elemental contents in the steel (Table 1), the majority of TiN precipitates in austenite, and other types of precipitates also form in the solid phase. Therefore, precipitate particles formed in the liquid phase are not considered in this work, as they have very small volume fractions, are highly stable, and are too large to effectively pin grain boundaries and dislocations.

Based on the solubility product formulas proposed by Taylor and Narita and the effective Ti content formula (Equations (3)-(7)), the dissolution temperatures of TiN, TiC, and VC in austenite in the original steel were calculated to be 1499°C, 990°C, and 800°C, respectively. According to the solubility sequence, all N in the steel combines with Ti to form TiN, so theoretically no VN precipitates exist. Furthermore, large TiN particles (micron-sized) cannot dissolve within the experimental temperature range and cannot re-precipitate during subsequent heat treatment. Consequently, only TiC and VC are discussed as potentially existing precipitates.

$$\lg[\mathrm{Ti}][\mathrm{N}]_\gamma = 5.4 - \frac{15790}{T}$$

$$\lg[\mathrm{Ti}][\mathrm{C}]_\gamma = 2.75 - \frac{7000}{T}$$

$$\lg[\mathrm{V}][\mathrm{N}]_\gamma = 3.46 - \frac{8830}{T}$$

$$\lg[\mathrm{V}][\mathrm{C}]_\gamma = 6.72 - \frac{9500}{T}$$

$$[\mathrm{TiC}]_{\mathrm{effective}} = [\mathrm{TiC}]_{\mathrm{total}} - 3.42[\mathrm{N}]_{\mathrm{TiN}} - 3[\mathrm{S}]_{\mathrm{Ti4C_2S_2}}$$

Figure 6a [Figure 6: see original paper] shows the precipitate particle morphology and EDS analysis of the steel after only solution treatment (960°C, 30 min). Since the solution temperature lies between the dissolution temperatures of VC and TiC, the precipitate particles in this state are primarily TiC, as reflected in the EDS spectrum. After normalizing treatment, the microstructure also contains VC interphase precipitation produced during the $\gamma \rightarrow \alpha$ transformation. After one cycle, the precipitate particle EDS spectrum simultaneously shows peaks for both Ti and V, with a very high Ti peak (Fig. 6b). This indicates that VC precipitated complexly on the existing TiC particles during tempering. During cycling, continuous quenching caused the VC precipitated during normalizing to redissolve, which then complexly reprecipitated on the previous TiC particles during final tempering. The likely reason is that Ti and V are both transition metals with extremely similar physical and chemical properties, and their carbonitrides have similar structures. These factors determine that their carbides are mutually soluble, forming multi-component complex carbides.

After various numbers of cyclic heat treatments, the types of precipitate particles in the steel remained unchanged. It can also be seen that the precipitate particle density in the experimental steel does not change with increasing cycle number.

The relationship between average complex carbide size and cycle number is shown in Figure 7 [Figure 7: see original paper]. The average complex carbide size increases monotonically with increasing cycle number. The reason may be that as the cumulative quenching time increases with cycle number, the amount of V in solid solution continuously increases, resulting in more VC complex precipitation during final tempering and consequently larger average particle sizes of the complex precipitates.

2.3 Mechanical Properties

The mechanical properties of the experimental steel were evaluated based on room-temperature tensile strength Rm (Resistance maximum) and impact absorption energy AKU. The primary focus was investigating the variation patterns of Rm and AKU with cycle number during cyclic heat treatment and analyzing the underlying reasons in conjunction with microstructural characteristics.

As shown in Figure 7 [Figure 7: see original paper], the steel' s Rm reaches its maximum after the first cycle and then gradually decreases with further cycling. The increase in Rm after the first cycle compared with the pre-treated condition (0 cycles) is due to the significant grain size reduction (see Fig. 4). Although the average grain size continues to decrease with increasing cycle number, Rm shows a decreasing trend. Clearly, grain refinement strengthening is no longer the dominant strengthening mechanism after the first cycle. On the other hand, the cumulative quenching time increases with cycle number, leading to continuously higher amounts of alloying elements in solid solution, while the final tempering conditions remain the same for all cycle numbers. Therefore, from the perspective of dispersion precipitation amount, Rm should gradually increase with cycle number. However, the opposite trend is observed, necessitating consideration of the effect of precipitate particles on Rm during cyclic heat treatment, i.e., precipitation strengthening mechanism.

Since the average complex carbide size in this experiment is relatively large (Fig. 7), the precipitation strengthening mechanism follows the Orowan mechanism, where, with constant complex carbide density (Section 2.2), smaller particle sizes produce better strengthening effects. Additionally, dislocation density increases with cycle number (cumulative quenching cycles), making the effect of second-phase particles on Rm more pronounced, as shown in Figure 8 [Figure 8: see original paper]. Figure 7 shows that as the average size of second-phase particles (complex carbides) increases monotonically with cycle number, the tensile strength Rm decreases monotonically. From this pattern, it can be concluded that the dominant strengthening mechanism after one cycle of heat treatment is precipitation strengthening, i.e., the average complex carbide size is the main factor affecting Rm.

The variation pattern of AKU can be analyzed in conjunction with the microstructural evolution of quenched specimens at different cycle numbers discussed in Section 2.1, as shown in Figure 9 [Figure 9: see original paper]. It can be seen that before the third cycle, austenite nucleation and growth occurred primarily within the original pearlite region, with continuous grain refinement in this region as cycling continued. Meanwhile, the proportion of proeutectoid ferrite, as a “soft phase,” remained unchanged and contributed positively to the material’s impact toughness. Therefore, AKU continuously increased with cycle number before the third cycle. Starting from the fourth cycle, although the average grain size continued to decrease with increasing cycle number, AKU began to decrease. The reason is that from the fourth cycle onward, some austenite grains began to grow into the ferrite region, causing the proeutectoid ferrite area to gradually decrease with cycle number, which led to a gradual decline in AKU. Thus, for the steel subjected to this heat treatment process, AKU is jointly influenced by average grain size and proeutectoid ferrite proportion, reaching its maximum at the third cycle (184 J), demonstrating significant toughening effects.

Conclusions

1. For Ti-V microalloyed steel, since the cyclic heat treatment pre-treatment ends with normalizing, the grain size of the pre-treated specimen reaches 9.4 m and continues to decrease with increasing cycle number, achieving 4.6 m at the seventh cycle, demonstrating remarkable grain refinement. The grain refinement occurs in two stages: localized grain refinement within the original pearlite region before the third cycle, and overall grain refinement starting from the fourth cycle.
2. During cyclic heat treatment, the precipitate particles in Ti-V microalloyed steel are complex carbides of TiC and VC, whose density does not change with increasing cycle number. The average size of complex carbides increases monotonically with increasing cycle number.
3. Affected by the average size of complex precipitates, the tensile strength Rm of Ti-V microalloyed steel decreases continuously with increasing cycle number after the first cycle. The impact absorption energy AKU is jointly influenced by average grain size and ferrite phase proportion, initially increasing and then decreasing with cycle number, reaching its peak at the third cycle (184 J), showing significant toughening effects.

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Note: Figure translations are in progress. See original paper for figures.