Metal Science and Heat Treatment *Vol. 43, Nos. 1 – 2, 2001*

MARAGING STEELS

UDC 669.15-194.55:548.735

THE ROLE OF TEXTURE IN THE PROCESSES OF AGING AND DELAYED FRACTURE OF MARAGING STEEL

V. V. Berezovskaya,[1] S. V. Grachev,[1] F. V. Mineev,[1] D. V. Titorova,[1] and V. B. Shirikhin[1]

Translated from Metallovedenie i Termicheskaya Obrabotka Metallov, No. 2, pp. 21 – 24, February, 2001.

Processes of aging of maraging steel 03N18K3M3T from commercial heats are investigated. Dilatometric, x-ray diffraction, and fractographic studies and tests for corrosion strength and delayed fracture (DF) are described. It is shown that after quenching from 820°C and aging at 400°C a DF crack propagates in the studied steel over grain boundaries at a high speed, which causes brittle fracture. The corrosion rate of the steel after such a treatment is also high. X-ray diffraction analysis has shown the presence of a strong and virtually single-component texture in martensite, which is preserved after forging, prior high-temperature quenching, and base quenching from 820°C.

INTRODUCTION

It is known [1 – 3] that delayed fracture of maraging steels manifests itself in the range of aging temperature close to 400°C and is connected with the appearance of local internal stresses. The maximum of this kind of fracture has not been determined due to the difficulties in the study of the early stages of decomposition by direct diffraction methods. The decomposition of a supersaturated solution in this stage is accompanied by simultaneous processes of rest and relaxation of stresses. A characteristic typical feature of delayed fracture is the intergrain brittle propagation of the crack. It is assumed that intergrain fracture is connected either with predominant decomposition of martensite over grain boundaries [1, 2] or with concentration of dislocations on grain boundaries as a result of their planar slip through coherent particles in the process of DF tests [3]. In this case, it is necessary that hydrogen had arrived at the tip of the growing crack in delayed fracture.

The aim of the present work consisted of studying the structure of maraging steel 03N18K3M3T in the initial aging stages and determining the possible causes of its catastrophic embrittlement in delayed fracture in this state.

METHODS OF STUDY

We studied[2] maraging steel 03N18K3M3T obtained by two methods, namely, (*I*) vacuum-induction melting (VIM) and (*II*) vacuum-induction melting and vacuum-arc remelting (VIM + VAR) (see Table 1).

After solution annealing the castings were forged for squares 90 × 90 mm in size and subjected to prior 2-h quenching from 1200°C in water. Then followed the base quenching of the preforms, namely, steel *I* was heated to 820°C for 1 h and cooled in water and steel *II* was subjected to three-stage quenching from 975°C (1 h), 950°C (1 h), and 925°C (1) and cooled in water. After the base quenching the forgings were cut into prismatic specimens (over the axis) and then aged at 350 – 500°C for 3 h.

DF tests were performed at a constant load by the method of pure flexure in distilled water at room temperature. The fracture behavior of the specimens was studied under a RÉMMA-202M scanning electron microscope. The texture was studied in cobalt K_α-radiation by the Schultz method of "reflection" from plane {110}. Photographs were taken from the end surface of prismatic specimens, which was perpendicular to the axis of the forging. We also performed a dilatometric analysis.

---

[1] Ural State Technical University (UPI), Ekaterinburg, Russia; VIZ-Stal' Company, Ekaterinburg, Russia; Physicotechnical Institute of the Ural Branch of the Russian Academy of Sciences, Izhevsk, Russia.

[2] The authors express their gratitude to D. B. Titorov for participating in the discussion of the results of the present work.

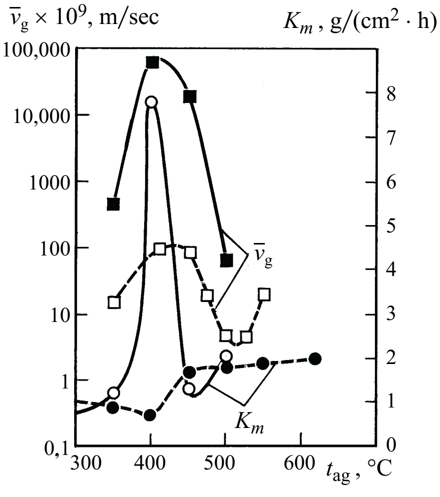

Fig. 1. Mean rate of crack growth $\bar{v}_g$ in tests for delayed fracture and the rate of corrosion K_m in a 20-h test in 3% aqueous solution of NaCl: solid lines) steel *I*; dashed lines) steel *II*.

RESULTS AND THEIR DISCUSSION

The tests have shown that the maximum rates of DF and corrosion for steel *I* correspond to an aging temperature of 400°C (Fig. 1). The maximum of the DF rate for steel *II* is shifted somewhat to the range of higher temperatures and is almost three orders of magnitude lower than in steel *I*; the corrosion rate increases monotonically starting from 450°C. The dependence of the corrosion rate of steel *I* on the aging temperature (Fig. 1) indicates the presence of local chemical inhomogeneity in the solid solution. The inhomogeneity of steel *I* after aging at 400°C (before the beginning of formation of a stable η-Ni_3Ti-phase) can be caused by the inhomogeneity of the solid solution with respect to titanium inherited from the γ-region and causing the formation of clusters enriched with nickel and titanium atoms due to the strong interaction between these atoms [4]. The latter circumstance is the reason behind the marked acceleration of corrosion in the alloy, because in this case the solid solution is depleted of titanium, i.e., the element that passivates the given alloy under the conditions of our experiment (in a solution of sodium chloride). In later aging stages (at 400°C), the particles of the hardening phase segregate not only in these zones but also on crystal lattice defects, i.e., heterogeneously [3], which leads to leveling of the chemical composition of the steel and reduction of the corrosion rate.

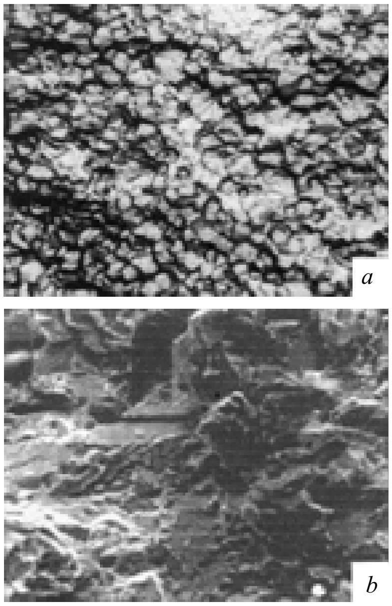

Fig. 2. Fractograms of steels *I* (*a*) and *II* (*b*), which have been aged at 400°C, after delayed fracture, × 650.

Scanning electron microscopy has shown that when steel *I* is tested for DF after aging at 400°C, it fractures over grain boundaries; steel *II* fractures in a brittle manner over the bodies of the grains (Fig. 2). The size of the grains $d = 8 - 10$ and $20 - 25$ μm for steels *I* and *II*, respectively.

The differential thermal analysis performed earlier and the study of the resistance have shown that in the initial stage of the aging at 400°C after quenching from 820°C before the formation of the principal hardening phase of a Ni_3Ti type, another process occurs in the steel. After triple quenching this aging stage is absent. It was suggested in [5] that concentrated inhomogeneities of nickel form in the solid solution in the early aging stages, in which a titanium-rich phase nucleates later at 450°C. However, the possible causes of formation of such inhomogeneities were not discussed in this work.

Our study of the $\alpha \to \gamma \to \alpha$ transformations performed by the method of dilatometric analysis of specimens heated at a rate of 200 K/h has shown the presence of anomalies above 620°C (Ac_1) similar to those observed earlier in [6, 7] in the range of reverse $\alpha \to \gamma$ transformations. They differed in steels *I* and *II* (Fig. 3). We can distinguish several stages in the reverse transformation at a temperature more than 620°C depending on the size of the dilatometric effect $\Delta l/l$. For

TABLE 1. Chemical Composition of Steel 03N18K3M3T after Two Melts

Steel (heat)	Content of elements, %									
	C	Ni	Co	Mo	Ti	Al	Si	Mn	S	P
I (VIM)	0.03	18	3.1	3.15	1.3	0.11	0.1	0.1	0.01	0.01
II (VIM + VAR)	0.02	18	3.05	3.41	1.11	0.11	0.04	0.03	0.006	0.007

Note. The method of melting is indicated in parentheses.

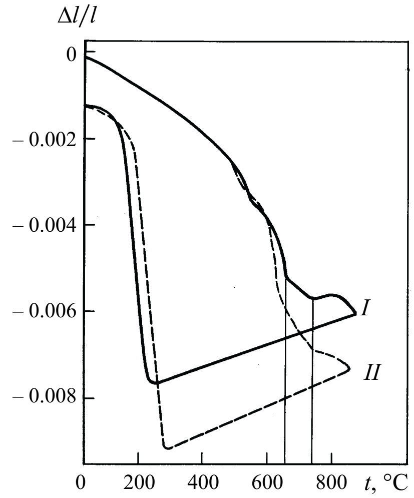

Fig. 3. Dilatograms of steels *I* and *II*.

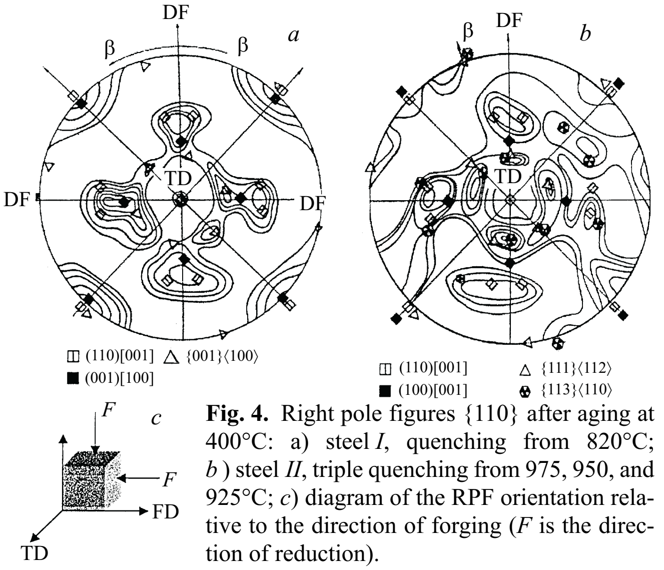

Fig. 4. Right pole figures {110} after aging at 400°C: a) steel *I*, quenching from 820°C; *b*) steel *II*, triple quenching from 975, 950, and 925°C; *c*) diagram of the RPF orientation relative to the direction of forging (*F* is the direction of reduction).

steel *I* they corresponded to the following temperatures: *1*) 620 – 670°C ($\Delta l/l = -0.15\%$), *2*) 670 – 740°C ($\Delta l/l = -0.05\%$), *3*) 740 – 800°C ($\Delta l/l = +0.01\%$); for steel *II* we obtained: *1*) 620 – 650°C ($\Delta l/l = -0.15\%$); *2*) 650 – 740°C ($\Delta l/l = -0.17\%$); *3*) 740 – 850°C ($\Delta l/l = -0.04\%$). In [6, 7], such anomalies were explained by the anisotropic change in the sizes of the specimen connected with texture effects. By the data of [6], the staged nature of the reverse $\alpha \to \gamma$ transformation is connected with the predominance of diffusion or of a martensitic mechanism of transformation at different temperatures. An analysis of the size of the dilatometric effect has shown the following. The transformation that occurs predominantly by a shift mechanism in the anisotropic field of internal stresses (stage *1*) is followed by a predominantly diffusion transformation (stage *2*) accompanied by the dissolution of the hardening phase Ni_3Ti and redistribution of the alloying elements; then follows a predominantly shift transformation again, because the temperature A_i of the titanium-enriched martensite is higher (stage *3*). As a result of the staged occurrence of the inverse $\alpha \to \gamma$ transformation, the solid solution formed after cooling has an inhomogeneous chemical composition. Comparing the dilatometric effects in stages *1* and *3* of the inverse transformation, we see that the transformation of titanium-enriched martensite makes the greatest contribution to the texture effects in the inverse transformation and that steel *I* has more substantial texture anomalies.

The x-ray diffraction analysis confirmed the results of the analysis of the dilatograms. Figure 4 presents right pole figures (RPF) of the studied steel after the two mentioned regimes of quenching and aging at 400°C. The pole figure of steel *I* shows the presence of a strong texture (110)[001] and (001)[100] and a very weak component of the family of planes {111}⟨112⟩. After triple quenching, the components (110)[001] and (001)[100] in steel *II* weaken and the component {111}⟨112⟩ intensifies, plus a new component {113}⟨110⟩ appears. The results obtained show that the texture of quenched maraging steel is preserved after hot forging and preliminary high-temperature quenching, which is connected with the shift nature of the martensitic transformation and the structural inheritance [8].

We can infer from the results of the dilatometric study that the chemical inhomogeneity of martensite is caused by the diffusion redistribution of nickel and titanium atoms in austenite. Martensite packets formed from titanium-enriched austenite have a preferred orientation. The subsequent process of decomposition of the solid solution is “attached” to the regions of the α-solid solution that have formed from titanium-enriched regions of austenite and consists of the formation of “titanium – nickel” clusters. In other words, the decomposition of martensite in the early aging stages with cluster formation is oriented, which amplifies the anisotropy of the properties of the steel. In the DF tests, the oriented growth of the tip of the crack and the hydrogen that permeates into it create high stress concentrations, which accelerates the fracture process in the steel by many times.

In the case of a weak texture, the chemical inhomogeneity of the austenite, and hence of the martensite, is not oriented. This prevents oriented homogeneous decomposition of the matrix, and the hydrogen that permeates into the steel is “scattered” in the field of uniformly distributed internal stresses. In this case, the stress concentration is not high and the fracture decelerates.

Our study of delayed fracture in maraging steel 03N18K3M3T has shown that one of the causes of its catastrophic embrittlement is the texture of the martensite.[3] The virtually single-component texture inherited after hot forging is highly stable after prior high-temperature quenching and

---

[3] The question of the role of texture in delayed fracture of maraging steel remains open, because it has not been explained how the texture appearing in the volume of the grains affects the intergrain crack propagation. (*Ed. note*).

base quenching from 820°C. The use of triple quenching results in smearing of the texture due to the appearance of additional components.

Aging of textured martensite at 400°C causes the formation of "nickel – titanium" clusters in titanium-enriched martensite packets due to the strong interaction between these atoms. The rate of corrosion in this steel increases substantially. Further segregation of Ni_3Ti particles by the mechanism of heterogeneous decomposition causes leveling of the chemical composition of the steel over the entire volume and decelerates the corrosion. After triple quenching and aging at 400°C, the stage of homogeneous decomposition of martensite does not occur, which decreases the corrosion rate in the steel.

CONCLUSIONS

The presence of strong and virtually single-component texture in steel 03N18K3M3T after forging, preliminary (high-temperature) quenching, and base (from 820°C) quenching is the reason behind the appearance of anisotropy of stresses in the matrix, which increases due to the formation of clusters. The "smearing" of the texture of martensite formed as a result of triple quenching (from 975, 950, and 925°C) performed after forging and prior quenching causes a more isotropic stress distribution in the steel, changes the mechanism of delayed fracture, and substantially diminishes the rate of crack propagation in the presence of a hydrogen-bearing medium.

REFERENCES

1. M. L. Perkas, "Structure, properties, and domains of application of high-strength maraging steels," *Metalloved. Term. Obrab. Met.*, No. 5, 23 – 33 (1985).
2. I. S. Toidorova, V. V. Zabil'skii, and V. I. Sarrak, "Delayed fracture of maraging steels," *Fiz. Met. Metalloved.*, No. 7, 5 – 11 (1991).
3. M. A. Smirnov, V. G. Pushin, N. G. Kareva, et al., "Effect of high-temperature deformation on delayed fracture of maraging steel," *Fiz. Met. Metalloved.*, **53**, Issue 5, 1021 – 1026 (1982).
4. M. D. Perkas and V. M. Kardonskii, *High-Strength Maraging Steels* [in Russian], Metallurgiya, Moscow (1970).
5. Y. Asayama, "Delayed failure and precipitation behavior in maraging steels," *Trans. Jpn. Inst. Met.*, **28**(4), 281 – 290 (1987).
6. V. I. Zel'dovich and V. D. Sadovskii, "A study of the $\alpha \to \gamma$ transformation and recrystallization in maraging steel," *Fiz. Met. Metalloved.*, **34**, Issue 3, 518 – 528 (1972).
7. Yu. R. Nemirovskii, T. A. Doroshkevich, M. R. Nemirovskii, et al., "Regular size changes in heat treatment of sheets from maraging steel," *Fiz. Met. Metalloved.*, **68**, Issue 4, 746 – 754 (1989).
8. M. A. Smirnov, V. M. Schastlivtsev, and L. G. Zhuravlev, *Fundamentals of Heat Treatment of Steel* [in Russian], UrO RAN, Ekaterinburg (1999).