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# RAISING THE STRUCTURAL STRENGTH OF SYSTEMATICALLY ALLOYED Fe – Cr – Ni – Mo-BASE MARAGING STEELS

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The effect of systematical Mo, Ti, Al and Ca alloying of corrosion-resistant maraging steels of type Kh11N10M2T and of the modes of their heat treatment on the phase composition, combination of properties and resistance to brittle fracture is studied. It is shown that the structural strength of the steels can be raised by additional alloying with copper and formation of a regulated content of metastable retained austenite.

***Key words:*** high-strength steels, alloying, phase composition, aging of martensite, retained austenite, impact toughness, crack resistance.

## INTRODUCTION

Maraging steels (MS) based on the Fe – Cr – Ni – Mo system have found wide application as structural materials due to their enhanced corrosion resistance, adaptability to manufacture and resistance to brittle fracture at a comparatively low content of expensive alloying elements [1 – 3]. At the same time, the strength level of this class of steels ($\sigma_r$ = 1400 – 1600 MPa) is inferior to that of maraging steels of type N18K9M5 and structural steels of other alloying systems. A promising direction for raising the strength of Fe – Cr – Ni – Mo-base maraging steels is development of compositions containing an elevated amount of elements forming fine hardening particles (Ti, Al, V). However, the range of application of novel corrosion-resistant MS in practical engineering is limited by the absence of systematic data on the characteristics of crack resistance that determine the structural strength and reliability of operation of these materials in parts and components of constructions.

The aim of the present work[3] was to study the effect of the chemical composition and modes of heat treatment of Fe – Cr – Ni – Mo-base maraging steels on the combination of mechanical properties and crack resistance in order to raise the structural strength and determine the ultimate level of alloying with elements forming fine hardening particles.

## METHODS OF STUDY

The chemical compositions of the studied MS are presented in Table 1. Test ingots with a mass of 25 kg from steels *1 – 8* were melted at the Bardin TSNIIchermet by a vacuum technique with the use of pure blend materials. Steels ChS92 (heats *9* and *12*), ÉP678 (heat *10*), ÉP679 (heat *11*) and ÉP832 (heat *13*) were produced at the Chelyabinsk Integrated Iron-and-Steel Works by the method of vacuum induction melting followed by vacuum-arc remelting. The content of carbon and alloying elements was determined by chemical analysis and controlled with the help of a SPECTROMAXx optical emission spectrometer. The phase composition of the steels was determined by the x-ray diffraction method with the help of a DRON-3 diffractometer. The metallographic analysis was performed with the help of a NEOPHOT-21 microscope.

The ingots were forged in hot condition for preforms with cross section 35 × 35 mm and bars with cross section 14 × 14 mm. Before the mechanical treatment the specimens were quenched in air from 850, 920, 950 and 1050°C. A part of the specimens after the quenching was subjected to accelerated heating in a salt bath to 850°C with 7-min hold and then cooled in air to room temperature in order to create retained austenite in the structure [4]. The aging was performed for 3 h at 450 – 620°C. Specimens 5 mm in diameter were subjected to uniaxial tension according to GOST 1497–84; compact specimens of type 3 with a thickness of 12 mm were tested for static crack resistance (fracture toughness) according to GOST 25.506–85 at room temperature using EUS-20 and INSTRON8001 universal testing

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**TABLE 1.** Chemical Compositions of Maraging Steels

| Heat | Content of elements, wt.% | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | C | Ni | Cr | Mo | Ti | Al | V | Cu | Σ(Ti + Al) |
| *1* | 0.027 – 0.033 | 9.45 – 9.50 | 10.8 – 10.9 | 1.85 – 1.90 | 1.06 | 0.36 | – | 0.52 | 1.44 |
| *2* | | | | | 1.01 | 0.34 | – | 1.03 | 1.35 |
| *3* | | | | | 0.99 | 0.32 | – | 1.53 | 1.31 |
| *4* | | | | | 1.00 | 0.30 | – | 2.48 | 1.30 |
| *5* | 0.026 – 0.031 | 9.40 – 9.50 | 10.7 – 10.8 | 1.80 – 1.90 | 0.81 | 0.87 | 0.21 | 1.40 | 1.18 |
| *6* | | | | | 0.35 | 0.96 | 0.21 | 1.30 | 1.31 |
| *7* | | | | | 1.10 | 0.21 | 0.23 | 2.10 | 1.31 |
| *8* | | | | | 1.47 | 0.15 | 0.19 | 1.80 | 1.62 |
| *9* | 0.010 | 10.20 | 11.00 | 1.80 | 0.86 | 0.33 | 0.14 | 0.90 | 1.19 |
| *10* | 0.026 | 9.35 | 10.65 | 1.97 | 0.90 | 0.09 | – | 0.10 | 0.99 |
| *11* | 0.000 | 8.94 | 11.24 | 2.04 | 1.01 | 0.11 | 0.02 | 0.09 | 1.12 |
| *12* | 0.020 | 9.46 | 10.70 | 1.55 | 1.10 | 0.45 | 1.07 | 1.37 | 1.55 |
| *13* | 0.017 | 9.28 | 11.40 | 2.14 | 1.14 | 1.07 | – | 1.83 | 2.21 |

**Notes.** 1. The contents of S and P in all the steels ranged within 0.004 – 0.006%.
2. In addition to the listed elements steel *10* contained 0.02% B, 0.06% Zr, 0.06% Ca, and 0.02% Nb.

machines. The tests for impact bending were conducted for specimens of types I and II of GOST 9454–78 with *U*- and *V*-notches at room temperature using a MK-30 pendulum impact machine and a "Tinius Olsen" IT 542M pendulum machine and recording the impact loading diagrams in coordinates "force – displacement." The total fracture energy of an impact specimen was represented by two components ($A = A_n + A_p$), where $A_n$ is the energy of crack nucleation and $A_p$ is the energy of crack propagation, in accordance with GOST 22848–77. The values of the parameter of dynamic crack resistance were computed by the method of [5] in terms of the Rice – Cherepanov – Bilby integral $J_{1d}$, i.e.,

$$J_{1d} = 2A_n / B(W - a), \qquad (1)$$

where $A_n$ is the energy of crack nucleation, $B$ is the width of the specimen, $W$ is the height of the specimen, and $a$ is the length of the stress concentrator (a notch or a fatigue crack). At least three identical specimens were tested for one point. The scattering of the data of the mechanical tests did not exceed ± 2.5%.

The fractures of the specimens were studied using JSM-U3 and TESCAN VEGA II XMU scanning electron microscopes.

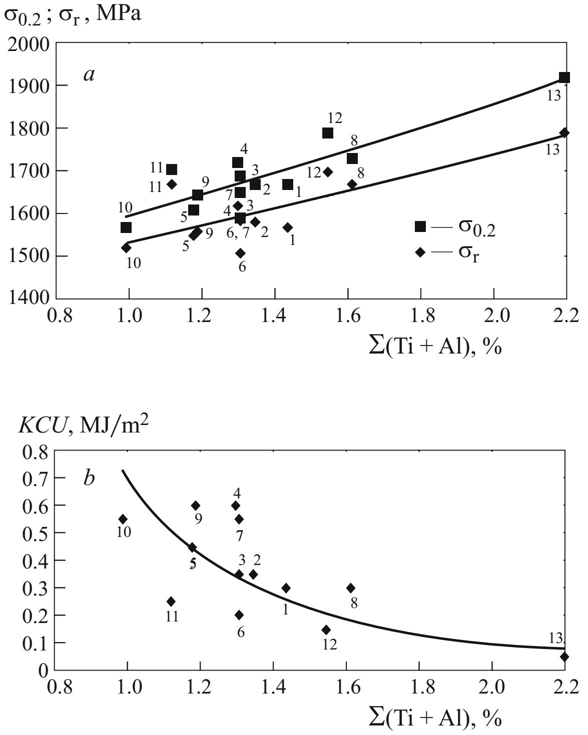

**Fig. 1.** Strength characteristics (*a*) and impact toughness (*b*) of maraging Fe – Cr – Ni – Mo steels as a function of the summed content of titanium and aluminum (the numbers of the heats are given at the curves).

## RESULTS AND DISCUSSION

Depending on the chemical composition of the metal the mechanical characteristics varied as follows: $\sigma_{0.2} = 1510 - 1790$ MPa, $\sigma_r = 1570 - 1920$ MPa, $\delta = 5 - 9\%$, and $\psi = 17 - 57\%$. A linear dependence of strength parameters on the summed content of titanium and aluminum is presented in Fig. 1*a*. It should be noted that the strength characteristics of heats *4*, *11* and *12* exceed the average trend. For heat *4* this may be connected with the elevated copper content (2.48%); for heat *12* — with the elevated content of copper and vanadium (1.37 and 1.07%, respectively); for heat *11* — with the elevated content of molybdenum (2.04%). The observed ef-

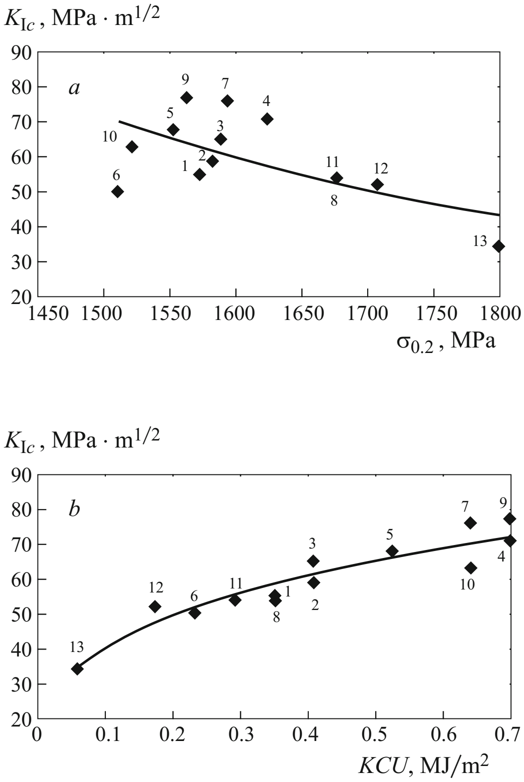

**Fig. 2.** Relation between the parameter of static crack resistance $K_{1c}$ and the conventional yield strength $\sigma_{0.2}$ (*a*) and impact toughness *KCU* (*b*) of Fe – Cr – Ni – Mo maraging steels (the numbers of the heats are given at the labels).

fect of the additives of Mo, Ti, Al and Cu on the strength of the steels agrees well with the data of [6], where the value of $\sigma_{0.2}$ of maraging steels has been shown to depend linearly on an experimentally chosen characteristic of chemical composition, i.e., a hardening equivalent HE (%)

$$\mathrm{HE} = \mathrm{Mo} + 2\mathrm{Ti} + 1.75\mathrm{Al} + \mathrm{Cu} + 0.2\mathrm{Co}, \tag{2}$$

where the contents of the elements are given at the respective notations in wt.%.

The plastic characteristics of most of the steels had at an approximately same level. We have not managed to determine the functional relation between $\delta$ and $\psi$ and the total content $\Sigma(\mathrm{Ti} + \mathrm{Al})$ in the steels. It should be noted that the minimum values of $\delta = 5\%$ and $\psi = 17\%$ corresponded to the composition of heat *13* with maximum content of $\Sigma(\mathrm{Ti} + \mathrm{Al}) = 2.21$ wt.%.

The values of the impact toughness of the studied MS range within $KCU = 0.05 – 0.6$ MJ/m$^2$. In accordance with results presented in Fig. 1*b* the impact toughness lowers substantially upon growth in the summed concentration of titanium and aluminum, and the relation between *KCU* and $\Sigma(\mathrm{Ti} + \mathrm{Al})$ can be approximated by a power law. It is known [7] that growth in the resistance to plastic deformation is accompanied by substantial lowering of the static crack resistance of carbon and alloyed structural steels including MS. However, at the same values of $\sigma_{0.2}$ the level of parameter $K_{1c}$ can be increased by optimum alloying, formation of a superfine-grain structure, and thermomechanical treatment. In works [8, 9] the approach of nonequilibrium thermodynamics (synergism) is used to introduce a parameter St with the aim to determine the optimum structural state of the chosen alloy or steel, i.e.,

$$S_t = K_{1c} / K_{1c\,\max}, \tag{3}$$

where $K_{1c}$ is the crack resistance after the applied variant of heat treatment and $K_{1c\,\max}$ is the crack resistance in the optimal structural state.

According to the level of $S_t$ the phase diagram of a material may be broken into three domains, i.e., (*1*) the domain of implementation of synergistic hardening effect ($S_t = 0.66 – 1$), (*2*) the domain of additive hardening ($S_t = 0.33 – 0.66$), and (*3*) the domain of damaged structural state ($S_t \leq 0.33$).

Synergistic hardening occurs at simultaneous growth of the resistance to deformation and of the resistance to breaking. In the domain of additive hardening growth in the strength is accompanied by decease in the crack resistance. In the domain of structural damage the resistance to breaking falls to a minimum level, which depends little on the subsequent structural changes in the metal as a result of additional treatment. This makes the determination of the possibilities of affecting the structural state of MS with the aim to transfer them to the domains of synergistic or, at least, additive hardening a very important task of the science of materials.

Figure 2*a* presents the dependence of the parameter of static crack resistance $K_{1c}$ on the yield strength $\sigma_{0.2}$, which characterizes the resistance to plastic deformation, for 13 heats of Fe – Cr – Ni – Mo corrosion-resistant MS. It can be seen that the results obtained are scattered considerably with respect to the trend line, and the array of the experimental data matches the lower range of $K_{1c}$ values in Romanov's diagram of structural strength [7]. The dependence of $K_{1c}$ on *KCU* is more manifested (Fig. 2*b*), which makes it possible to predict static crack resistance ($K_{1c}$) of the studied MS from the values of impact toughness. The dependence of $K_{1c}$ on the total content of titanium and aluminum presented in Fig. 3 is nonlinear in contrast to the dependences of $\sigma_{0.2}$ and $\sigma_r$ on $\Sigma(\mathrm{Ti} + \mathrm{Al})$, and can be approximated by a second-degree polynomial. It should be noted that the greatest deviation from the trend line toward higher values of $K_{1c}$ has been detected for heats *9*, *7* and *4* with elevated copper content (0.9, 2.1, and 2.48% respectively). On the contrary, deviations toward reduced values of $K_{1c}$ have been observed for heats *6* and *11* with elevated content of aluminum, titanium and molybdenum.

The detected difference in the impact toughness and static crack resistance of the MS agree well with the results of the study of fracture surfaces. Figure 4*a* presents the frac-

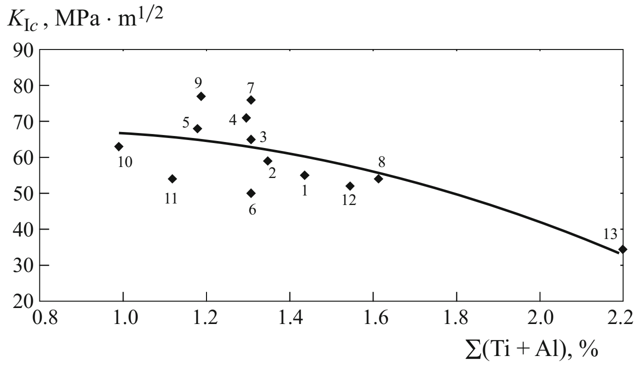

**Fig. 3.** Parameter of static crack resistance $K_{1c}$ of Fe – Cr – Ni – Mo maraging steels as a function of the summed content of titanium and aluminum.

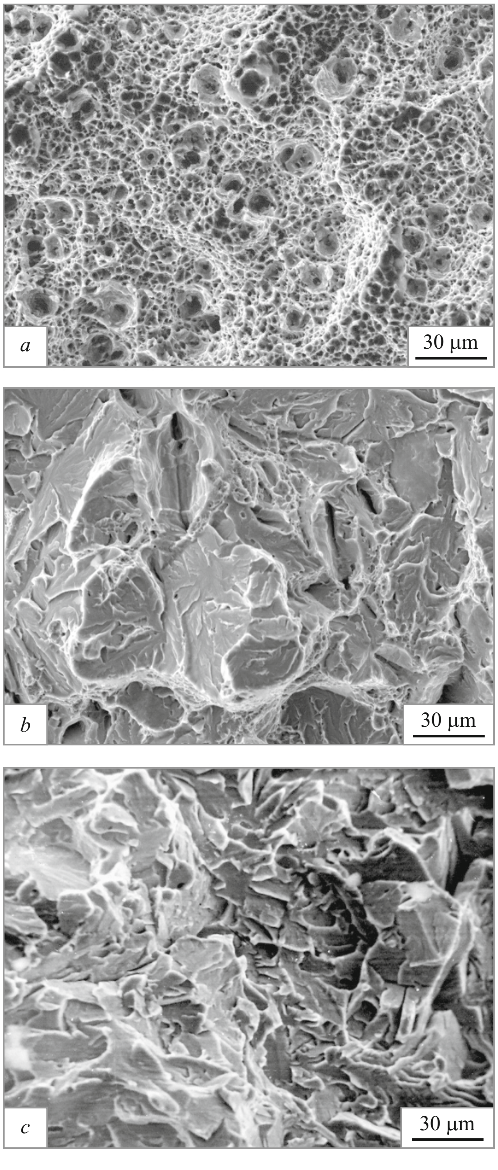

**Fig. 4.** Fracture surfaces of Fe – Cr – Ni – Mo maraging steels after quenching from 1050°C, 3-h aging at 500°C, and testing for impact toughness: *a*) steel ChS92 (heat *9*); *b*) steel ChS92 (heat *12*); *c*) steel ÉP832 (heat *13*).

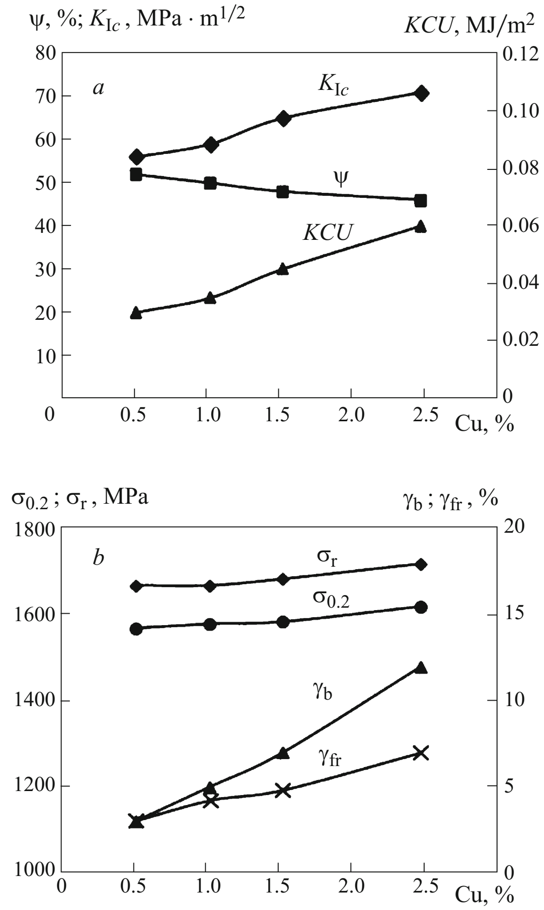

**Fig. 5.** Dependence of mechanical characteristics ($\sigma_r$ , $\sigma_{0.2}$ , δ), impact toughness (*KCU*), static crack resistance ($K_{1c}$), and content of austenite phase in the bulk of a specimen ($\gamma_b$) and in a fracture ($\gamma_{fr}$) on the copper content in model steel 02Kh11N10TYu.

ture surface of steel *9* with maximum values of impact toughness and crack resistance ($KCU = 0.6$ MJ/m$^2$ and $K_{1c} = 77$ MPa · m$^{1/2}$). The fracture surface has a fully dimple texture. On the fracture surface of steel *12* with lower values of $KCU = 0.15$ MJ/m$^2$ and $K_{1c} = 52$ MPa · m$^{1/2}$ and a little number of dimples the dominant mechanism of fracture is quasi-cleavage (Fig. 4*b*). The fracture surface of steel *13* with minimum values of $KCU = 0.05$ MJ/m$^2$ and $K_{1c} =$ 34 MPa · m$^{1/2}$ has a typically brittle structure with manifested facets of quasi-cleavage and river-line fracture elements (Fig. 4*c*).

We studied the effect of copper additives in MS of type Kh11N10M2T on the phase composition, mechanical properties and fracture resistance characteristics for heats *1* – *4* with virtually equal contents of the other alloying elements. The data of Fig. 5 show that the growth in the copper content from 0.52 to 2.48 wt.% in the steels after quenching from 1050°C and 3-h aging for maximum strength at 500°C promotes simultaneous increase in the strength, impact toughness and static crack resistance at minor decrease in the contraction. The growth in the strength characteristics of the steels with elevated copper content may be connected with the hardening action of the ε-phase represented by virtually pure copper and weakening of its embrittling action in the presence of an austenitic phase [10]. The accompanying increase in the impact toughness and static crack resistance (Fig. 5*a*) is a result of the growth in the content of retained austenite in the structure from 2.5 to 12%, the plasticizing action of which is connected with localization of austenite layers over boundaries of martensitic packets [11]. The additional effect of the martensitic phase on the properties of the MS is connected with its deformation instability. By the data of [1, 4] the retained and reverted austenite present in the structure of maraging steels may transform into strain martensite. By the data of the x-ray diffraction analysis about 50% of retained austenite in Fig. 5*b* transforms into strain $\alpha'$-martensite, and this promotes growth in the characteristics of brittle fracture strength by analogy with TRIP-steels [12, 13].

The effect of the temperatures of quenching and aging on the mechanical properties and characteristics of crack resistance has been studied for steels ÉP678, ÉP679 and ÉP832 (heats *10*, *11* and *13* respectively). It can be seen from the data of Table 2 that steels ÉP832 and ÉP678 exhibit a common tendency to lowering their plasticity, impact toughness and static crack resistance upon growth in the temperature of heating for quenching from 850 to 1050°C. This tendency is the most obvious for steel ÉP832 with minimum total level of plasticity, impact toughness and crack resistance. Accelerated heating to 820°C after quenching and subsequent aging for 7 min and cooling at room temperature produces 13 and 10% retained austenite in the structure of steels ÉP832 and ÉP678 respectively (Table 2). The partial transformation of the retained austenite into strain martensite in the fracture zone promotes growth in the plasticity, impact toughness and crack resistance of the steels. However, the resistance of steel ÉP832 to brittle fracture remains at the initial low level corresponding to the domain of damaged structural state even after the treatment for retained austenite at excess alloying with titanium and aluminum ($\sum$(Ti + Al) = 2.21 wt.%). It is known [1, 2, 14 – 17] that MS of type 03Kh11N10M2T after quenching have a structure of dislocation lath martensite and contain up to 10% retained austenite and a low content of primary carbonitrides. The succession of structural changes in the steels upon increase of the heating temperature may be represented as follows: formation of a metastable $\beta$-$Ni_3Ti$ phase (400 – 480°C), formation of a stable $\eta$-$Ni_3Ti$ phase (480 – 560°C), appearance of reverted austenite (590 – 680°C), formation of $\lambda$-$Fe_2$(Ti, Mo) and $\chi$-(Fe, Ni)$_{38}Cr_{10}Ti_6$ Laves phases (600 – 850°C), refinement of austenite grains at 850°C, and growth of austenite grains at 900°C and higher temperatures.

**TABLE 2.** Mechanical Properties and Content of γ-Phase after Quenching and Aging at 500°C (3 h) for Steels ÉP832 and ÉP678

| $t_q$, °C | $\sigma_{0.2}$, MPa | $\sigma_r$, MPa | δ, % | ψ, % | *KCU*, MJ/m$^2$ | $K_{1c}$, MPa · m$^{1/2}$ | γ-phase, % |
|---|---|---|---|---|---|---|---|
| Steel ÉP832 (heat *13*) | | | | | | | |
| 850 | 1830 | 1880 | 7.5 | 39 | 0.15 | 37 | 0 |
| 950 | 1810 | 1885 | 7.0 | 38 | 0.10 | 36 | 0 |
| 1050 | 1790 | 1920 | 5.0 | 17 | 0.05 | 34 | 5/3 |
| 1050 + 820* | 1730 | 1760 | 6.5 | 23 | 0.12 | 40 | 13/7 |
| Steel ÉP678 (heat *10*) | | | | | | | |
| 850 | 1530 | 1590 | 12 | 59 | 0.80 | 69 | 0 |
| 1050 | 1520 | 1570 | 11 | 57 | 0.55 | 63 | 3/3 |
| 1050 + 820* | 1505 | 1555 | 14 | 63 | 0.95 | 75 | 10/5 |

* Quenching from 1050°C in air, accelerated heating to 820°C, and cooling in air to room temperature.

**Note.** The numerators present the content of γ-phase in the specimen; the denominators present its content in the fracture.

**TABLE 3.** Characteristics of Impact Toughness and Dynamic Crack Resistance of Steels ÉP687 and ÉP679 after Quenching from 920°C and Aging

| Aging mode | $A_p/A_n$ | *KCV*, MJ/m$^2$ | $J_{1d}$, MJ/m$^2$ |
|---|---|---|---|
| ÉP678 (heat *10*) | | | |
| 500°C, 3 h | 0.53 | 0.45 | 0.89 |
| ÉP679 (heat *11*) | | | |
| 500°C, 3 h | 0.06 | 0.10 | 0.21 |
| 540°C, 3 h | 0.10 | 0.125 | 0.30 |
| 580°C, 3 h | 0.69 | 0.38 | 0.45 |
| 620°C, 3 h | 1.88 | 1.07 | 0.73 |

The temperatures of 850 and 1050°C may be treated as those of the start of recrystallization and secondary recrystallization, respectively, for the studied MS. Thus, the lowering of the parameters of plasticity, impact toughness and crack resistance in steels ÉP832 and ÉP678 with growth of the temperature of heating for quenching to 1050°C may be associated with growth of the austenite grains, the size of which after such a treatment, by the data of the metallographic analysis, attains 90 and 130 μm respectively.

It can be seen from Table 1 and Fig. 6 that the different contents of the Mo, Ti and Al hardening elements are responsible for higher values of resistance to plastic deformation after aging at 450 – 525°C and, accordingly, lower values of static crack resistance $K_{1c}$ in steel ÉP679 (heat *11*) than in steel ÉP678 (heat *10*). Still higher differences in the resistance to brittle fracture have been detected in the tests of *V*-notched specimens for impact toughness and dynamic crack resistance. According to the data of Table 3 after aging at 500°C the values of *KCV* and J1d and the ratio $A_p/A_n$ of the energies of crack propagation and crack nucleation characterizing the margin of structural strength differ by a factor of 4 – 9. The level of the impact toughness and dynamic crack resistance of steel ÉP678 aged at 500°C can be attained in steel ÉP679 only after softening aging at a temperature exceeding 580°C.

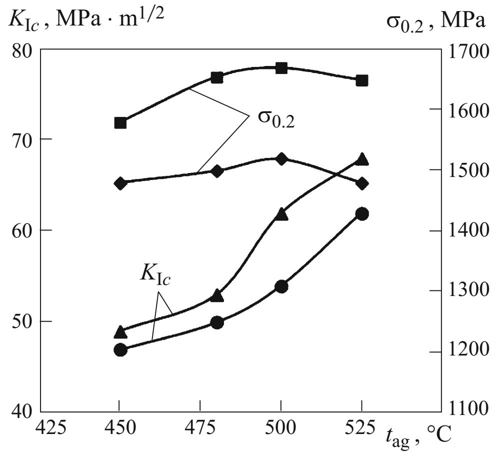

**Fig. 6.** Conventional yield strength $\sigma_{0.2}$ and parameter of static crack resistance $K_{1c}$ as a function of the aging temperature of steels ÉP679 (■, ●) and ÉP678 (◆, ▲).

## CONCLUSIONS

1. We have studied the effect of alloying with elements forming hardening intermetallic particles and with copper on the phase composition, combination of mechanical properties and characteristics of resistance to brittle fracture for 13 heats of maraging steels (MS) based on the Fe – Cr – Ni – Mo system.

2. Increase in the summed content of titanium and aluminum in the MS within 0.99 – 2.21 wt.% causes linear growth of their strength characteristics to $\sigma_{0.2} \le 1790$ MPa and $\sigma_r \le 1900$ MPa at monotonic decrease in the impact toughness and static crack resistance.

3. The relation between *KCU* and $\sum(\mathrm{Ti} + \mathrm{Al})$ may be approximated by a power law, and the trend lines of $K_{1c}$ are describable by a second-power polynomial function.

4. Growth in the copper content from 0.52 to 2.48 wt.% (test steels *1 – 4* ) results in their synergistic hardening manifested in simultaneous growth of the strength properties, impact toughness and static crack resistance. The elevated strength properties and resistance to brittle fracture are provided by the elevated content (12%) of metastable retained austenite in the structure of the steels.

5. Increase in the temperature of heating for quenching from 850 to 1050°C causes decrease in the plasticity, impact toughness and crack resistance due to growth of the austenite grains. Metastable retained austenite (10 – 13 wt.%) produces positive effect on the resistance of the steels to brittle fracture after accelerated heating to the $\gamma$-range with subsequent quenching.

6. Steel ÉP679 in aged condition has higher strength characteristics than steel ÉP678 at a comparable level of static crack resistance but lower impact toughness and dynamic crack resistance.

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