

Austenite in Nanostructured Maraging Steel

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Received May 22, 2018; revised August 27, 2018; accepted September 25, 2018

Abstract—The structural formation and properties of austenite in martensite aged (maraging) 03N18K9M5T steel were analyzed. The morphological features of austenite, determining its resilience to heating, supercooling, and plastic deformation, were established. The difference between the characteristics of residual and reverted austenite was substantiated. The contributions of phase hardening and dispersion of crystals to the yield strength of residual austenite were determined. The role of both types of austenite in the formation of steel was shown as well.

Keywords: martensite aged steel, thermal treatment, strength, structure, martensite, residual and reverted austenite, phase $\alpha \rightarrow \gamma$ transition

DOI: 10.1134/S2075113319040415

INTRODUCTION

An increase in reliability and durability as the main parameters of structural strength of martensite aged steel products is related to the presence of austenite in both single- and two-phase steels [1–3]. Austenite may be residual after quenching at various temperatures and stabilization conditions (γ_{res}), or reverted as a result of reversible $\alpha \rightarrow \gamma$ transformation (γ_{revI} ; when the temperature ranges of $\alpha \rightarrow \gamma$ transformation and aging match, then γ_{revII}) and hence possessing differences in structure and properties [1–4].

Martensitic steel 03N18K9M5T after special thermal treatment has the complex structure of α -martensite, γ_{res} , and γ_{revII} , which allows the viscosity to be increased by more than a factor of two in comparison with steel without the γ phase [1]. It is also established that the presence of residual and reverted austenite in the steel structure favors the improvement of plasticity and viscosity at retention of high strength [1]. In this respect, the present work aims at the comprehensive analysis of the complex structure, properties of composing austenite phases (γ_{res} and γ_{revII}), and their role in the increase in mechanical characteristics of 03N18K9M5T steel.

EXPERIMENTAL

Samples were 03N18K9M5T steels with the following chemical composition: 0.009% C; 18.3% nickel; 8.9% cobalt; 5.1% molybdenum; 0.66% titanium; 0.06% aluminum; 0.03% silicon; 0.03% manganese; 0.004% sulfur; 0.007% phosphorus; the rest was iron. The austenite content was determined via

X-ray diffraction on a DRON-3 diffractometer from the intensity ratio of the (110) (α phase) and (111) (γ phase) peaks. The unit cell parameter was found from the (220) peak position. The diffraction maximum profile was established from the points with a set at least of 10^6 pulses for each maximum. The plastic deformation resilience of austenite (m_γ , or the degree of deformation $\gamma \rightarrow \alpha$ transition) was evaluated using the ratio $[(\gamma - \gamma_k)/\gamma] \times 100\%$, where γ is the initial austenite content in the sample and γ_k is the austenite content after plastic deformation or in the fracture of the sample. The fine structure of steel was probed on a JEM-100 electron microscope, and the critical temperature M_n was determined on a Shevenar dilatometer. The conditions for obtaining the complex structure at all stages of its formation in accordance with the above method [1], intended for stabilizing austenite in the form of a dispersed nickel-enriched phase in the steel, were as follows: the preliminary annealing in the intercritical $A_n - A_k$ temperature range (hereinafter, the annealing); the quenching at temperatures above A_k with a short exposure (hereinafter, the “ST quenching” or simply “quenching”), and the aging.

RESULTS AND DISCUSSION

Preliminary Annealing

As known [5–7], reverted austenite I (γ_{revI}) in the martensite aged steel is obtained by the annealing in a two-phase ($\alpha + \gamma$) domain at temperatures of the $\alpha \rightarrow \gamma$ transformation (500–800°C), which is accompanied by the nickel redistribution between α and γ phases, as well as the enrichment of austenite and its

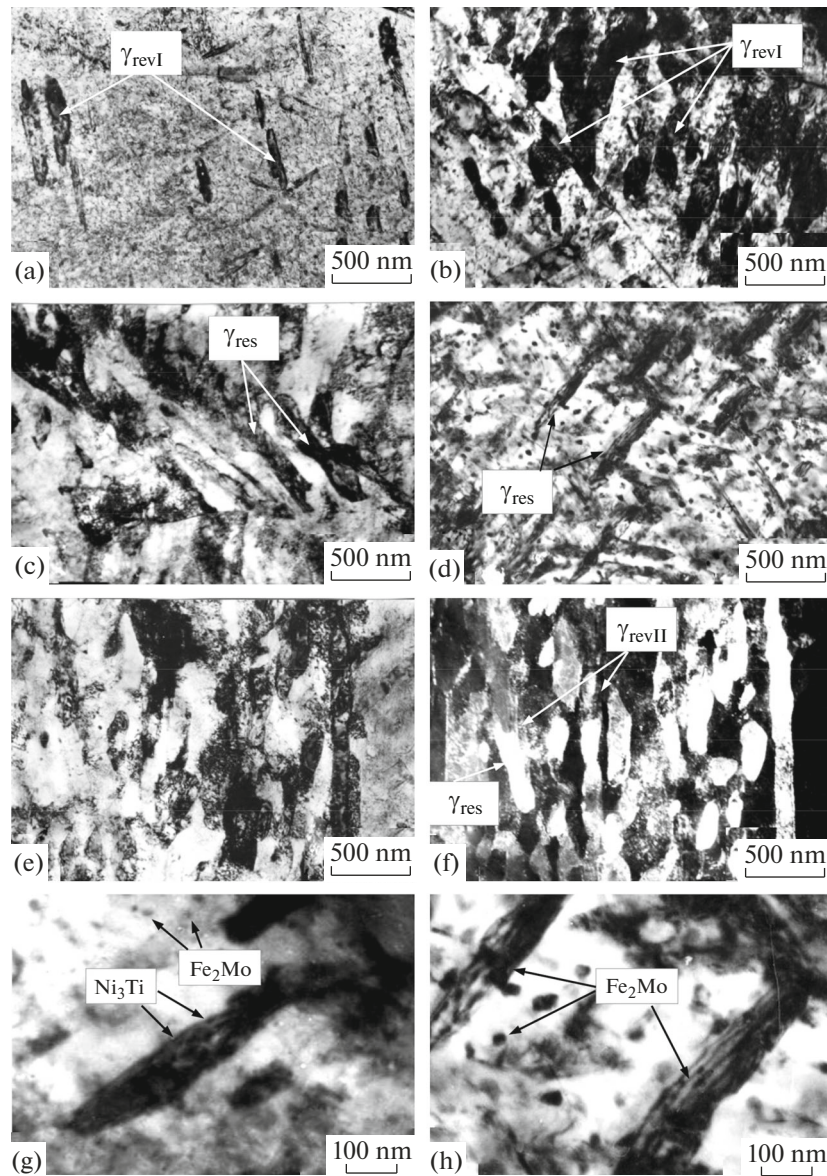


Fig. 1. Martensite–austenitic structure of 03N18K9M5T steel with various types of γ phase: (a, g) α + 23% γ_{revI} (550°C, 3 h); (b) α + 53% γ_{revI} (600°C, 1 h); (c) α + 24% γ_{res} (600°C, 1 h + 820°C, 6 min); (d, h) α + 60% γ_{res} (600°C, 1 h + 820°C, 2 min); (e, f) α + 24% γ_{res} + 15% γ_{revII} (600°C, 1 h + 820°C, 6 min + 490°C, 3 h); (f) dark-field image in the (200) reflex of γ austenite.

stabilization, whose obstacles are stated in [5, 6]. As seen in Figs. 1a and 1b, the γ_{revI} phase looks like randomly oriented rods (laths) inside the initial α phase (α -martensite) with fragmented laths at the boundaries of martensite crystals and packets. The crystal size in the γ_{revI} phase depends on the temperature and duration of the annealing (Table 1). Since the mechanism of the γ_{revI} formation is crystallographically ordered [5, 6], the γ_{revI} structure exhibits twins, dislocations, intermetallic particles with needle (Ni_3Ti) and equiaxial (Fe_2Mo) shapes, inherited from the parental α phase (see Figs. 1a, 1b, and 1g).

The amount of γ_{revI} phases is determined by the annealing temperature and varies through a maximum

which is sensitive to the isothermal exposure time at the annealing temperature (Fig. 2a). The losses of its stability after the maximum in the temperature dependence are due to the depletion of austenite in nickel in this temperature range in accordance with the Fe–Ni state diagram, which is confirmed by a decrease in the critical temperature M_n (Fig. 2b), the quantitative estimate of the unit cell parameter in the α and γ phases (Fig. 3a), and the change in the physical broadening (β) of diffraction maxima from these phases (Fig. 3b).

The study of resilience of the γ_{revI} phase to plastic deformation reveals that austenite obtained at 575°C for 1 h is stable at degrees of deformation up to 50%. At

Table 1. Mean statistical austenite crystal sizes at different stages of the formation of ($\alpha + \gamma$) structure of 03N18K9M5T steel

No.	Treatment conditions	Phase composition	Crystal size, nm	
			width	length
Reverted austenite				
1	Annealing 550°C, 3 h	$\alpha + 23\% \gamma_{\text{revI}}$	60 ± 20	330 ± 120
2	Annealing 600°C, 1 h	$\alpha + 53\% \gamma_{\text{rev}}$	90 ± 20	320 ± 90
Residual austenite				
3	Annealing 600°C, 1 h + quenching 820°C, 2 min	$\alpha + 60\% \gamma_{\text{res}}$	100 ± 30	380 ± 70
4	Annealing 600°C, 1 h + quenching 820°C, 6 min	$\alpha + 24\% \gamma_{\text{res}}$	60 ± 20	270 ± 40
Residual austenite + reverted austenite II				
5	Annealing 600°C, 1 h + quenching 820°C, 6 min + 490°C, 3 h	$\alpha + 24\% \gamma_{\text{res}} + 15\% \gamma_{\text{revII}}$	80 ± 20	340 ± 80

temperatures of 600–700°C, austenite with a smaller nickel content undergoes the $\gamma \rightarrow \alpha$ transformation already at low degrees of deformation (~5%) (Fig. 4).

Quenching

Heating of steel with a (α -martensite + γ_{revI}) structure for 2–12 min at temperatures above A_k allows one to fix a structure with residual austenite (α -martensite + γ_{res}) (Figs. 1c and 1d). Residual austenite is located in the former volumes of reverted austenite and possesses a pronouncedly fragmented structure with a high dislocation density (Fig. 1c). The structure after 2-min quenching retains a large amount of equiaxial intermetallic compounds (Figs. 1d and 1h), which are observed in both the crystals of the α phase and the crystals of the γ phase. A decrease in the amount of γ_{res} upon 12-min quenching (Fig. 5a) is due to the development of homogenization, which is confirmed by the changes in the lattice parameters (Fig. 6a). Residual austenite is found to be resistant to supercooling and decomposition upon heating [1, 7].

The well-developed dislocation structure in γ_{res} crystals is the manifestation of the phase hardening, whose degree can be estimated from the blurring of X-ray diffraction maxima (Fig. 6b). The longer the high temperature exposure, the larger is the (311) line width of austenite, which is on account of the more intense $\gamma \rightarrow \alpha$ transformation, and, consequently, the higher degree of phase hardening. A decrease in the (311) austenite peak width, as well as in the (220) martensite line at subsequent aging, is caused by the elimination of elastic microdistortions [8].

As follows from Fig. 5, the γ_{res} content depends on (a) the annealing conditions and (b) the ST quenching exposure time, reaching ~60% (see Table 1). In accordance with earlier remarks, incomplete homogenization upon quenching enables residual austenite to be maintained in former volumes of the γ_{resI} phase, which contain the highest nickel concentrations. In the case of equal volumes of reverted and residual austenite,

the shape and sizes of γ_{res} crystals are close to those of γ_{resI} crystals (see Figs. 1a–1d and lines 1–4 in Table 1). The stabilization of the γ_{res} phase in the steel with a crystal size of ~60 nm causes a decrease in the critical temperature A_n of the $\alpha \rightarrow \gamma$ transition by 60–70°C, leading to favorable conditions for the nucleation of γ_{resII} upon aging [7].

Aging

Besides the release of a dispersed Ni_3Ti reinforcing phase, the aging of the (α -martensite + γ_{res}) steel induces the formation of the γ_{resII} phase in martensite (Fig. 7, curve 2, Figs. 1e and 1f). As a rule, austenite crystals are composed of two zones with a diffraction contrast and the pronounced boundary between them. According to the mutual orientation of zones within a crystal, the γ phase zones with a mild internal structure were considered to be reverted austenite (Figs. 1e and 1f). These observations gave reason to believe that the γ_{resII} phase forms on residual austenite as a nucleus of chemical inhomogeneity. This is also confirmed by an increase in the γ phase crystal sizes (lines 4 and 5 in Table 1).

The stability of the γ_{res} and γ_{revII} phases to plastic deformation was concluded from the degree of the $\gamma \rightarrow \alpha$ transformation (m_γ) in the fractures of samples. As seen in Fig. 8, the impact strength of (α -martensite + γ_{res}) samples (aging at 430°C, 3 h) is higher than in those with the (α -martensite + $\gamma_{\text{res}} + \gamma_{\text{revII}}$) structure (aging at 490°C, 3 h). An increase in the exposure time during the ST quenching, when the nickel content in austenite decreases, makes the difference in m_γ values more pronounced (Fig. 8, curves 3 and 4). It is evident that only residual austenite undergoes the $\gamma \rightarrow \alpha$ transformation, whose degree increases with exposure time. When the transformation passes completely (Fig. 8, curves 1 and 3), the austenite content in the fractures of (α -martensite + $\gamma_{\text{res}} + \gamma_{\text{revII}}$) samples corresponds to

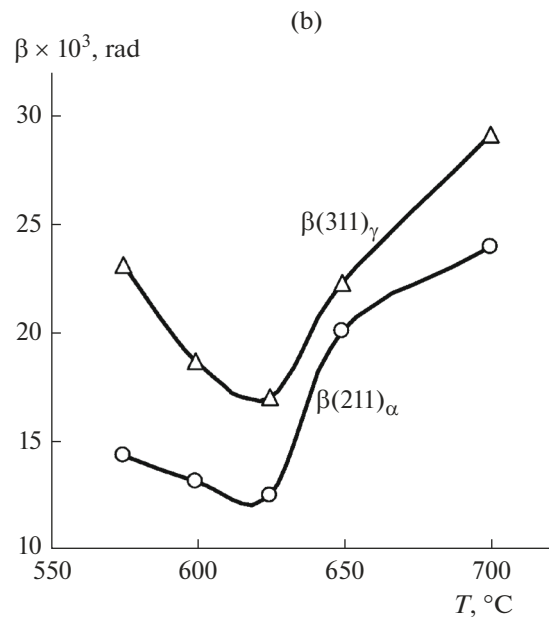
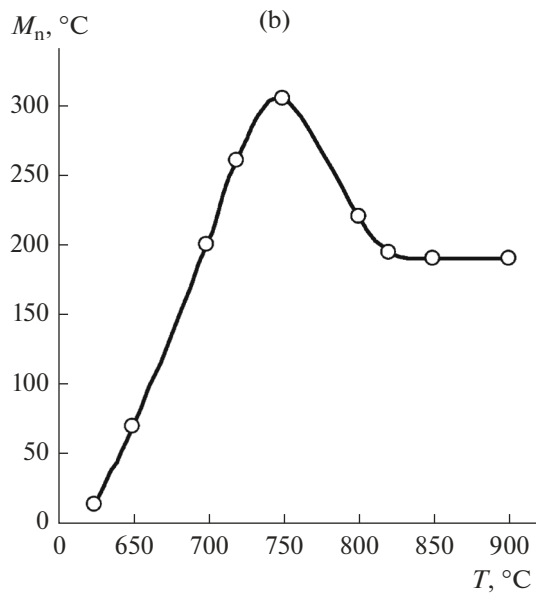
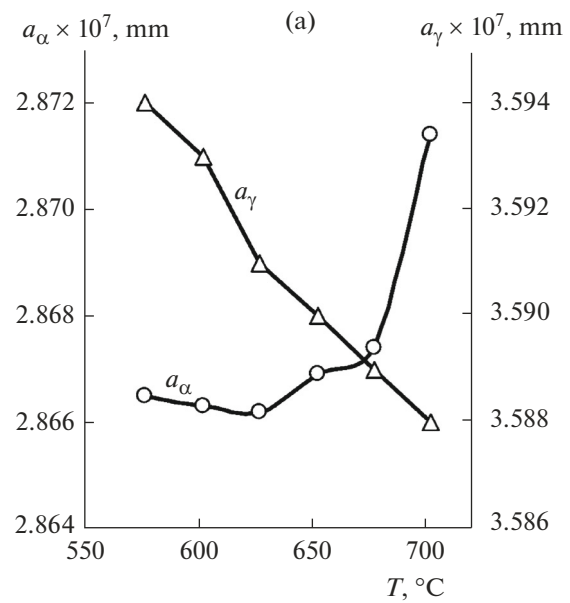
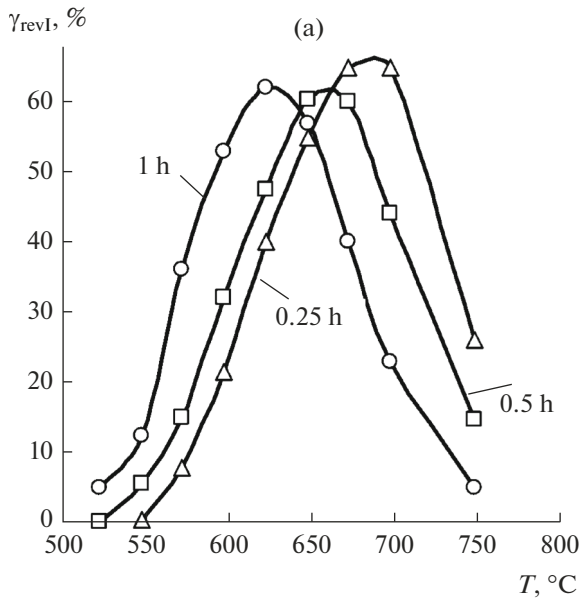


Fig. 2. (a) Reverted austenite I concentration as a function of heating temperature and exposure time at pre-annealing; (b) critical temperature M_n versus heating temperature at 1-h exposure of 03N18K9M5T steel.

Fig. 3. (a) Lattice parameters a_α and a_γ of martensite and reverted austenite I; (b) X-ray diffraction line width β as a function of annealing temperature in 03N18K9M5T steel. The exposure time was 1 h.

the amount in γ_{revII} (Fig. 7, curve 2; Fig. 8, curve 2), which is stable to temperatures of -196°C [1].

The contributions of crystal size and fine structure to the hardening of residual austenite were evaluated using the Hall–Petch relation, which is suitable for both single- and two-phase alloys. The contribution of dispersion of γ_{res} crystals to the yield strength of steel at its content of 24% was ~ 300 MPa, while the contribution from phase hardening yielded ~ 500 MPa [8], which is comparable with the increment in the yield strength ($\Delta\sigma_{0.2}$) of austenite alloys due to phase hard-

ening or plastic deformation (300–600 MPa) [5, 9]. The dislocation density in residual austenite was calculated to be $\sim 2 \times 10^{11} \text{ cm}^{-2}$ at $\Delta\sigma_{0.2} = 500$ MPa [8], which is within an order of magnitude of the value experimentally found in phase-hardened austenite [5]. In the steel under study, the yield strength of residual austenite is close to that of non-aged martensite, being ~ 800 – 1000 MPa, whereas the strength of γ_{rev} is two times higher than that of the γ_{res} phase, being at the same level with strength characteristics of aged martensite ($\sigma_v \approx 2100$ MPa and $\sigma_{0.2} \approx 2000$ MPa) [1, 8].

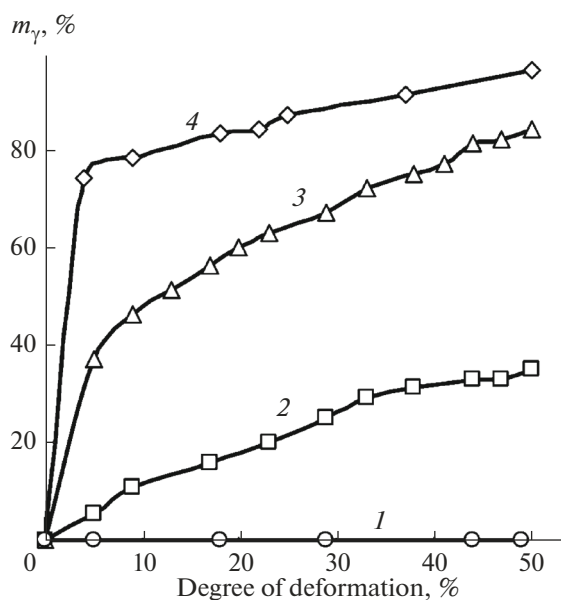


Fig. 4. Resistance of reverted austenite I to plastic deformation by rolling (m_γ) in 03N18K9M5T steel at various annealing temperatures: (1) 575; (2) 600; (3) 650; (4) 700°C. The exposure time was 1 h.

The difference in the properties of γ_{res} and γ_{revII} phases is, as follows. The dislocation density in packet martensite of N18K9M5T steel is $\sim 10^{11} - 10^{12} \text{ cm}^{-2}$ [10]. Residual austenite partially inherits (via the γ_{resI} phase) the dislocation structure of parental martensite [9]. The additional increase in dislocation density in the γ_{res} phase is due to plastic deformation occurring upon the martensite $\alpha \leftrightarrow \gamma$ transformations during the quenching owing to the change in specific volume and shape of crystals surrounding the microvolumes of γ_{res} [5]. The high dislocation density is also retained in residual austenite owing to short-term exposures at the quenching temperature, as well as to the presence of intermetallic particles in the initial (α -martensite + γ_{revI}) phase, which seem to reduce the dislocation mobility until they are completely dissolved. One cannot exclude that a certain contribution to the hardening of residual austenite is made by concentration microinhomogeneities formed through the dilution of intermetallic compounds that increase the efficiency of phase hardening via the interaction with defects of the γ_{res} phase.

Shorter exposure times at the quenching temperature ensure a lower degree of phase hardening, which manifests itself by a decrease in the (311) line of austenite (Fig. 6b). The degree of dispersion of γ_{res} decreases as well (see Table 1 and Fig. 1d). Meanwhile, the yield strength of the γ_{res} phase does not decrease, but even increases at exposure times less than 4 min ($\gamma_{\text{res}} > 35\%$). This is evident from data acquired on quenched samples (Table 2). A decrease

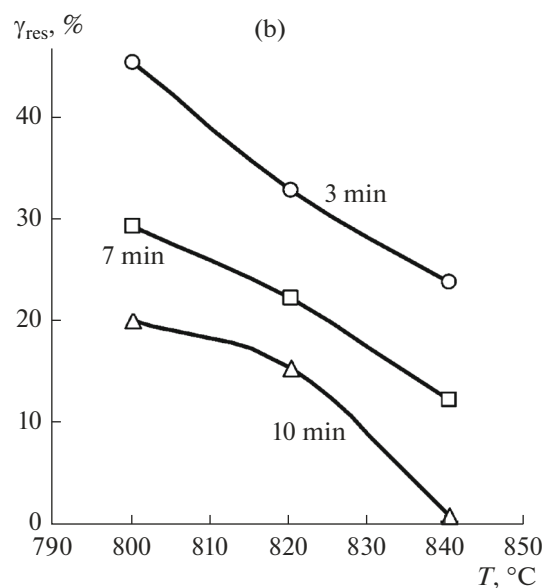
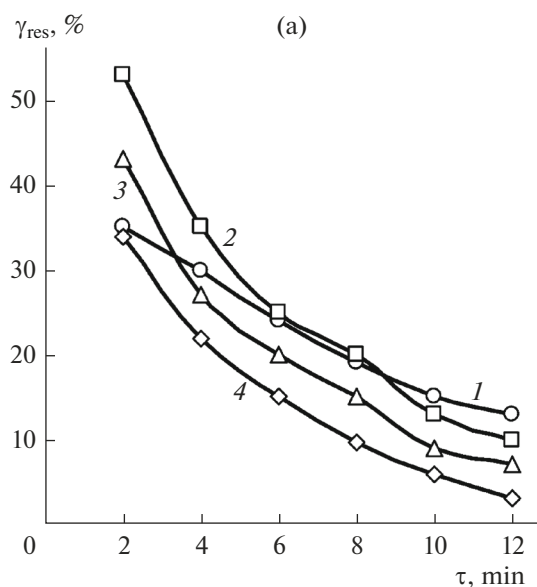


Fig. 5. The amount of residual austenite (γ_{res}) versus the exposure time (τ) upon ST quenching (820°C) in 03N18K9M5T steel pre-annealed for 1 h at temperatures of (1) 575, (2) 600, (3) 650, and (4) 700°C (a) and at temperatures above A_{c3} upon annealing at 575°C for 1 h (b).

in yield strength of the γ_{res} phase seems to be compensated or even overlapped owing to an increase in the amount of phase microinhomogeneities in the γ phase, as well as the presence of intermetallic particles at small exposure times (2 min), because of the slight reduction in the dislocation density and dispersion of crystals (see Figs. 1d and 1h).

As mentioned above, the yield strength of reverted austenite exceeds that of residual austenite by ~ 1000 MPa. This difference can be caused by neither the dispersion

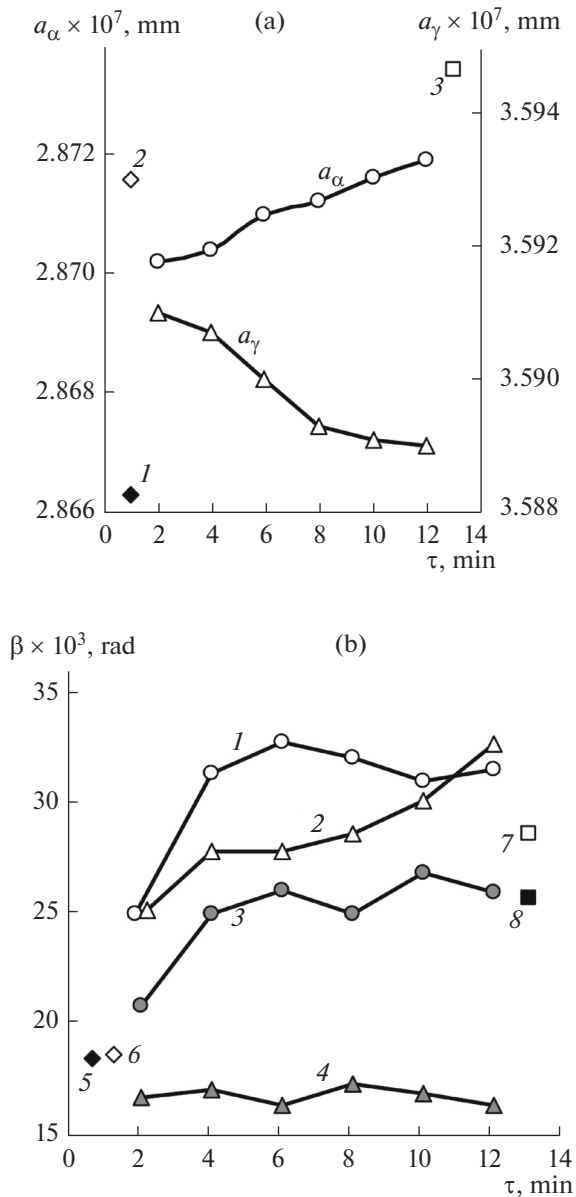


Fig. 6. (a) Lattice parameters of martensite (a_α) and residual austenite (a_γ) and (b) widths (β) of $(220)_\alpha$ and $(311)_\gamma$ XRD lines at various exposure times (τ) upon the quenching of 03N18K9M5T steel: (a) (1, 2) a_α and a_γ (initial state—annealing at 600°C, 1 h); (3) a_α after conventional quenching (820°C, 1 h); (b) (1, 3 and 2, 4) $\beta(220)_\alpha$ and $\beta(311)_\gamma$, respectively, after the ST quenching at 820°C and aging at 490°C, 3 h; (5, 6) initial values of $\beta(220)_\alpha$ and $\beta(311)_\gamma$ (annealing at 600°C, 1 h); (7, 8) values of $\beta(220)_\alpha$ after the quenching at 820°C, 1 h and subsequent aging at 490°C, 3 h.

of crystals nor the degree of phase hardening. The formation of γ_{revII} on the γ_{res} phase augments the crystal sizes of the γ phase, and the degree of phase hardening may be even smaller than in the case of γ_{res} , because γ_{revII} arises at the temperature of 490°C, and the shear process of the $(\alpha\text{-martensite} + \gamma_{\text{res}})$ structure formation ends at room temperature.

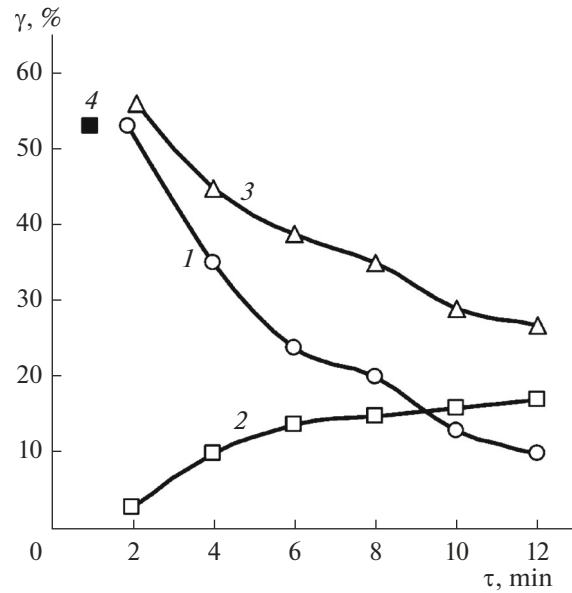


Fig. 7. Austenite content as a function of exposure time at a quenching temperature of 820°C in 03N18K9M5T steel after aging at 490°C, 3 h: (1) γ_{res} ; (2) γ_{revII} ; (3) $\gamma_{\text{total}} = \gamma_{\text{res}} + \gamma_{\text{revII}}$. The initial state—annealing at 600°C, 1 h.

A decrease in the degree of phase hardening of reverted austenite may also be caused by the orientation influence of the γ_{res} phase on the nucleation and growth of γ_{revII} crystals. It is not excluded that the displacement of atoms during the $\alpha \rightarrow \gamma$ transition occurs in reciprocal ways with partial annihilation of dislocations [5, 6, 9].

In our opinion, the abrupt increment in the yield strength of the γ_{revII} phase is due to dispersive hardening. Meanwhile, this process for Fe–Ni–austenite necessitates more than 1% Ti. Furthermore, even at a Ti content of ~3%, the yield strength of austenite increases by only 400–500 MPa, whereas the difference upon the combined phase hardening and aging is 600–700 MPa. On the other hand, as shown in works on austenitic steels [5, 9], the efficient hardening of austenite on account of dispersive hardening requires higher aging temperatures (600–700°C). It is evident that dispersed intermetallic particles arising upon aging in martensite are inherited by reverted austenite crystals within the movement of the γ/α interface during the $\alpha \rightarrow \gamma$ transformation. This is also evident from the fact that intermetallic compounds occur at a rate greatly exceeding the rate of γ_{revII} formation [6]. Moreover, the dilution rate of intermetallic compounds in austenite is almost excluded because of comparatively low aging temperatures.

Intermetallic compounds inherited by reverted austenite are well resolved after heating to 550 and 600°C (Figs. 1a, 1b, and 1g). Although observing them after aging at 490°C meets some difficulties, intermetallic compounds are detected in regions of local

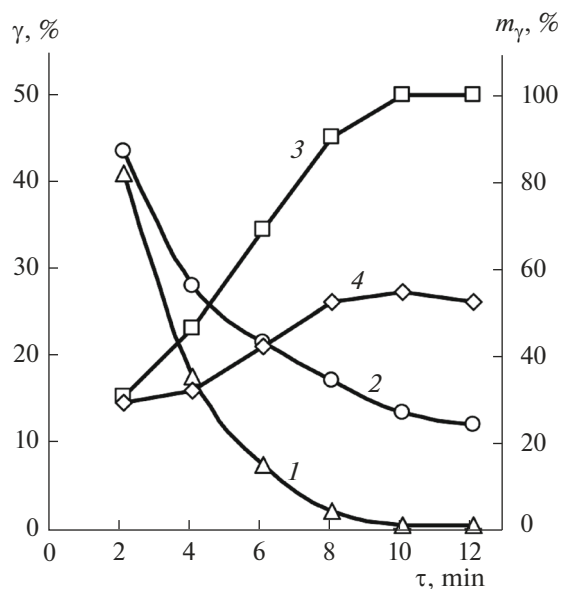


Fig. 8. Austenite content (1, 2) in the fracture of sample on KCU and degree of deformation $\gamma \rightarrow \alpha$ transformation (3, 4) in 03N18K9M5T steel versus exposure time at a quenching temperature of 820°C and aging temperature. The aging temperatures are (1, 3) 430°C and (2, 4) 490°C (aging for 3 h).

change in contrast. The large increment in yield strength of the γ_{revII} phase on account of inherited particles is likely due to their high degree of dispersion, as well as to the large volume content, which cannot be achieved in pure austenite, when the aging causes the formation of Ni_3Ti particles with hcp and fcc lattices isomorphic to the matrix lattice [5, 9]. It is obvious that the γ_{revII} phase of the steel inherits only the hcp Ni_3Ti particles, whose lattice exhibits the larger mismatch with that of the matrix.

As follows from the analysis of the fine structure, morphology, and other characteristics of austenite, the creation of dispersed nickel-enriched γ_{revI} martensite with a fine structure, developed by phase hardening, in the (A_n – A_k) biphase domain together with parental α -martensite, as well as the formation of a concentration inhomogeneity on its basis upon high-temperature ST quenching, leads to the steel structure stabilization to 60% γ_{res} . The presence of residual austenite and nickel-enriched martensite microvolumes near the γ_{res} domain in the steel favors a decrease in the

Table 2. Strength characteristics of 03N18K9M5T steel after ST quenching

Exposure at quenching, min	$\sigma_{0.2}$, MPa	σ_v , MPa
2	1210–1250	1315–1346
4–12	970–1030	1090–1114

A_n critical temperature of the inverse $\alpha \rightarrow \gamma$ transformation [7], which leads to the formation of nanoduplex austenite (γ_{revII} on residual austenite) upon aging.

Since residual austenite possesses high plasticity and is metastable to plastic deformation, the complex structure of the steel retains good relaxation ability. Exhibiting high strength and resilience to supercooling and plastic deformation [1], reverted austenite II helps to strengthen α/γ interfaces, which enables one to maintain excellent strength characteristics.

CONCLUSIONS

1. The fundamental reasons for stability of austenite in martensite aged 03N18K9M5T steel were established from structural features and properties of austenite. Among them were dispersion of austenite crystals, concentration inhomogeneity of a solid solution, phase hardening, and presence of intermetallic compounds.

2. The difference in characteristics of residual and reverted austenite II was substantiated. High yield strength values of residual austenite were due to small crystal sizes of the γ phase and a high density of dislocations ($\sim 10^{11} \text{ cm}^{-2}$). A twofold increase in strength of reverted austenite II in comparison with residual austenite is due to the inheritance of Ni_3Ti dispersed particles during the aging-induced $\alpha \rightarrow \gamma$ transition, which were earlier released in martensite of residual austenite.

3. The role of residual and reverted austenite II in the formation of 03N18K9M5T steel was shown. High viscosity values at elevated strength originate from the complex structure based on α -nanomartensite and nanoduplex austenite with peculiar morphology and characteristics. The presence of residual austenite with high plasticity and metastability to plastic deformation ensures high relaxation ability of the structure. Being stable to plastic deformation, reverted austenite II, arising upon aging on a soft substrate of residual austenite, reinforces the α/γ interfaces and maintains high strength of the steel.

FUNDING

This work was supported by the Russian Foundation for Basic Research (project no. 18-10-1-36).

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Translated by O. Maslova