Optimization of Thermal and Deformation Effect during Plastic Deformation and Thermal Treatment of Hot Rolled Heavy Wall Pipes Made of Medium Carbon Martensitic Steel

A I Ziza¹, V V Tsukanov¹

¹ Kurchatov Institute Research Centre – Prometey Central Research Institute of Structural Materials, Shpalernaya Street 49, Saint Petersburg, RU-191015, Russia

E-mail: oknir@crism.ru

Abstract. The method of stress relaxation in isothermal aging after deformation is employed to determine recrystallization temperature range ($T_{rec}$) for 38HN3MFA (35HN3MFA) steel grade. The paper shows that billet final deformation at the temperature lower than $T_{rec}$ followed by annealing including heating and aging at $T_{rec}$ enables austenite grain refinement.

1. Introduction

Improved ductility, impact strength and failure resistance for a specified level of material strength is obtained by increasing degree of structural fineness [1]. Size of transformed structure elements is directly related to initial austenite grain. According to [2] martensite can form three packages within prior-austenite grain boundaries, the packages further divide into blocks and sub-blocks. Before $\gamma \rightarrow \alpha$ transformation starts recrystallization of deformed $\gamma$ phase is used for austenite grain refinement [3]. However, martensitic steels show a tendency to structural heredity representing a phenomenon when martensite blocks are formed within prior austenite grain, which is recovered when being reheating [4]. Structural heredity is prevented by using recrystallization annealing [5], which includes heating up to austenite recrystallization temperature and aging at this temperature.

In this case determination of precise recrystallization temperature range plays a key role for structure refinement. State-of-the-art testing equipment allows determining $T_{rec}$ by using stress relaxation method [6].

Besides, when material deformation is performed at the temperature lower than $T_{rec}$, austenite structure can split into disordered fragments [7]. Boundaries of such fragments as well as grain boundaries are locations of $\alpha$ phase primary nucleation [8]. Besides, martensite can partially inherit austenite disordered structure.

Hot rolled heavy wall pipes with a wall thickness of 65 mm (before machining) are widely used for manufacturing high pressure cylinders with a working pressure of 39.2 MPa. These pipes are made of high strength medium carbon martensitic steel of 38HN3MFA and 35HN3MFA grades, their chemical composition is shown in Table 1.
Variations in mechanical properties of material and functional reliability of cylinders can be improved by refining cylinder fabrication practices and particularly billet production process. This is because failure occurs according to “weak link” principle, fracture growth along fine grains boundaries requires continuous change of its direction, therefore fracture growth along coarse grains boundaries is more energy-efficient. Structural heredity can manifest in fracture growing along the boundaries of prior austenite grains, despite the actual refined grain size, and orientation [10]. Impact strength is highly refined homogeneous structure. Structural heredity results in the fact that coarse grain with austenite resistance. Recrystallization annealing includes heating followed by aging at the recrystallization temperature ($T_{\text{rec}}$). This thermal treatment contributes to emerging of new finer austenite grains. In this case preliminary deformation and phase hardening play a crucial role for the process, as they are main driving forces for recrystallization. Furthermore, material recrystallization temperature shall be defined for grain refinement.”

Table 1. Chemical composition of 38HN3MFA and 35HN3MFA grades.

| Steel grade | C  | Mn  | Si  | Cr  | Ni  | Mo  | V  | P  | S  |
|-------------|----|-----|-----|-----|-----|-----|----|----|----|
| 38HN3MFA    | 0.33 | 0.25 | 0.17 | 1.20 | 3.00 | 0.35 | 0.10 | 0.025 | ≤ 0.025 |
| 35HN3MFA    | 0.34 | 0.20 | 0.17 | 1.10 | 2.75 | 0.30 | 0.08 | 0.025 | ≤ 0.020 |

Pipe billet for a high pressure cylinder is made by piercing an ingot and its further consistent drawing over the mandrel gradually reducing the diameter of rings and mandrel. After the billet undergoes preliminary thermal and mechanical treatment, necks are forged. After final thermal treatment (quenching + high tempering) the structure of cylinder steel is tempered martensite with blocks size corresponding to numbers 7–8 according to GOST 5639. Mechanical properties of steel for cylinders shall meet the following requirements: tensile strength $\sigma_{\text{u}} \geq 1128$ MPa, yield stress $\sigma_{0.2} \geq 981$ MPa, elongation $\delta_5 \geq 11 \%$, impact strength $KCU_{\text{20}} \geq 68.7$ J/cm$^2$.

Selection of preliminary thermal treatment

4.2. Selection of preliminary thermal treatment

38HN3MFA and 35HN3MFA grades tend to structural heredity, which makes it difficult to obtain highly refined homogeneous structure. Structural heredity results in the fact that coarse grain with a martensite or bainite ordered structure will not become finer when heated anew higher than $A_c_3$ point, but will replicate the initial one in terms of size and orientation [10]. Impact strength is a structure-responsive feature and heavily depends on the size of structural constituents. This is because failure occurs according to “weak link” principle, fracture growth along fine grains boundaries requires continuous change of its direction, therefore fracture growth along coarse grains boundaries is more energy-efficient. Structural heredity can manifest in fracture growing along the boundaries of prior austenite grains, despite the actual refined grain [10].

Previous works [5] show that recrystallization annealing is practical for grain refinement of Cr-3Ni-Mo-V steels with high austenite resistance. Recrystallization annealing includes heating followed by aging at the recrystallization temperature ($T_{\text{rec}}$). This thermal treatment contributes to emerging of new finer austenite grains. In this case preliminary deformation and phase hardening play a crucial role for the process, as they are main driving forces for recrystallization. Therefore, material recrystallization temperature shall be defined for grain refinement.

2. Selection of preliminary thermal treatment

The second reason for defining ($T_{\text{rec}}$) is related to control of deformation finishing temperature. During plastic deformation at a temperature lower than $T_{\text{rec}}$ austenite hardening occurs and substructure appears, that is deformation at a temperature lower than $T_{\text{rec}}$ contributes to formation of austenite subgrain structure caused by deformation. It is inherited by transformed austenite structure, which causes formation of optimal structure – lath martensite. At the same time last pass during billet
deformation at a temperature higher than $T_{rec}$ can cause partial grain growth due to secondary recrystallization running at too high temperature of billet heating. In this case the driving force of secondary recrystallization is surface energy of grains, as metal tends to more balanced state, which goes along with reduction of total length of grains boundaries, that is grain coarsening.

3. Recrystallization temperature determination

Gleeble 3800 thermomechanical simulator was used to determine ($T_{rec}$). Cylindrical samples of Ø10×15 mm were heated up to 1220 ºС and cooled down to the supposed $T_{rec}$ (860, 880, 940, 960 ºС) – deformation temperature, at which uniaxial compression followed by ageing at this temperature was performed. The method of stress relaxation [11] was employed to observe austenite recrystallization kinetics.

![Figure 1](image.png)

Figure 1. Diagrams of stress relaxation after deformation completion at temperatures of 860, 880, 940, 960 ºС.

Figure 1 shows stress relaxation diagrams for different temperatures demonstrating dependence of samples softening on aging, typical for running of the recrystallization process. Apparently, recrystallization doesn't stop at a temperature of 860 ºС during time interval selected. Temperature of 860 ºС can be considered as a lower level of recrystallization temperature range. Recrystallization stops as quick as possible during isothermal aging at temperatures of 940 and 960 ºС. Therefore 940 ± 20 ºС range can be considered as the desired $T_{rec}$ range.

4. Microstructure examination

Gleeble 3800 thermomechanical simulator was used to simulate deformation patterns with a temperature of last pass lower than ($T_{rec}$) range at 880 ºС and higher than ($T_{rec}$) range at 980 ºС and subsequent preliminary (annealing at 940 ºС and high tempering at 690 ºС) and final (quenching at 880 ºС and double tempering at 610 ºС) thermal treatment. Samples were etched under vacuum according to the procedure [12] to find out austenite grain size and assess efficiency of measures taken to have finer structure blocks. Figure 2 shows samples microstructure. When deformation temperature of the final pass exceeds ($T_{rec}$) range, an average austenite grain size corresponds to number 9 according to GOST 5639 (Figure 2 (a)).

An average austenite grain size of the sample deformed with a final pass temperature exceeding ($T_{rec}$) range corresponds to number 9 according to GOST 5639 (Figure 2 (a)). When deformation temperature of the final pass is lower than ($T_{rec}$) range, an average austenite grain size corresponds to number 10 according to GOST 5639 (Figure 2 (b)).
However, it should be noted that, microstructures were compared after simulation of full heat treatment cycle to obtain representative values against earlier test results of high pressure cylinder material.

![Microstructure Images](image1.jpg)  

**Figure 2** Sample material microstructure after deformation and thermal treatment:
(a) final deformation at 880 °C;  
(b) final deformation at 980 °C, × 200.

For further assessment of grain refinement efficiency, the samples were etched under vacuum after having been cut out of high pressure cylinders fabricated both under old and improved technology, according to the following above-mentioned recommendations: Final stage of billet deformation at a temperature lower than ($T_{rec}$) range followed by annealing at a temperature within ($T_{rec}$) range. Figure 3 shows samples microstructure. Austenite grain size of material for cylinders made under old technology corresponds to numbers 7–8 according to GOST 5639 (Figure 3 (a)). In this case maximum grain size will reach 88.3 μm. Austenite grain size of material for cylinders made under improved technology corresponds to numbers 9–10 according to GOST 5639 (Figure 3 (b)). In this way, the structure can be considered more homogeneous with a maximum grain size of 43.7 μm.
4. Conclusions
1. State-of-the-art research simulators provide manufacturing process modeling, which enables to obtain metal structure like this obtained in production environment.
2. Final deformation at the temperature lower than recrystallization temperature followed by annealing including heating and aging within recrystallization temperature range contributes to refinement of structural elements for 38HN3MFA and 35HN3MFA steel billets.

References
[1] Moroz L S 1984 Mechanics and Physics of Material Deformation and Failure (Leningrad: Mashinostroyeniye) p 225
[2] Morito S, Saito H, Ogawa T, Furuhama T and Maki T 2005 ISIJ International vol 45 1 91–94
[3] Miao C L, Shang C J, Zhang G D and Subramanian S V 2010 Mater. Sci. Eng. A 527, 4985
[4] Nakada N, Tsuchiyama T, Takaki S and Hashizume S 2007 ISIJ International vol 47 10 1527–32
[5] Tsukanov V V and Ziza A I 2015 Issues of Materials Science 2(82) 9–16
[6] Perttula J S and Karjalainen L P 1998 Materials Science Technologies 14 626-30
[7] Kodzhaspirov G Ye, Rybin V V and Apostolopoulos H 2007 Metal Science and Heat Treatment 1 (619) 30–34
[8] Quispe A, Medina S F, Gomez M and Chaves J I 2007 Materials Science and Engineering A 447 11–18
[9] Zakharenko Yu V, Ziza A I, Ilyin A V and Tsukanov V V 2017 Metalworking 4 (100) 29–36
[10] Schastlivtsev V M, Zeldovich V I, Mirzayev D A and others 2008 Development of V.D. Sadowsky ideas. Collected works ed M A Filippov and Yu V Kaletina (Yekaterinburg: Institute of Metals Physics, Ural Branch of Russian Academy of Science) p 409
[11] Zisman A A, Soshina T V and Khlusova Ye I 2012 Notes on Materials vol 2 1 3–8
[12] Soshina T V, Zisman A A and Khlusova Ye I 2013 Metallurgist 2 63–70