Inversion of Nitrogen Redistribution in Austenitic Steel by Severe Plastic Deformation

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Abstract—Using the Mössbauer spectroscopy and transmission electron microscopy (TEM) methods, the temperature boundary of a strain-induced transformation with the inversion of the direction of nitrogen redistribution is determined in the structure of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ austenitic steel. Deformation by high pressure torsion in Bridgman anvils below the temperature limit (298 K) leads to an increase in the amount of nitrogen in the interstitial solid solution and deformation above the limit (373 K) leads to a decrease in this value. An increase in the deformation temperature leads to the complete dissolution of the products of cellular decomposition and the formation of submicrocrystalline austenite with secondary nanocrystalline nitrides. Changes in the direction of nitrogen redistribution are explained by the competition between the mechanisms of relaxation of the structure along the paths of dispersion, dissolution of nitrides by dislocation, and decomposition of a solid solution supersaturated with nitrogen.

Keywords: high-nitrogen austenitic steels, severe plastic deformation, Mössbauer spectroscopy

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INTRODUCTION

High-nitrogen chromium–manganese austenitic steels have a number of favorable physicochemical properties (corrosion resistance, tribological characteristics, etc.), which make it possible to use these steels as new materials in mechanical engineering [1–4]. The mechanical properties of nitrogen steels are largely determined by structural phase transformations under external actions [5–7]. First of all, this concerns transformations under conditions of strong effects related to severe plastic deformation (SPD), such as the shock and friction effects, extrusion, equal channel angular pressing (ECAP), high pressure torsion (HPT), etc. In [8, 9], attention was drawn to the processes of dissolution of CrN (Cr$_2$N) nitrides in the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ and FeCr$_{22}$Mn$_{1.4}$N$_{1.24}$ aging high-nitrogen austenitic steels, which are induced by sliding friction and HPT. In particular, the authors attribute the improvement of wear resistance characteristics in the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ austenitic steel to an increase in the strengthening due to the solid-solution mechanism during friction [8]. The high diffusion mobility of nitrogen can promote relaxation processes associated with dissolution of nitrides and the formation of secondary nitrides under SPD conditions. Transmission electron microscopy (TEM) data on the formation of nanocrystalline nitrides in the FeCr$_{22}$Mn$_{1.4}$N$_{1.24}$ steel deformed by HPT at room temperature can serve as an indirect confirmation of the formation of secondary nitrides in the austenite matrix [9].

Studies devoted to SPD with use of ball mills and shear in rotating Bridgman anvils showed that a change in the temperature of large plastic deformation in a relatively small range drastically changes the direction of structural phase transformations from disordering and dissolution of particles at ambient temperatures (conditionally cold deformation) to accelerated ordering and the formation of secondary phases at temperatures above room temperature (conditionally warm deformation). The processes of ordering and the formation of secondary phases during grinding in ball mills are caused by high local temperatures in the shock effect zones. Theoretically, similar dynamic effects at $T_{\text{impact}} \leq T_{\text{melting}}$ are substantiated by the appearance of additional diffusion relaxation channels of high mechanical energy under SPD [10–12].

The aim of this study was to analyze the effect of the SPD temperature on the structural phase transitions in the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ aging high-nitrogen austenitic steel.
We investigated the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ high-nitrogen chromium–manganese austenitic steel (0.05 wt % C, 22.1 wt % Mn, 17.9 wt % Cr, 0.83 wt % N, and the rest Fe) prepared using the casting technology with nitrogen backpressure [2, 13]. Steel samples were homogenized at 1423 K, subjected to hot forging, and then quenched from 1423 K in water. Some of the quenched samples were aged at 1073 K for 2 and 30 h. To perform SPD by the HPT method in rotating Bridgman anvils, 0.3 mm thick plates were cut off from rods with a cross section of $10 \times 10$ mm$^2$ by the electrospark method and thinned by mechanical grinding to 0.25 mm. Steel in the austenitic aged state was deformed at temperatures of 298, 373, and 573 K by the techniques described in [14]. At the selected temperature, the samples were loaded with a pressure of 8.0 GPa and sheared by rotating the lower anvil. After shearing, the sample was unloaded and returned to room temperature. The sample after HPT had the shape of a disk with a thickness of 0.15 mm and a diameter of 7 mm.

True deformation under HPT was estimated by the following formula [15]:

\[
e = \ln \left( \frac{h_0}{h_f} \right) + \ln \left( 1 + \left( \frac{\phi R_i}{h_f} \right)^2 \right)^{1/2},
\]

where $h_0$ and $h_f$ are the thicknesses of the samples before and after deformation, respectively; \(\phi = n \times 2\pi\) is the angle of rotation of the anvil ($n$ is the number of revolutions); $R_i$ is the distance from the center of the specimen to the region in which the structure was studied (half of the sample radius). The deformation was carried out at a rate of $\omega = 0.3$ rpm. The strain value was $e = 5.9$ ($n = 3$ revolutions).

The Mössbauer spectra of absorption of 14.4-keV $\gamma$ quanta by $^{57}$Fe nuclei were recorded at room temperature under a constant acceleration mode with a $^{57}$Co(Rh) source. For Mössbauer measurements, the samples were thinned to a thickness of 20 $\mu$m. The entire area of the sample was used as a spot for the passage of $\gamma$ quanta. The standard calibrator was $\alpha$-Fe foil. The Mössbauer spectra were processed using the MS Tools software package [16]. The calculation included the reconstruction of the distribution of the centers of gravity for the singlet (instrumental) lines on the scale of Doppler velocities $p(V)$, which improve the spectrum resolution. Next, the $p(V)$ distribution was used, along with a priori data, to simulate and approximate the spectra by the sum of several subspectra corresponding to different nonequivalent environments of the Mössbauer probe, i.e., $^{57}$Fe atoms.

The same samples were studied by TEM using the method of thin foils for transmission on a JEM-200CX microscope.

**RESULTS**

**TEM Analysis of the Results of Deformation by HPT of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$Fe Steel**

Figure 1a shows the structure of the initial quenched steel without visible nitride precipitates. As a result of HPT at room temperature, a submicrocrystalline state is formed in the quenched steel (Fig. 1b).
The figure also shows the ring microdiffraction pattern containing reflections from highly misoriented austenite grains. The size of the matrix fragments (grains and subgrains) is 50–80 nm.

The aging of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ steel for 30 h at 1073 K gives rise to almost complete (about 90%) decomposition of the solid solution with the formation of a pearlite-like cellular decomposition structure that is composed of alternating plates of austenite and chromium nitride Cr$_2$N (Fig. 2a) [8]. The HPT deformation at room temperature leads to refinement of the austenite matrix structure and partial dissolution of nitrides (Fig. 2b).

Under warm (573 K) deformation, the structure of the quenched and aged steel sample is completely transformed in comparison with the initial structure. Lamellar nitrides are not preserved in the structure. Dispersed nitride and austenite crystallites are formed. There are reflections near the first diffraction ring of reflections from the austenite matrix, which can be attributed to nitrides with nonstoichiometric composition (Cr$_x$N, see Figs. 1c and 2c). Not only misoriented matrix fragments appear in the dark-field image in the complex (111)$_\gamma$ + (200)$_\gamma$ + Cr$_2$N reflection, but also dispersed precipitates about 2 nm in size. Nanocrystalline nitrides are presumably secondary phases that are formed as a result of the decomposition of a solid solution based on the fcc phase supersaturated with nitrogen. The decomposition of a γ solid solution supersaturated with nitrogen under strain at different temperatures was analyzed by the Mössbauer spectroscopy method, and the results are discussed below.

**Mössbauer Analysis of the Nitrogen Content in Austenite of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ Steel**

Changes in the austenite composition detected after annealing, dry sliding friction, and HPT at room temperature have been studied earlier [8]. The Mössbauer spectrum of the hardened steel sample has the shape of a broadened asymmetric singlet (see Fig. 3a). The broadening is caused by nonequivalent surrounding of the resonant iron atom by substitutional (Mn and Cr) and interstitial (N) impurity atoms. To improve the resolution of the spectra, we used the procedure for reconstructing the $p(V)$ distribution. It is clearly seen that the $p(V)$ distribution reveals an asymmetric structure of the spectrum when approximated by Gaussian $p(V)$ shapes and is a superposition of the D(0) + D(1) + D(2) components (see Figs. 3 and 4). The use of the $p(V)$ calculation data and a priori data on the spectra of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ stainless steel [17, 18] and a solid solution of nitrogen in iron with the fcc crystal lattice [19, 20] allows us to propose a model of the Mössbauer spectrum of the steel under study with the parameters indicated in Tables 1 and 2.

The D(0) doublet is attributed to the electric field gradient on the $^{57}$Fe nucleus from the environment with substitutional impurities (Mn and Cr), and its hyperfine parameters (isomeric and quadrupole shifts $I_s$ and $Q_s/2$, respectively) are close to the values typical
of stainless steel [17, 18]. The D(1) and D(2) doublets have parameters similar to those of the subspectra caused by the insertion of one and two (in the dumbbell configuration) nitrogen atoms into the octahedral interstices closest to the iron atoms [19, 20] (see Fig. 4 and Tables 1 and 2).

The proposed model of the hyperfine structure of the steel under study is confirmed by changes in the $p(V)$ distribution and the partial contribution of the D(1) and D(2) components of the spectrum after aging of the steel (see Fig. 3 and Table 1). The release of nitrogen from the interstitial sites of the fcc solid solution into the CrN and Cr$_2$N nitrides during aging leads to a decrease in the intensity of the D(1) and D(2) components. According to X-ray diffraction (XRD) data, the lattice period of austenite decreases from 0.3630 to 0.3614 nm as a result of aging at 1073 K for 30 h.

Table 1. Parameters of the Mössbauer subspectra of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ steel after quenching and aging at 1073 K

| Treatment                        | Subspectra and their parameters |
|----------------------------------|---------------------------------|
|                                 | D(0)                            | D(1)                            | D(2)                            |
|                                 | $I_s$, mm/s | $Q_s/2$, mm/s | $G_{1/2}$, mm/s | *$\Delta D(0)$, % | $I_s$, mm/s | $Q_s/2$, mm/s | $G_{1/2}$, mm/s | *$\Delta D(1)$, % | $I_s$, mm/s | $Q_s/2$, mm/s | $G_{1/2}$, mm/s | *$\Delta D(2)$, % |
| Quenching for 30 min at 1373 K   | −0.11 | 0.07 | 0.29 | 88 | −0.05 | 0.20 | 0.29 | 11 | 0.01 | 0.35 | 0.30 | 1 |
| Aging for 2 h at 1073 K          | −0.11 | 0.07 | 0.29 | 91 | −0.05 | 0.20 | 0.29 | 9 | −− | −− | −− | −− |
| Aging for 30 h at 1073 K         | −0.12 | 0.06 | 0.30 | 93 | −0.05 | 0.19 | 0.29 | 7 | −− | −− | −− | −− |

* The $\Delta D(0)$, $\Delta D(1)$, and $\Delta D(2)$ values are determined by extrapolation to zero thickness of the absorber.
**Mössbauer Analysis of the Results of HPT of the Quenched and Aged FeMn\textsubscript{22}Cr\textsubscript{18}N\textsubscript{0.83} Steel at Different Temperatures**

The quantitative estimation of the nitrogen content \(x\) in the FeMn\textsubscript{22}Cr\textsubscript{18}N\textsubscript{0.83} steel was carried out under assumption of repulsive distribution (mutual repulsion) of nitrogen atoms in a solid solution on the basis of contribution of the D(1) configuration from iron atoms with one nitrogen atom in the nearest octahedral interstices, i.e., the relative integrated intensity \(S_{D(1)}\), in accordance with the following formula [19]:

\[
S_{D(1)} = 6p(1-p),
\]

where \(p = x(1-x)\) is the fraction of octahedral interstices in austenite filled with nitrogen.

To take into account the self-absorption effect, the \(S_{D(1)}\) intensity was extrapolated to the zero thickness of the absorber. For the hardened state of steel, \(x = 0.2\) (2 at \% \(N\), which is noticeably lower than the value specified during smelting (3.3 at \%). As a result of deformation of the hardened steel sample at room temperature \((n = 3\) revolutions\), the \(S_{D(1)}\) intensity increases from 11 to 17 vol \% (see Figs. 4a and 5a and Table 2).

According to formula (2), this means an increase in the nitrogen content from 2 to 3 at \%, which is close to the total amount of nitrogen in steel (3.3 at \%) (see Fig. 5a). The same tendency, namely, the growth of \(S_{D(1)}\) from 6 to 10 vol \%, is observed in the spectrum of a steel sample aged at 1073 K and deformed \((n = 3\) revolutions\) by HPT at room temperature, which corresponds to an increase in the nitrogen content from 1.1 to 1.8 at \% (see Figs. 4c and 5b). An increase in the nitrogen content in austenite of the steel sample deformed by HPT at a temperature of 298 K correlates with an increase in the lattice period of austenite to 0.3648 nm.

The tendency of the \(S_{D(1)}\) intensity of quenched and aged steel samples to increase after cold deformation changes to the opposite tendency with an increase in the deformation temperature within a relatively narrow transient temperature range. The deformation of both quenched and aged steel samples at 373 K does not lead to an increase in the \(S_{D(1)}\) intensity within the

### Table 2. Parameters of the Mössbauer subspectra of the hardened and aged FeMn\textsubscript{22}Cr\textsubscript{18}N\textsubscript{0.83} steel after HPT at 298 and 573 K

| Treatment                  | Subspectra and their parameters | D(0) | D(1) | D(2) |
|---------------------------|---------------------------------|------|------|------|
|                           | \(I_s\), mm/s | \(Q_{1/2}\), \(G_{1/2}\) | \(S_{D(0)}\)\% | \(I_s\), mm/s | \(Q_{1/2}\), \(G_{1/2}\) | \(S_{D(1)}\)\% | \(I_s\), mm/s | \(Q_{1/2}\), \(G_{1/2}\) | \(S_{D(2)}\)\% |
| Quenching for 30 min at 1373 K | –0.11 0.07 0.29 88 | –0.05 0.20 0.29 11 | –0.01 0.35 0.30 1 |
| HPT at 298 K after quenching | –0.10 0.07 0.30 79 | –0.04 0.19 0.30 17 | 0.02 0.34 0.30 2 |
| HPT at 573 K after quenching | –0.11 0.06 0.30 92 | –0.05 0.20 0.30 7 | 0.00 0.35 0.30 0.5 |
| Aging for 30 h at 1073 K    | –0.12 0.06 0.30 93 | –0.05 0.19 0.29 7 | – – – – |
| HPT at 298 K after aging    | –0.11 0.07 0.30 90 | –0.03 0.20 0.29 10 | – – – – |
| HPT at 573 K after aging    | –0.11 0.06 0.30 93 | –0.05 0.19 0.29 6 | – – – – |

*The \(S_{D(0)}, S_{D(1)}, \) and \(S_{D(2)}\) values are determined by extrapolation to zero thickness of the absorber.

![Fig. 5. Dependences of the nitrogen concentration \(C_N\) in austenite of the FeMn\textsubscript{22}Cr\textsubscript{18}N\textsubscript{0.83} steel on the HPT temperature: (a) initial quenching in water from 1423 K; (b) initial aging for 30 h at 1073 K. The dashed lines indicate the initial nitrogen content in solid solutions of the (a) hardened and (b) aged steels.](image-url)
experimental temperature to 573 K radically changes the direction of $S_{\text{D(1)}}$ change. As it follows from the results of calculating the spectrum of a quenched sample deformed by HPT at 573 K, $S_{\text{D(1)}}$ decreases from 11 to 7 vol %, which is close to the results obtained after aging the steel sample for 30 h at 1073 K. The same trend, namely, a decrease in the $S_{\text{D(1)}}$ intensity from 7 to 6 vol %, is also observed upon an increase in the deformation temperature of the aged sample.

**DISCUSSION**

The Mössbauer spectroscopic data on the increase in the intensity of the D(1) and D(2) doublets after cold (298 K) deformation of quenched and aged steel samples by HPT reveal an increase in the number of nitrogen atoms in the state of implantation into the fcc matrix of the steel. This result was confirmed by XRD data and demonstrated earlier in experiments on the friction and HPT effects on the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ steel under study [8] and on the FeCr$_{22}$Mn$_{1.4}$N$_{1.24}$ steel at room temperature [9].

The processes associated with dissolution of dispersed particles of the secondary phases (intermetallic compounds, carbides, borides, oxides, and nitrides) due to large plastic deformation have been observed earlier [21, 22]. For the hardened state of steel, an increase in the number of nitrogen atoms in the interstices closest to iron atoms is explained by deformation disordering of the inhomogeneous nitrogen distribution developed during smelting and pretreatment [8, 9].

The change in the direction of redistribution of nitrogen in austenite to the opposite direction in a narrow transient temperature range of SPD, which is detected in the present study, means the presence of a temperature boundary of strain-induced transition from dissolution to precipitation of nitrides.

As follows from the TEM data, the temperature limit detected by the Mössbauer method—above which the nitrogen content in the austenite-based solid solution decreases—is characterized by structural transformation with complete dissolution of cellular decomposition products at 573 K and the formation of submicrocrystalline austenite with dispersed nanosized nitride precipitates (see. Figs. 1c and 2c). As follows from the Mössbauer analysis, the degree of decomposition, i.e., the amount of nitrogen in the solid solution based on the γ-phase, after deformation at 573 K both in the quenched and aged steels approaches the nitrogen content in austenite after thermal aging for 30 h at 1073 K. Hence, it can be concluded that an increase in the temperature of HPT accelerates both the processes of dissolution of cellular decomposition products and the formation of secondary nitrides on the one hand. But on the other hand, the process of formation of secondary nitrides predominates with an increase in the deformation temperature to 573 K.

The anomalous effect of the SPD temperature on the processes of mechanical alloying was demonstrated earlier on the example of strain-induced phase transformations (during rolling and HPT) of dispersed dissolution—precipitation type particles of the intermetallic γ-phase in aging Fe–Ni(Ti, Al) alloys with the fcc crystal lattice [14, 21, 22] and the CrN (Cr$_2$N) nitride particles in the ferrite phase of the FeCr$_{22}$Mn$_{1.4}$N$_{1.24}$ steel [9]. The reason for that is the mechanism of dissipation of mechanical energy during SPD, in which a large number of point defects that take part in the relaxation of the structure along the path of diffusion decomposition of supersaturated solid solutions are generated in addition to dislocation cross cutting and dissolution of particles of the secondary phases [14, 22]. This process is characterized by the acceleration of decomposition at a relatively low temperature (above 373 K) when the normal thermal annealing of the sample for a finite annealing time does not appreciably change the nitrogen content in the matrix. The accelerated kinetics of dynamic aging is confirmed by comparison of the results obtained after (i) HPT at 573 K (performed for about 10 min with three revolutions of the anvil at a rate of 0.3 rpm) and (ii) isothermal annealing (at the same temperature) of the hardened steel preliminarily deformed at room temperature. The acceleration of dissolution and formation of particles during warm deformation is associated with the formation of a developed network of grain boundaries and the saturation of the structure with dislocations and point defects. The thermodynamic condition for alloy formation in the Miedema model modified for the case of mechanical alloying [23, 24] is the formation of segregations of alloying elements in a developed network of nanograin boundaries [25]. The observed decrease in the content of nitrogen in austenite during warm deformation by HPT is associated with its outcropping to the boundaries of nanograins in the form of segregations and extremely dispersed secondary nitrides. However, it should be specially noted that the main condition for the acceleration of dynamic aging processes is the continuous generation of mobile point defects during plastic deformation. It is the continuous saturation of the structure with mobile point defects, such as the vacancy complexes, that is responsible for an increase in the mobility of substitutional elements (in this case, Cr) [26, 27] and, as a result, a decrease in the temperature of decomposition of austenite supersaturated with nitrogen with the formation of secondary nitrides [14, 28]. The increased diffusion mobility of nitrogen at lowered temperatures obviously promotes relaxation along the path of decomposition of the solid solution. The high diffusion mobility of nitrogen at low temperatures is confirmed, for example, in exper-
imments on the increase in the tetragonality of martensite freshly quenched by 80 K after heating to room temperature [3].

The formation of vacancy complexes in high concentrations close to the concentrations achieved at premelting temperatures was experimentally confirmed when studying the SPD of copper by ECAP and HPT [29, 30]. In [26], a direct relationship between the strain-induced short-range ordering and the mobility of vacancy complexes was shown for a ferritic Fe–Cr alloy. According to the Mössbauer spectroscopy and TEM data, the dynamic decomposition during warm deformation of the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ steel proceeds along the path of formation of segregations of alloying elements and highly dispersed secondary nitrides in a developed network of grain boundaries in the nanocrystalline matrix. In accordance with the concept put forward in [10], the formation of the temperature boundary of changing the direction of nitrogen redistribution in austenite is a consequence of the appearance of additional channels of relaxation of high mechanical energy at relatively low temperatures by the mechanism of accelerated diffusion. These kinds of dynamic effects caused by saturation of the structure with point defects include dynamic recrystallization, dynamic aging, ordering, etc.

CONCLUSIONS

Structural phase transitions in the FeMn$_{22}$Cr$_{18}$N$_{0.83}$ high-nitrogen chromium–manganese austenitic steel under strain in rotating Bridgman anvils at temperatures from 298 to 573 K with a true deformation of $\varepsilon \approx 5.9$ have been investigated by the Mössbauer spectroscopy and TEM methods. It is shown that SPD by the HPT method in a relatively narrow transient temperature range (from room temperature to 373 K and higher) results in a boundary of changing the direction of nitrogen redistribution in the steel structure due to an increase of its volume in a solid solution based on austenite during cold (298 K) deformation and to a corresponding decrease during warm (373–573 K) deformation. With an increase in the HPT temperature, both the processes of dissolution of the cellular decomposition products and the formation of secondary nanocrystalline nitrides are accelerated. Above the temperature limit of the inversion of nitrogen redistribution, the processes of decomposition prevail in austenite supersaturated with nitrogen. The decay processes are accelerated because of the generation of mobile vacancies responsible for the diffusion of substitutional elements (chromium) and the formation of secondary nitrides. Data on the temperature dependence of the nitrogen content in the solid solution based on austenite lead to the conclusion that the mechanical energy dissipates during HPT by a complex mechanism that combines the dislocation dissolution of nitrides and the diffusion decomposition due to generation of mobile vacancy complexes.
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