Experimental simulation research on TMCP of 30MnCr22 oil well pipe

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Abstract
Thermo-mechanical controlled process (TMCP) of 30MnCr22 oil well pipes was investigated by experimental simulation. The results showed that controlled rolling of TMCP effectively exerted the comprehensive effects of grains refinement by recrystallization in piercing and continuous rolling and deformation strengthening by strains accumulation in the non-recrystallization zone of reducing deformation. And the key point of TMCP is that quenching realized the microstructural heritability of fine and strengthened austenite, which achieved the best refinement of martensite laths to be 170 nm, nanoscale carbides with the average size of 101 nm and dislocations with the high-density of $8.38 \times 10^9$ mm$^{-2}$, presenting a kind of complicated multi-layer microstructural characteristics. Finally, the synergistic strengthening effects on fine grain strengthening, laths refinement strengthening, dislocation strengthening and precipitation strengthening were realized by TMCP. Further, the synergistic strengthening mechanisms are explored to be that quenching enforced the interaction between martensite laths and dislocations, as well as the entanglement between $\theta$-(Fe, Cr)$_3$C and dislocations.

1. Introduction
At present, with the increasingly fierce competition for resources of petroleum, the special natural environments of exploration hotspots such as deep strata, abyssal sea, and polar alpine regions put forward extremely strict requirements on the comprehensive mechanical properties of oil well pipes especially the strength, which has become an urgent problem in the development of high-grade oil well pipes. Traditionally, the mechanical properties of oil well pipes were mainly improved by adding alloys or off-line heat treatment, i.e. quenching and tempering. However, this not only limited the improvement of mechanical properties due to losing the strengthening effect of rolling deformation, but also increased the cost of alloys, as well as energy by reheating, not conducive to the further development of circular economy for pipe factories.

TMCP can still achieve the ultra-fine microstructure with excellent mechanical properties by precisely controlling and optimizing the whole hot rolling and cooling process without adding too much alloys. And the synergistic effects of multiple strengthening including fine grain strengthening, deformation strengthening, phase transformation strengthening and precipitation strengthening can be fully exerted by TMCP, especially recrystallization controlled rolling followed by on-line heat treatment. Thus, TMCP has already become a promising technique for realizing the resource-saving production of steel products recently [1–5]. Moreover, the new generation of TMCP represented by ultra-fast cooling has been widely applied in the production of hot rolled plates, rods and wires [6–10]. However, due to the hollow section and the complex rolling and cooling conditions, the development and application of ultrafast cooling TMCP technology in hot-rolled steel pipes production is still in its infancy. Nevertheless, some works still have been conducted on TMCP of pipes. It is believed that controlled rolling and controlled cooling are the main two focuses of TMCP. Some reports showed that TMCP played an obvious effect on the hot deformation behavior of pipes by conducting the experimental simulation. Pussegoda L N et al found that recrystallization controlled rolling achieved the refined grains during
The specimens with dimension of $2.5 \times 3.0 \times 15.0$ mm were taken from the continuous casting billet of 30MnCr22 steel pipe. The chemical composition is given in Table 1. Considering the actual PQF rolling process of oil well pipes, the controlled rolling process of TMCP including one-pass piercing, six-pass PQF rolling and seven-pass reducing was specially designed and followed—conducted on the Gleeble-1500D thermal-mechanical simulator. The recrystallization behavior of controlled rolling was systematically studied through the true stress-true strain curves and verified by the microstructural evolution through tracing microstructure heritability. Moreover, the microstructural characteristics obtained by ultra-fast cooling were emphatically investigated using transmission electron microscopy (TEM) for clearing the synergistic strengthening mechanisms of TMCP on 30MnCr22. In order to overcome the current energy shortage and meet the development of low-cost oil well pipes with high performance, the aim of this research is to provide the experimental basis for realizing TMCP in the economical steel pipes, and further confirm TMCP effects on improving the comprehensive properties.

### 2. Materials and methods

The specimens with dimension of $8 \times 15$ mm were taken from the continuous casting billet of 30MnCr22 oil well pipe, and the chemical composition is given in Table 1. Considering the actual PQF rolling process of oil well pipes, the controlled rolling process of TMCP including one-pass piercing, six-pass PQF rolling and seven-pass reducing was specially designed and followed—conducted on the Gleeble-1500D thermal-mechanical simulator, and the detailed parameters are shown in Table 2. Especially, in order to reduce the influence of friction and barreling of the specimen during the compression, graphite and tantalum sheet were stuck between the specimen and the grip by using high-temperature lubricant. Additionally, in order to trace the microstructure heritability, the microstructures of water quenched specimens at different deformation stages of TMCP were observed by Zeiss metallographic microscope (OM) and scanning electron microscopy (SEM). Notably, the specimens were taken from the central area along the cross section of the specimens after deformation where the strain distribution is relatively homogeneous, then mechanically polished and corroded by nitric acid alcohol solution for observation. Next, the cooling rates of $1 \degree C s^{-1}$, $30 \degree C s^{-1}$ and quenching (the cooling rate is about $55 \degree C s^{-1}$), equivalent to air, fast and ultra-fast cooling, separately, were adopted in order to confirm the best cooling effect of TMCP. Further, the microstructural characteristics of fine substructures were comparatively observed in-depth using a JEOL TEM at an acceleration voltage of 200 kV. And the TEM specimens were prepared as thin slices, cut off from the specimens after controlled cooling of TMCP, mechanically grinded to a thickness of approximately $30–50 \mu m$. Then a disc of $3 \mm$ diameter was punched and thinned by electrolytic double spraying for TEM observation. Notably, the tensile test was replaced by the hardness test to characterize the mechanical properties after controlled cooling because the specimens are too small to test the strength after compression deformation.

### Table 1. Chemical composition of 30MnCr22 steel (wt%).

| Element | C  | Si  | Mn  | P   | S   | Cr  | Mo  |
|---------|----|-----|-----|-----|-----|-----|-----|
| wt%     | 0.29 | 0.27 | 1.36 | 0.015 | 0.006 | 0.16 | 0.08 |

piercing, continuous rolling and reducing deformation of hot-rolled steel pipes [11, 12]. Besides, the results of Mihara F et al. indicated that stable dynamic recrystallization occurred during piercing and complete static recrystallization happened between passes during continuous rolling, followed by dynamic recrystallization appeared at the fourth pass of reducing deformation by strains accumulation [5] through conducting the hot torsion tests on 10C-10V and 12C-16V steel pipes. For controlled cooling, i.e. the core of TMCP, a few scholars have conducted some preliminary experimental studies such as on-line normalization, quenching and accelerated cooling [13–16], but not obtained the ideal microstructure due to the uncertain mechanisms of microstructure control and the unknown microstructure heritability from rolling to cooling. Accordingly, a complete system of TMCP has not been formed so far due to lacking of the overall research on the whole process from heating, rolling to cooling of hot-rolled steel pipes, and the comprehensive mechanical properties of steel pipes have not been fully explored. Therefore, TMCP of hot-rolled pipes still needs to be in-depth studied for evaluating TMCP effects, including the unknown recrystallization behavior and the corresponding microstructure heritability, as well as the synergistic strengthening mechanisms of multiple strengthening effects of TMCP, which are all important to achieve the expected microstructures and mechanical properties of steel pipes.

30MnCr22 steel with low alloys is a typically economical steel used for oil well pipes [17–19]. In order to fully research TMCP on 30MnCr22 pipes, this work focuses on realizing the precise track and control of microstructures during controlled rolling and cooling process, and exploring the synergistic strengthening mechanisms of TMCP on 30MnCr22 steel by investigating the microstructural characteristics in detail. According to the Premium Quality Finishing (PQF) process of 30MnCr22 steel pipes, TMCP was specifically designed and follow-up conducted on the Gleeble-1500D thermal-mechanical simulator. The recrystallization behavior of controlled rolling was systematically studied through the true stress-true strain curves and verified by the microstructural evolution through tracing microstructure heritability. Moreover, the microstructural characteristics obtained by ultra-fast cooling were emphatically investigated using transmission electron microscopy (TEM) for clearing the synergistic strengthening mechanisms of TMCP on 30MnCr22. In order to overcome the current energy shortage and meet the development of low-cost oil well pipes with high performance, the aim of this research is to provide the experimental basis for realizing TMCP in the economical steel pipes, and further confirm TMCP effects on improving the comprehensive properties.
3. Results and discussion

3.1. Recrystallization behaviour of 30MnCr22 steel

The recrystallization behavior of 30MnCr22 steel in TMCP including piercing, continuous rolling and reducing deformation was researched through analyzing the corresponding true stress-true strain curves which were obtained from the experimental simulation and shown in figure 1.

According to the true stress-true strain curve obtained in one-pass piercing and one-pass continuous rolling of TMCP shown in figure 1(a), it can be seen that the stress increased at the very beginning, peaked at stress of −46.0 MPa and its corresponding strain was −0.24, then followed by a stable platform due to the softening effect.
which indicated the occurrence of full dynamic recrystallization. This is mainly due to the large piercing strain of 
−1.3, which is very helpful to obtain the refined piercing microstructure. But the time of piercing is so short that
the grains of dynamic recrystallization haven’t enough time to grow up. Then the stress dropped sharply when
the compression was uploaded and metadynamic recrystallization occurred during the interval time of 60 s
between piercing and one-pass continuous rolling. In theory, a fraction softening term is used as a measure of
the softening that occurs during the inter-hit time due to recrystallization as shown in

\[ F = \frac{\sigma_m - \sigma_2}{\sigma_m - \sigma_1} \]  

(1) 

Where, \( \sigma_m \) is the final stress of the first hit, \( \sigma_1 \) and \( \sigma_2 \) are the yield stress of the first and the second hit. And the
fraction recrystallized can be determined by the following equation.

\[ X = (F - 0.2) / 0.8 \]  

(2) 

The fraction of metadynamic recrystallization is calculated to be 70% according to equation (2), meaning that
metadynamic recrystallization has occurred between piercing and one-pass continuous rolling. Besides, dynamic
and metadynamic recrystallization are both helpful to reduce the deformation resistance and improve the hot
workability of billets during the subsequent deformation. Next, from the true stress-true strain curve obtained in
six-pass PQF and three-pass extractor continuous rolling shown in figure 1 (b), it can be easily found that each
strain of the first three passes was small to make dynamic recrystallization happen, but enough to cause static
recrystallization, leading to decreasing the stresses between passes apparently. Besides, the fraction of static
recrystallization are calculated to be 85% and 86% respectively after the first and second pass of continuous rolling
by equation (2), which will refine the austenite grains further. As it should be, the strains between passes can’t be
accumulated, and the interval times are also long (0.42 s and 0.45 s, separately), making dynamic recrystallization
difficult to occur during continuous rolling. Finally, the true stress-true strain curve in seven-pass reducing
dereformation is shown in figure 1 (c). It can be noted that each strain of the first five passes increased sequentially,
indicating that each strain was too small to make static and dynamic recrystallization occur, but could be
accumulated due to the short interval time of 0.1 s. However, the total strain of the first five passes has not
exceeded the critical strain of dynamic recrystallization yet. Hence, strains accumulation in the non-
recrystallization zone of reducing deformation will cause the strengthening and hardening of deformed austenite
and refine the phase transformation microstructure in the cooling. At this time, if ultra-fast cooling is adopted,
fine phase transformation microstructure and excellent mechanical properties can be obtained in the end.

3.2. Microstructural heritability of TMCP on 30MnCr22 steel

In order to investigate the microstructural heritability of TMCP on 30MnCr22 steel, the microstructures of
water quenched specimens at different deformation stages were observed, as demonstrated in figure 4.
Meanwhile, the variation in hardness of microstructures obtained after reducing at different cooling rates is
presented in table 3.

The microstructural heritability of TMCP on 30MnCr22 steel demonstrated the recrystallization behavior
analyzed above. A comparison of figures 3(a) and (b) revealed that the original austenite grains have been greatly
refined by one-pass piercing, and the average grain size was significantly refined from 200 \( \mu m \) to 60.3 \( \mu m \) after

Figure 2. Schematic diagram of static and metadynamic recrystallization softening.
Figure 3. Microstructural heritability of TMCP on 30MnCr22 steel: (a) original austenite; (b) after piercing; (c) after piercing and one-pass continuous rolling; (d) after piercing and two-pass continuous rolling; (e) after continuous rolling; (f) cooling at 1 °C s⁻¹ after reducing; (g) cooling at 30 °C s⁻¹ after reducing; (h) quenching after reducing.

Table 3. Hardness of microstructures obtained after reducing at different cooling rates.

| Cooling rate after reducing / (°C s⁻¹) | Hardness / HV | Average hardness / HV |
|--------------------------------------|--------------|----------------------|
| 1                                    | 241/236/234  | 237                  |
| 30                                   | 439/451/454  | 448                  |
| Quenching                            | 499/506/496  | 500                  |
statistical measurements by using nano-measurer software. This also proved that the obvious grain refinement was achieved by sufficient dynamic recrystallization resulted from a large piercing strain of \(-1.3\) as mentioned above. In order to verify the heredity of microstructure in continuous rolling, the microstructures after the first two passes with larger strains were observed, as shown in figures 3(c) and (d), separately. It was measured that the average grain size of austenite grains was further refined to 46.4 \(\mu\)m and then 34.2 \(\mu\)m after the first and second pass of continuous rolling, which verified that static recrystallization between the first three passes also had a more obvious effect on refining austenite grains. Finally the average grain size after continuous rolling was refined to 23.8 \(\mu\)m, as shown in figure 3(e). As for the refinement effect of cooling rates on the grains, by comparing figures 3(f)–(h), the average grain size after phase transformation was calculated to be 12.7 \(\mu\)m, 11.2 \(\mu\)m and 10.0 \(\mu\)m, separately. So it can be concluded that quenching of TMCP achieved the best refinement of grains by perfectly inheriting the fine microstructure resulted from recrystallization in piercing and continuous rolling, as well as strains accumulation in the non-recrystallization zone of reducing deformation, which will refine the phase transformation microstructure in the cooling. In addition, it can also be noticed from figure 3(h) that some martensite laths obtained by quenching shared the similar orientation formed a block of martensite, similar to a sub-grain, as pointed by arrow A. Whereas the block of martensite next to A, such as that pointed by arrow B, were growing at a different orientation from A. Thereby, the original austenite grains were divided into several sub-grains by blocks of martensite with different orientations at quenching of TMCP, which acted as the obvious role of sub-grain strengthening.

The influence of TMCP effects on the mechanical properties of 30MnCr22 can also be proved by the hardness shown in table 3. In theory, the hardness can be used to characterize the strength indirectly. In other words, the variation of hardness at different cooling rates can explain the controlled cooling effects of TMCP on the strength. It can be noticed from table 3 that the hardness increased from 237 HV to 448 HV with the cooling rate increasing from \(15^{\circ}\)C s\(^{-1}\) to \(30^{\circ}\)C s\(^{-1}\), finally reached the maximum value of 500 HV at quenching, showing that quenching of TMCP greatly increased the hardness, i.e. the strength of 30MnCr22.

### 3.3. Microstructure characteristics of TMCP

In more detail, microstructural characteristics obtained at \(30^{\circ}\)C s\(^{-1}\) and quenching of TMCP were in-depth investigated by TEM, as shown in figure 4.

A comparison of figures 4(a) and (b) revealed that typical martensite laths obtained at quenching are much finer and more uniform than those obtained at \(30^{\circ}\)C s\(^{-1}\). According to the size distribution histogram shown in figure 4(c), it was statistically measured that the width of martensite laths at \(30^{\circ}\)C s\(^{-1}\) was mainly 300–400 nm, accounting for 75%, and the average size was calculated to be 360 nm. Whereas, martensite laths at quenching were all less than 300 nm, mainly 100–200 nm which accounted for 65%, and the average size was 170 nm, finer than that at \(30^{\circ}\)C s\(^{-1}\) by 53%. So it can be concluded that quenching of TMCP on 30MnCr22 achieved the nanoscale martensite laths which enhanced the lath refinement strengthening effect. In addition, online quenching can be realized using the waste heat after rolling the field pipes, which simplified the heat treatment process, conducive to energy saving and emission reduction. Furthermore, some short rod-shaped carbides paralleled with each other can be found inside martensite laths, as shown in figures 4(d) and (e). According to figure 4(f), it was measured that the size of carbides at \(30^{\circ}\)C s\(^{-1}\) was mainly 100–150 nm, accounting for 38%, and the average size was calculated to be 123 nm. As for quenching, the size of carbides was mainly 50–100 nm which accounted for 45%, and the average size was 101 nm, finer than that at \(30^{\circ}\)C s\(^{-1}\) by 18%. This is because quenching restrained the growth of carbides in a short period of time. Thus, quenching of TMCP on 30MnCr22 obtained a uniform distribution of nanoscale carbides, having an obvious precipitation strengthening effect.

Furthermore, according to the corresponding energy spectrum shown in figure 4(g), it can be found that Cr element existed in this carbide marked by red circle in figure 4(e). And from the selected area electron diffraction (SAED) pattern along the [562] zone axis of the carbide illustrated in figure 4(h), this carbide has an orthogonal structure which is just consistent with that of \(\theta\)-Fe\(_3\)C. Therefore, this carbide is confirmed to be \(\theta\)-(Fe, Cr\(_3\))\(_2\)C. In fact, carbon atoms or other alloy elements still have a certain diffusion ability to segregate on dislocations or other crystal defects, and carbides can precipitate dispersely after quenching, which is called the self-tempering phenomenon of martensite [21]. And the deformation strengthening effect of reducing is also helpful to diffusion and precipitation of carbides by supplying more precipitation sites. What’s more, quenching makes carbon contents in martensite laths over-saturated, also conducive to precipitating carbides. By in-depth observation, it can be noted from figure 4(i) that some dislocation grids distributed near the interfaces of martensite laths. This is mainly because the dislocations generated during martensite transformation and kept slipping and tangling with the transformation proceeding in order to provide the plastic coordination needed for the continuous formation of martensite laths.

In theory, the quantitative density of dislocation varied at different cooling rates and its contribution to the strength can be determined by XRD tests for characterizing the dislocation strengthening effect. Hence, the XRD
diffraction patterns of 30MnCr22 at 1 °Cs−1, 30 °Cs−1 and quenching were all obtained through analyzing XRD tests using MDI Jade5.0 software, as shown in figure 5.

It can be seen from the XRD diffraction patterns in figure 5(a) that the planes such as (310), (220), (211), (200) and (110) are the main focuses of analysis due to more obvious diffraction peaks with higher intensities. Notably, the diffraction peak of plane (110) is taller and narrower than that of the others, and its intensity also much larger than the others, indicating that the grains are preferentially oriented in this direction. Additionally, the intensity of diffraction peak becomes lower and the half width slightly wider with the increase of cooling rate, which revealed the occurrence of grain refinement.

Based on figure 5(a), the half width of each diffraction peak was obtained using Jade software, and the corresponding dislocation density at each cooling rate can be calculated by the following equation [22]:

\[ \rho = \frac{D^2}{2 \ln 2 \pi b^2} \]

Where, \(D\) is the half width of each diffraction peak, \(b\) is the Burger vector.

According to equation (3), the dislocation density histogram of 30MnCr22 at different cooling rates were also statistically regressed and illustrated in figure 5(b). And the total dislocation density was calculated to be \(0.55 \times 10^8 \text{ mm}^{-2}\), \(6.07 \times 10^8 \text{ mm}^{-2}\) and \(8.38 \times 10^8 \text{ mm}^{-2}\) at 1 °Cs−1, 30 °Cs−1 and quenching, respectively. Obviously, it can be seen that the total dislocation density increased with the increase of cooling rate and reached the maximum value at quenching, meaning the dislocation strengthening effect became stronger with the increase of cooling rate, especially at quenching, could get the strongest dislocation strengthening effect. This is also consistent with the variation of hardness shown in table 3.

In more detail, the increased strength \(\sigma_p\) caused by the dislocation strengthening can also be quantitatively calculated by the following equation:

\[ \sigma_p = M_o G b \rho^{1/2} \]

Where, \(M_o\) is the Taylor factor which is 0.5; \(\alpha\) is a constant which is 0.24; as for martensitic steel, \(G\) is the shear modulus which is \(7.9 \times 10^4 \text{ MPa}\); \(b\) is the Burger vector which is \(2.5 \times 10^{-7} \text{ mm}\). It can be calculated from

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Figure 4. TEM microstructural characteristics at 30 °Cs−1 and quenching of TMCP: (a) martensite laths at 30 °Cs−1; (b) martensite laths at quenching; (c) size distribution histogram of martensite laths; (d) precipitates at 30 °Cs−1; (e) precipitates at quenching; (f) size distribution histogram of precipitates; (g) energy spectrum on the precipitate marked by red circle in figure (e); (h) selected area electron diffraction (SAED) pattern along the [562] zone axis of the precipitate; (i) interaction between martensite laths, dislocations and \(\theta\)-(Fe, Cr)\(_2\)C.
equation (4) that the increased strength caused by the dislocation strengthening effect at 1 °C s⁻¹, 30 °C s⁻¹ and quenching is 55.6 MPa, 184.6 MPa and 217.0 MPa, respectively, indicating quenching of TMCP had an obvious dislocation strengthening effect.

3.4. Discussion

Of the above, it can be concluded that recrystallization controlled rolling and quenching were the two keys for realizing TMCP of 30MnCr22. On the one hand, based on the results of recrystallization behavior and the microstructural heritability of TMCP, the original austenite grains were significantly refined from 200 μm to 60.3 μm, as shown in figures 4(a) and (b). This is because the large deformation of piercing, a total strain of about −1.3 shown in figure 1(a), promoted sufficient dynamic recrystallization in piercing. Then the grains were further refined to 23.8 μm due to static recrystallization of the previous passes in continuous rolling, as proved by figures 1(b) and (e). This further confirmed the obvious refinement effect of recrystallization controlled rolling on austenite grains. Based on this kind of fine recrystallized austenite microstructure inherited from piercing and continuous rolling, seven-pass reducing deformation of TMCP greatly enhanced the deformation strengthening effect of austenite by the strains accumulation in the non-recrystallization zone, as verified by figure 1(c). Hence, a large number of defects such as deformation bands and dislocations generated in strengthened austenite grains, contributing to refine the microstructure after martensite transformation. On the other hand, in order to inhibit softening and coarsening, quenching of TMCP was adopted to ensure this fine and strengthened austenite containing a lot of high-energy ‘defects’ maintained until martensite transformation. In this way, the nucleus can be stimulated due to more nucleation sites resulted from this inherited microstructure, as well as more deformed storage energy, which promoted the subsequent transformation effectively. Additionally, high lattice distortion energy caused by a large stress and strain field generated during martensite transformation at quenching also provided a strong driving force for the formation of martensite laths. Finally, as displayed in figure 4(b), the average width of martensite laths can be greatly refined to 170 nm due to realizing the microstructural heritability of fine and strengthened austenite by quenching, which had a significant lath refinement strengthening effect.

Furthermore, the effects of quenching can also explain the microstructural characteristics of 30MnCr22 in this research. As mentioned above, the stimulated nucleation sites by quenching were helpful to form many fine sub-grains with different orientations, i.e. the blocks of martensite displayed in figure 4(h), which had the apparent effects of sub-grain strengthening. Simultaneously, abundant and refined substructures especially high density dislocations also generated in large quantities by quenching, as shown in figure 4(i). And the movement of dislocations was hindered by tangle with each other, as well as by the grain boundaries, having a significant dislocation strengthening effect. As calculated above, the dislocation density of 8.38 × 10⁹ mm⁻² obtained by quenching increased the strength by 217.0 MPa.

Further, it was reported that the interfaces of martensite laths were the main obstacles to the movement of dislocations [23]. Accordingly, the refinement of martensite laths caused by quenching also made these entangled dislocations more likely accumulate and proliferate, conducive to reinforcing the dislocation strengthening effect greatly. Conversely, the continuous growth of martensite laths was blocked by the entanglement of high-density dislocations, leading to the refinement of martensite laths further. As a result, quenching enhanced the interaction between martensite laths and dislocations due to refining martensite laths and increasing the dislocation density, as proved by figure 4(i), which improved the hardness and strength of 30MnCr22 significantly. This is confirmed to be the main strengthening mechanism of TMCP on 30MnCr22.
Additionally, the great undercooling of quenching also refined \( \theta-(Fe,Cr)_3C \) to the average size of 101 nm and made these carbides more evenly distributed, which obtained a significant precipitation strengthening effect, as displayed in figure 3(e). In such a case, \( \theta-(Fe,Cr)_3C \) was easier to be captured by high-density dislocations, which stabilized the martensite matrix and strengthened the pinning effect of \( \theta-(Fe,Cr)_3C \) on dislocations greatly, as also noted in figure 3(i). And this is considered to be another strengthening mechanism of TMCP on 30MnCr22.

In summary, the key point of TMCP on 30MnCr22 is to realize the microstructural heritability by quenching, and the premise is that fine and strengthened austenite grains can be obtained by controlled rolling. Consequently, a kind of complicated multi-layer microstructural characteristics in 30MnCr22 containing fine sub-grains, ultra-fine martensite laths, high-density dislocations and nanoscale \( \theta-(Fe,Cr)_3C \) were obtained by TMCP, realizing the synergistic strengthening effects of fine grain strengthening, laths refinement strengthening, dislocation strengthening and precipitation strengthening simultaneously. And the main strengthening mechanisms of TMCP on 30MnCr22 are explored to be that quenching enforced the interaction between martensite laths and dislocations, as well as the entanglement between \( \theta-(Fe,Cr)_3C \) and dislocations.

4. Conclusions

This article systematically studied TMCP of 30MnCr22 oil well pipes based on the recrystallization behavior and microstructural heritability by experimental simulation, the following results were drawn:

(1) The large strain in piercing, as well as continuous rolling promoted recrystallization which significantly refined the original austenite grains from 200 \( \mu \)m to 23.8 \( \mu \)m, and the reducing strains accumulated in the non-recrystallization zone greatly strengthened the austenite grains. Based on this fine and strengthened austenite, quenching of TMCP achieved the best refinement of martensite laths by realized the microstructural heritability.

(2) A kind of complicated multi-layer microstructure in 30MnCr22 consisted of fine sub-grains, ultra-fine martensite laths with the average width of 170 nm, dislocations with the high-density of \( 8.38 \times 10^9 \) \( \text{mm}^{-2} \) and nanoscale carbides with the average size of 101 nm can be achieved by TMCP. This finding experimentally confirmed the synergistic strengthening effects of TMCP on fine grain strengthening, laths refinement strengthening, dislocation strengthening and precipitation strengthening simultaneously. Further, the synergistic strengthening mechanisms are mainly attributed to the following causes: quenching enforced the interaction between martensite laths and dislocations, as well as the entanglement between \( \theta-(Fe,Cr)_3C \) and dislocations.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Methods

TMCP of 30MnCr22 oil well pipes was investigated by thermal-mechanical simulation technology. And the microstructure heritability of TMCP was tracked by OM and SEM. Further, Methods of characterizing the dislocation strengthening effect adapt XRD analysis with the calculation. Finally the microstructural characteristics obtained by TMCP of 30MnCr22 oil well pipes were in-depth investigated using TEM.

Competing interests

No conflict of interest exits in the submission of this manuscript, and manuscript is approved by all authors for publication. I would like to declare on behalf of my co-authors that the work described was original research that has not been published previously, and not under consideration for publication elsewhere, in whole or in part.
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