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Study of the Effect of Quenching and Tempering Modes on the Strength Level of Alloyed Structural Steels Used to Produce Fasteners

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Abstract: The development of high-strength fasteners is required due to the modern trend of reducing the weight of engineering structures. At present, structural bolts of property classes 8.8–10.9 are widely used, and higher-strength bolts of property classes 12.9 and 14.9 are being developed. In this paper, we analyzed the possibilities and conditions for manufacturing fasteners of different strength categories from steels of seven grades, containing and not containing boron. The necessary corresponding temperature ranges of processing are determined based on the study of hot-rolled round steel. Tensile strength, yield strength, relative elongation, reduction in area, and Vickers microhardness were measured. An analysis was made of the hardenability and the influence of the holding time during tempering on the mechanical properties of the considered round steel. Using the method of scanning electron microscopy, the effect of carbide precipitation during tempering on the mechanical properties of fasteners was established.

Keywords: high-strength fasteners; round bolts; property class 12.9; quenching; tempering; austenitization; mechanical properties

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1. Introduction

Modern trends in reducing the weight of engineering structures to save both material and energy have led to the development of lighter and stronger materials, including in the automotive industry [1]. In turn, this require high-strength fasteners that can reduce the size of the fastening element. The traditional process of their manufacture is upsetting and/or cold forging of steel wire [2]. To obtain high indicators of the mechanical properties of finished products, they are subjected to final heat treatment, including austenitization, quenching, and tempering [3]. In the last decade, structural bolts of property classes 8.8, 9.8, and 10.9 have been widely used [4]. At the same time, a number of studies have been carried out to develop higher-strength bolts of classes 12.9 and 14.9 (tensile strength of at least 1220–1400 MPa, with a yield strength coefficient of more than 0.9) [5–7]; however, the available information for the selection of steel grades and their mass production is not enough.

In global practice, in the production of high-strength bolts, medium-carbon and alloyed steels containing boron, manganese, and chromium are usually used, for example, 10B21 [3], 20MnTiB [3], 32CrB4 [3,8], 33B2 [6], 34Cr4 [3,6], and 34CrNiMo6 [5]. These alloying elements increase strength and hardenability. A feature of alloying with boron is a significant increase in hardenability at very low concentrations (thousandths of a percent) [9,10]. At the same time, the implementation of the positive effect of boron in practice causes problems associated with the difficult-to-control behavior of this element in steel, due to its high tendency to form boron oxide, nitride, and carboborides. In fact, only that part of the boron present in the steel, which is in a solid solution, contributes to the increase in hardenability, while the formation of a noticeable amount of boron oxide, nitride, and carboborides leads to a decrease in hardenability [11]. This feature of boron-containing steels leads to an instability of strengthening by quenching of the parts, and the production of boron-containing steels is associated with certain difficulties. Among them, there are the difficulties of a metallurgical nature: the need to obtain strictly normalized concentrations in the process of steel smelting, maintaining the content of nitrogen, titanium, and aluminum at a given level. In addition, there are still no scientifically based principles for choosing a heat-treatment mode depending on the technological features of manufacturing and the content of boron and carbon. In many cases, boron steels do not have sufficient hardenability to make fasteners of a large size (diameter).

Therefore, not only boron-containing steels for the manufacture of high-strength bolts are currently being developed, but also boron-free steels. The most studied steel is 42CrMo [12–14]. Few works are devoted to steels with a lower carbon content, for example, 35CrMo [15] and 34CrNiMo6 [5,16]. At the same time, the implementation of hardening mechanisms in these steels is different and requires additional research. For 42CrMo, its higher carbon content promotes the formation of more carbide, and finer grains are obtained after quenching [15]. However, by varying the tempering temperatures after quenching, it becomes possible to change the amount of carbide precipitates, thereby changing the contribution of precipitation strengthening and achieving different levels of mechanical properties.

This work was aimed at studying the possibilities and conditions for manufacturing fasteners of different strength categories from seven different steel grades, containing and not containing boron.

2. Materials and Methods

Hot-rolled round steels of industrial production, with a diameter of 22, 21, and 19 mm from steel of grades 42CrMo4, 32CrB4, and 40KhN2MA, respectively, as well as of laboratory production with a diameter of 20 mm from steel of grades 34CrNiMo6, 31CrMoB2, 32KhGMR, and 35KhNMFA, were studied. The chemical composition is presented in Table 1. The 32KhGMR and 35KhNMFA steels are experimental, as their composition was first developed in this work. All other steels are commonly used for the manufacture of fasteners.

Table 1. Chemical composition of the studied steels (wt.%).

Steel	C	Si	Mn	S	P	Cr	Ni	Cu	Mo	Al	Ti	V	B	N	Ca
32CrMo4	0.41	0.24	0.7	0.002	0.010	1.02	0.04	0.04	0.198	0.026	0.002	0.005	-	0.009	0.0017
32CrB4	0.32	0.21	0.65	0.003	0.010	0.91	0.03	0.04	0.002	0.024	0.040	0.005	0.0024	0.008	0.0026
40KhN2MA	0.39	0.25	0.53	0.002	0.008	0.67	1.27	0.03	0.17	0.025	0.002	0.003	-	0.008	0.0030
34CrNiMo6	0.38	0.21	0.66	0.005	0.005	1.53	1.3	0.02	0.29	0.02	-	-	-	0.007	-
31CrMoB2	0.29	0.30	0.94	0.005	0.005	0.53	0.18	0.02	0.15	0.02	0.04	-	0.006	0.007	-
32KhGMR	0.34	0.24	1.12	0.005	0.005	0.54	0.31	0.02	0.35	0.01	0.03	-	0.006	0.007	-
35KhNMFA	0.33	0.35	0.91	0.005	0.005	1.08	1.5	0.02	0.43	0.14	0.07	0.16	-	0.007	-

The austenitization temperature (Table 2) for each steel grade was determined using proprietary software (Bardin TsNIIChermet, Moscow, Russia) based on data from the steel atlas [17]. The temperature at which homogeneous austenite should form for the case of metal heating at a rate of 1 °C/s was taken as the austenitization temperature, which approximately corresponds to the conditions for achieving a uniform temperature distribution over the cross section of hardened fasteners in a furnace for 13–15 min. Since, under real conditions, the temperature distribution in the furnace may differ from the theoretical calculation, a positive temperature margin of 10–15 °C was created.

Table 2. Austenitization temperature values for the studied steels.

Steel	42CrMo4	32CrB4	40KhN2MA	34CrNiMo6	31CrMoB2	32KhGMR	35KhNMFA
T_{aust}, °C	845	860	850	850	860	850	850

To analyze the hardenability, samples with a length of 150 mm were heated in a laboratory tube resistance furnace SUOL-0.4.4/12 (Tulaterm, Tula, Russia) to the selected research temperature for 15 min, followed by holding for 45 min, and then quenched into oil. Cylinders with a height of 5 mm and a diameter corresponding to rolled products were cut from the obtained samples from the middle along the length, and microsections were made to study microhardness using a Struers Duramin-20 tester (Struers ApS, Copenhagen, Denmark). Vickers microhardness was measured in three different areas of the microsection, according to the scheme shown in Figure 1.

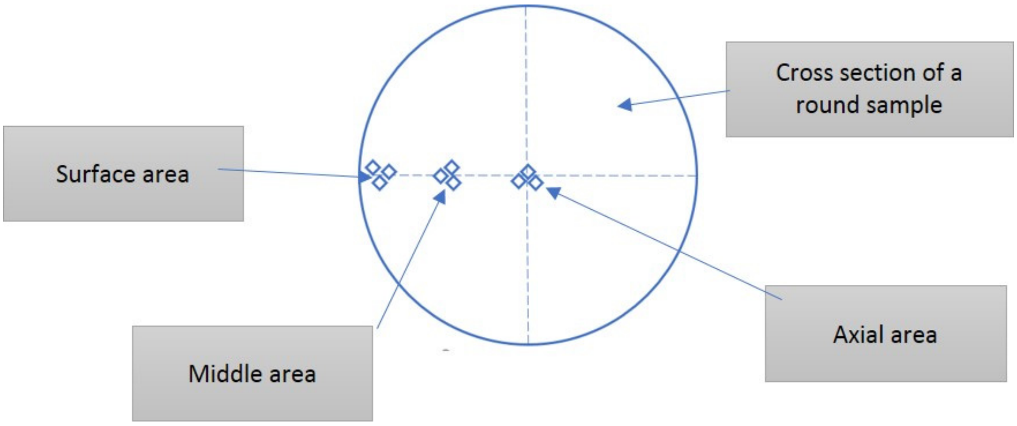


Figure 1. Conventional scheme of microhardness measurements on the cross section of a round bar sample.

Samples of hot-rolled steels with a length of 65 mm were subjected to austenitization, according to the selected mode, followed by oil quenching. After quenching, the samples were tempered in a laboratory tube resistance furnace at temperatures of 450, 500, and 550 °C for 60 min. The tempering temperatures were chosen on the basis of the results [8], which showed that when the tempering temperature of 32CrB4 rolled products decreases, especially below 450 °C, the relative elongation sharply decreases. In addition, the formation of the microstructure of round rolled products made of 40KhN2MA grade steel was studied after tempering at temperatures from 400 to 550 °C.

To substantiate the chosen duration of tempering, the influence of the holding time in the furnace was studied, which was 30, 45, 60, 75, and 90 min. Samples with a diameter of 5 mm and a length of 25 mm were turned from the obtained samples to determine the mechanical properties. The measurement was carried out on an INSTRON-150LX tensile testing machine (Illinois Tool Works, Inc., Norwood, MA, USA).

The microstructure was studied using scanning electron microscopy on a JEOL JSM-6610LV microscope (JEOL Ltd., Tokyo, Japan).

3. Results and Discussion

3.1. Analysis of the Hardenability of Rolled Products under Study

Table 3 shows the results of the study of the hardenability of rolled products of all considered steels, based on the measurement of microhardness, according to the scheme shown in Figure 1.

Table 3. Results of measurement of Vickers microhardness on samples of hot-rolled studied steel after quenching.

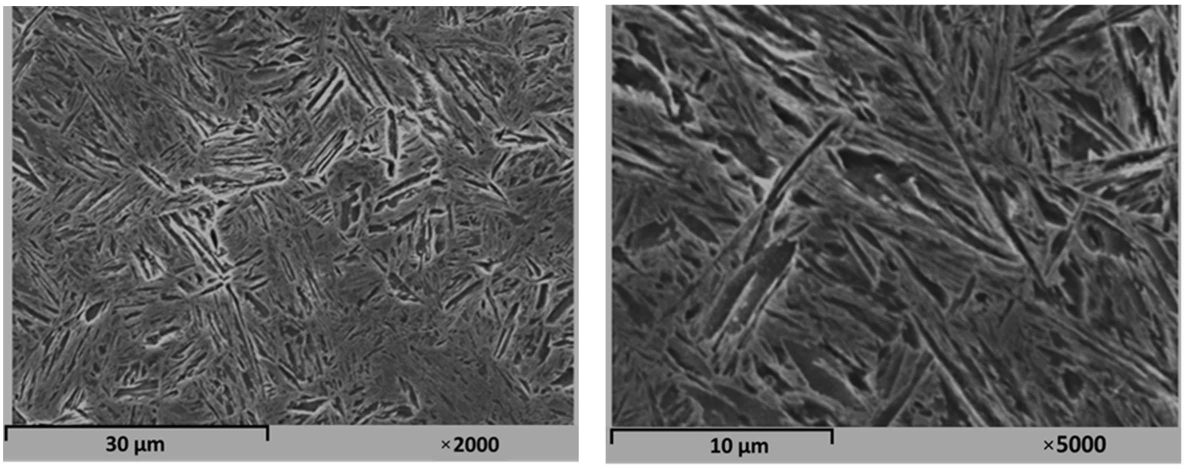
Steel	Measurement Number	Microhardness Value, HV				Deviation of HV in the Axial Zone from the Average, %
		Measurement Area			Sample Average	
		Surface	Middle	Axial		
42CrMo4	1	660	659	644	656.3	−1.9
	2	655	666	638		
	3	664	672	649		
32CrB4	1	547	560	556	549.9	−0.1
	2	546	546	542		
	3	544	557	552		
40KhN2MA	1	602	628	601	610.6	−0.9
	2	604	625	605		
	3	599	621	610		
34CrNiMo6	1	627	629	635	632.9	0.9
	2	632	636	648		
	3	625	632	632		
31CrMoB2	1	538	552	546	545.7	0.7
	2	537	553	555		
	3	532	550	548		
32KhGMR	1	598	585	600	598.1	0.6
	2	604	591	598		
	3	606	594	607		
35KhNMFA	1	604	596	604	601.7	0.8
	2	597	606	603		
	3	598	595	612		

From the data in Table 3, it can be seen that for all the studied samples of rolled products, a uniform distribution of hardness is observed, namely there is no regular decrease in hardness from the surface to the axial zone by more than 5% (relative). At the same time, for samples of all considered steels, including those not containing boron (40KhN2MA, 34CrNiMo6, 35KhNMFA), except for 42CrMo4 steel, the deviation is less than 1%. This indicates a sufficiently high hardenability of the studied steels.

3.2. Regularities of Influence of Temperature and Duration of Tempering

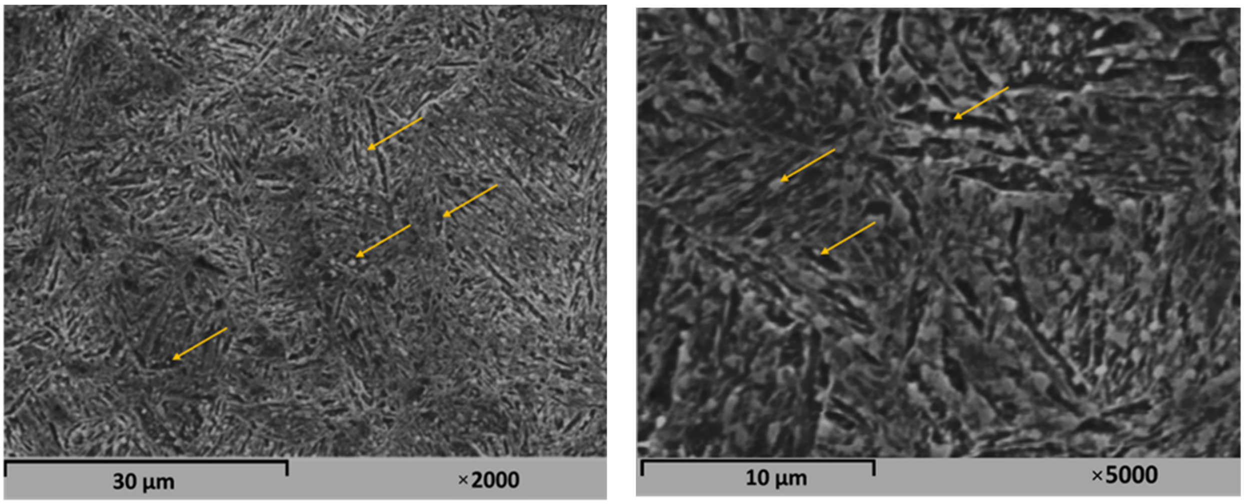
To substantiate the optimal temperature and time parameters of tempering, the effect of tempering temperature on the formation of the microstructure of round rolled products from 40KhN2MA grade steel, after quenching and tempering at temperatures from 400 to 550 °C, was studied. Figure 2 shows the microstructure of 40KhN2MA steel after austenitization at a temperature of 850 °C for 45 min and subsequent oil quenching.

Figure 2 shows that after the austenitization, rolled products made of 40KhN2MA grade steel have a uniform martensitic structure. Figure 3 shows a view of the microstructure of this steel at tempering temperatures of 400, 425, 450, 500, and 550 °C for one hour.



(**a**) (**b**)

Figure 2. Microstructure of a round-rolled sample from 40KhN2MA grade steel after austenitization at a temperature of 850 °C for 45 min and subsequent oil quenching: (**a**) magnification ×2000 and (**b**) magnification ×5000.



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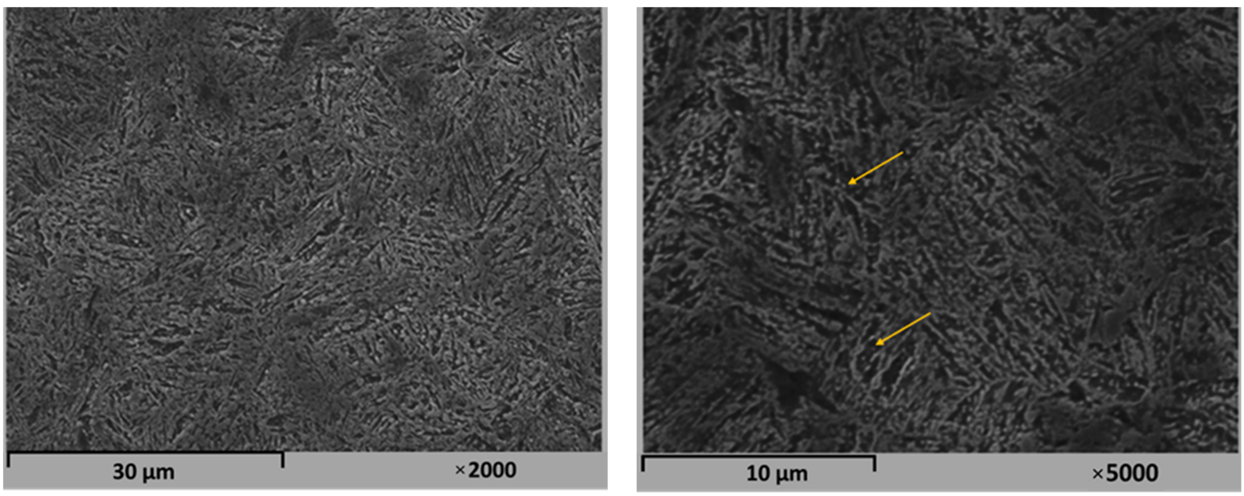


(**c**) (**d**)

Figure 3. *Cont.*

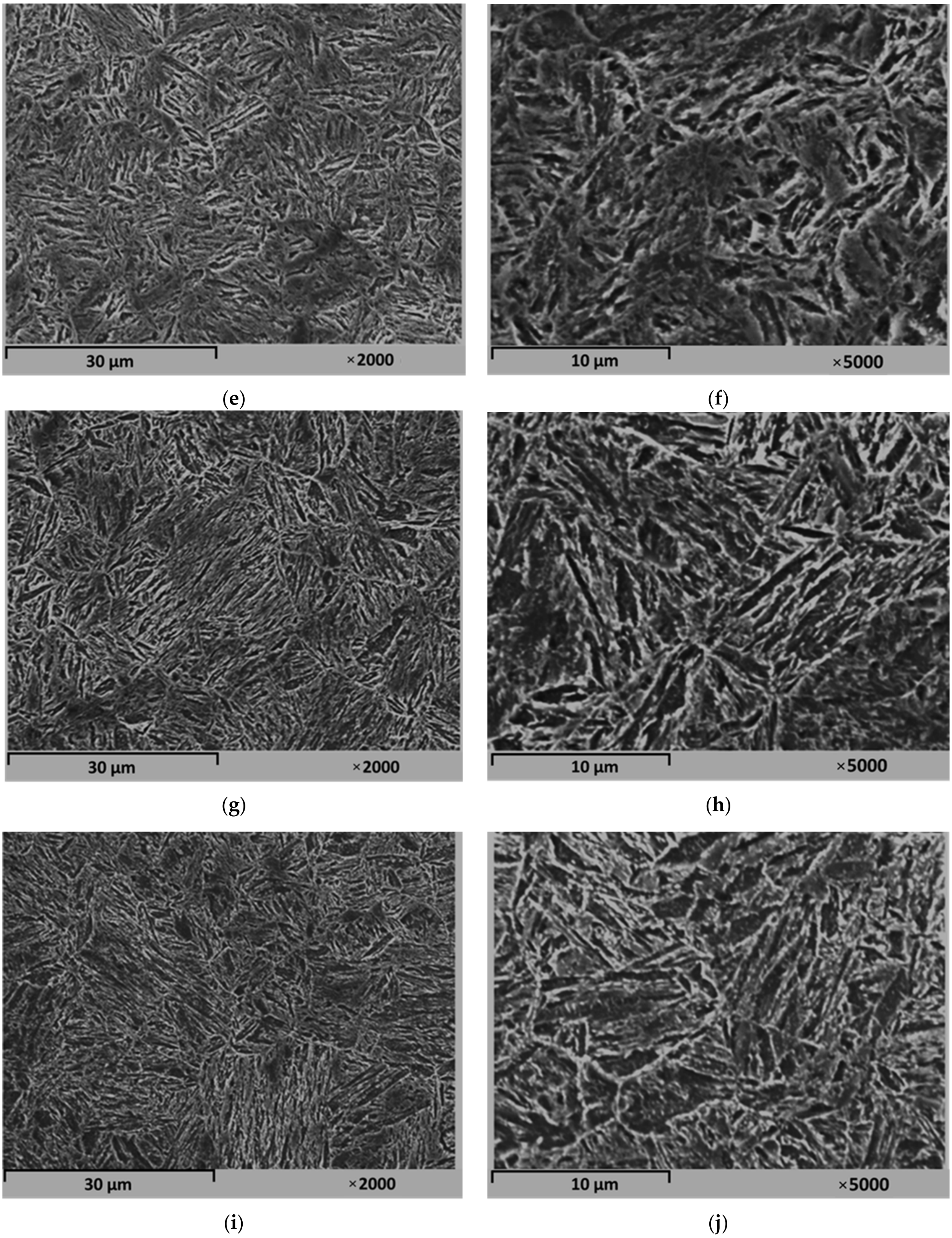


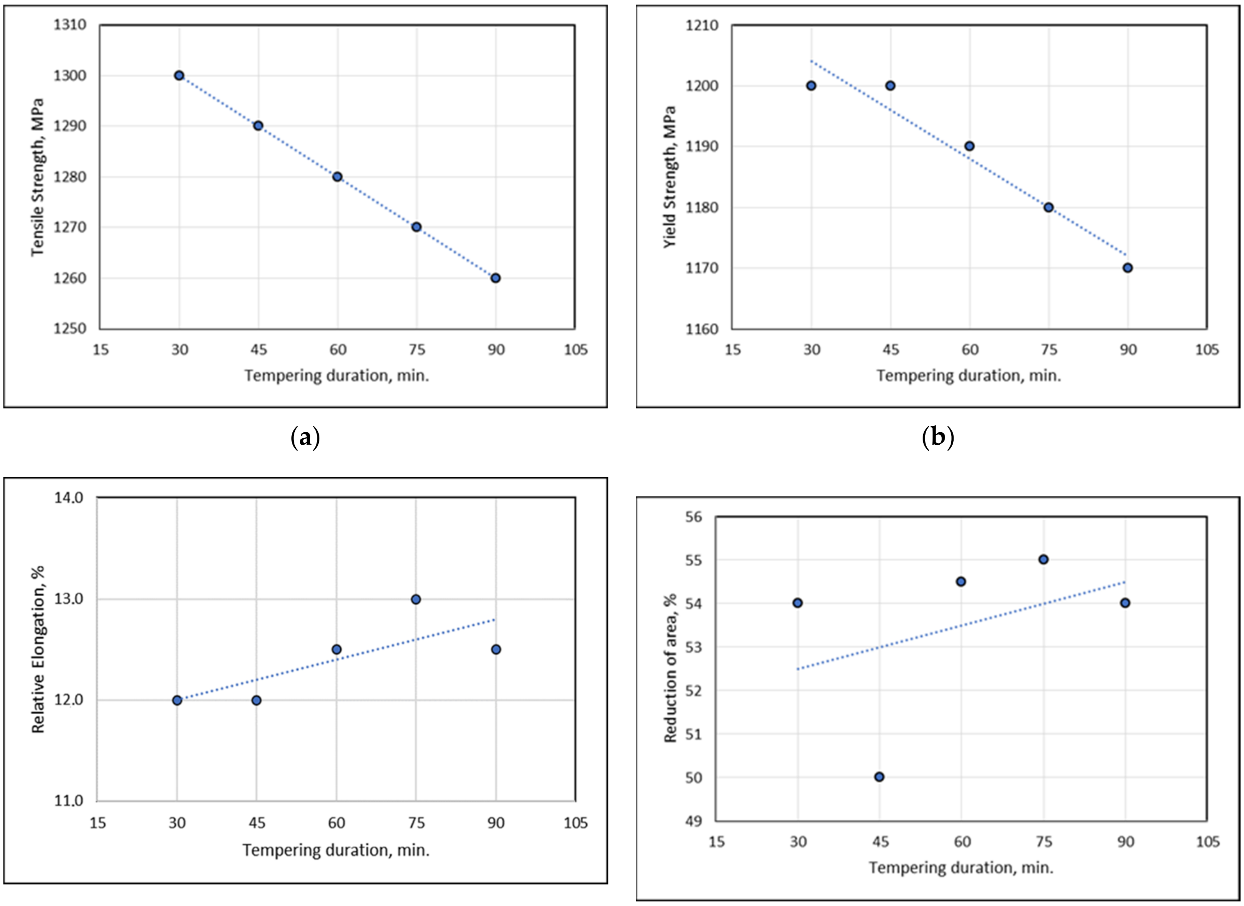
Figure 3. Microstructure of a round-rolled sample made of 40KhN2MA grade steel after quenching and tempering for 1 h at a temperature of: (**a**,**b**) 400 °C; (**c**,**d**) 425 °C; (**e**,**f**) 450 °C; (**g**,**h**) 500 °C; and (**i**,**j**) 550 °C. Arrows point to carbides.

It can be seen from Figure 3 that at tempering temperatures of 400 and 425 °C, only the initial stage of the formation of submicron carbide precipitates is observed directly inside the martensite. Starting from a temperature of 450 °C and higher, a sharp increase in the intensity of the formation of carbide precipitates along the boundaries of martensite blocks is seen, which compensates for the drop in strength due to the gradual decomposition of martensite. This is certainly more preferable, since internal stresses in the metal are significantly reduced, which can adversely affect the mechanical and other service properties of high-strength fasteners. Thus, the data obtained explain the decrease in the plasticity of round bars at tempering temperatures below 450 °C [8].

In order to determine the optimal duration of tempering, its influence on the mechanical properties of hot-rolled round bars made of 42CrMo4 steel was studied. The results of the determination of mechanical properties are presented in Table 4 and Figure 4.

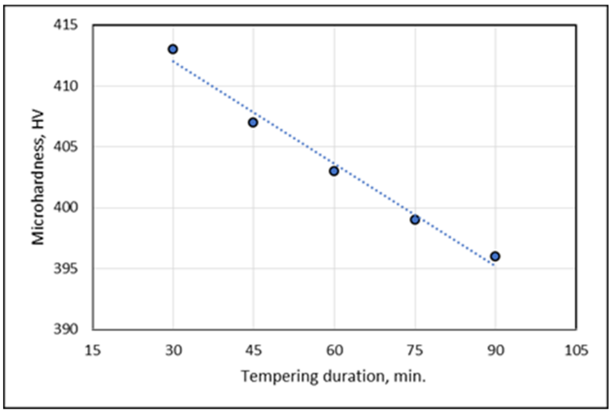
Table 4. Mechanical properties of samples of hot-rolled 42CrMo4 grade steel after quenching and tempering at 500 °C after various durations.

Tempering Durations, min	Mechanical Properties				
	$\sigma_{0.2}$, MPa	σ_B, MPa	δ, %	Ψ, %	HV
30	1200	1300	12.0	54.0	413
45	1200	1290	12.0	50.0	407
60	1190	1280	12.5	54.5	403
75	1180	1270	13.0	55.0	399
90	1170	1260	12.5	54.0	396



(c) (d)

Figure 4. *Cont.*



(**e**)

Figure 4. Dependences of mechanical properties of rolled products from 42CrMo4 grade steel on the duration of tempering at 500 °C: (**a**) tensile strength; (**b**) yield strength; (**c**) relative elongation; (**d**) reduction in area; and (**e**) microhardness HV.

From the data in Table 4 and Figure 4, it follows that in the entire investigated tempering time interval at a temperature of 500 °C, the required mechanical properties for property class 12.9 are achieved. However, it should be noted that with a tempering duration of 45 min, a decrease in the reduction in area value was detected. Since, when tempering for 60–90 min is used at the studied temperature, the necessary strength properties are achieved, and the plasticity of the metal increases slightly, it is advisable to use the duration of tempering in this range.

3.3. Investigation of the Possibility and Conditions for Manufacturing Fasteners of Various Property Classes from the Studied Steels

The results of determining the mechanical properties of samples of hot-rolled round bars from the studied steels after quenching and tempering at various temperatures are presented in Table 5.

From Table 5, it follows that the property class 12.9 corresponds to rolled products from steels 42CrMo4, 34CrNiMo6, and 32KhGMR, after tempering at temperatures of 450 and 500 °C, and rolled products from steels 40KhN2MA, 31CrMoB2, and 32CrB4, after tempering only at a temperature of 450 °C. It should be noted that for 34CrNiMo6 steel in [5], containing wt.% 0.035 C–0.649 Mn–0.129 Si–1.616 Cr–1.554 Ni–0.250 Mo–0.013P–0.008 S, similar results were obtained after tempering for 60 min at 520 °C. The tensile strength, yield strength, and reduction in area were 1300 MPa, 1230 MPa, and 61%, respectively.

Rolled products from 35KhNMFA steel mainly meet the requirements for ultra-high-strength property class 14.9, with an obvious possibility of obtaining fasteners of property class 12.9 at a tempering temperature of 550 °C and higher. The lowest strength properties are found for steels 32CrB4 and 31CrMoB2. After tempering at temperatures of 500 °C, they correspond to property class 10.9.

In [6], KNDS4 steel of a similar composition was studied, with wt.% 0.39 C–0.45 Mn–0.05 Si–0.004 P–0.006 S–0.033 Al–1.07 Cr–1.09 Mo–0.042 Ti–0.085 V. After austenitization at 925 °C for 60 min and quenching and tempering at 550 °C for 90 min, the tensile strength and yield strength were obtained as higher than those of 35KhNMFA steel: 1504 MPa and 1267 MPa, respectively. At the same time, the ratio of yield strength to tensile strength was only 0.84, while for 35KhNMFA steel, this value is 0.95. The authors attribute the high tensile strength to the high austenitization temperature and the complete dissolution of alloying elements during the austenitizing treatment, which increased the volume fraction of carbides precipitated during tempering. Apparently, this was also facilitated by a higher content of carbon and molybdenum. According to [18–20], the presence of molybdenum in the steel composition facilitates the nucleation and inhibits the growth of carbide precipitates, which increases the contribution of dispersion strengthening.

Table 5. Mechanical properties of hot-rolled round bars after quenching and tempering at various temperatures.

Steel	Tempering Temperature, °C	Diameter, mm	Mechanical Properties				
			$\sigma_{0.2}$, MPa	σ_B, MPa	δ, %	Ψ, %	HV
42CrMo4	450	22	1320	1400	12.0	53	445
	500		1200	1280	14.0	55	404
	550		1080	1180	15.0	58	376
32CrB4	450	21	1200	1270	14	62	390
	500		1050	1110	17	63	342
	550		880	960	19	66	297
40KhN2MA	450	19	1210	1300	14.0	54	407
	500		1110	1210	15.5	62	379
	550		980	1080	17.0	63	341
34CrNiMo6	450	20	1330	1470	13.0	53	454
	500		1220	1330	14.5	57	417
	550		1120	1230	16.0	60	382
31CrMoB2	450	20	1220	1270	14.0	62	392
	500		1080	1130	15.5	63	353
	550		930	1000	17.0	66	313
32KhGMR	450	20	1300	1380	13.5	60	424
	500		1180	1250	15.0	60	390
	550		1060	1140	16.5	60	359
35KhNMFA	450	20	1410	1490	12.0	55	457
	500		1350	1420	14.0	55	443
	550		1300	1360	15.5	56	430
ISO 898-1 requirements for property class 10.9			≥940	≥1040	≥9.0	≥48	320–380
ISO 898-1 requirements for property class 12.9			≥1100	≥1220	≥8.0	≥44	385–435

It is important to note that the relative elongation and reduction in area for rolled products from all considered steels satisfies the requirements for property class 12.9 of fasteners in the entire investigated tempering temperature range from 450 to 550 °C. Therefore, the tempering temperature range was refined to determine the interval, at which the values of all mechanical properties are achieved, which meets the requirements for property class 12.9 of fasteners for 42CrMo4, 34CrNiMo6, 32KhGMR, and 40KhN2MA steels. For 32CrB4, 40KhN2MA, 31CrMoB2, and 32KhGMR rolled steels, tempering temperatures were also analyzed to obtain property class 10.9.

Figure 5 shows the obtained values of tensile strength and microhardness depending on tempering temperature. The horizontal lines represent the upper and lower limits of the tensile strength and Vickers hardness values for fasteners of property classes 12.9 and 10.9. The graphical analysis carried out by the method of linear interpolation made it possible to determine the upper and lower limits of the tempering temperatures at which the required mechanical properties are achieved (Table 6).

An analysis of the data in Table 6 shows that round bars made of 42CrMo4, 40KhN2MA, 34CrNiMo6, and 32KhGMR steels, meeting the requirements for property class 12.9, can be obtained after tempering in a wide temperature range: about 60–70 °C. For 31CrMoB2 rolled products, it is necessary to use an extremely narrow interval of tempering temperatures. This, due to technological difficulties, can lead to an increase in the probability of rejection by mechanical properties.

Rolled products made of 40KhN2MA and 32KhGMR steels can be used for the manufacturing fasteners of both property classes: 12.9 and 10.9. At the same time, compared with 40KhN2MA steel, 32KhGMR rolled products have a wider tempering temperature range to obtain property class 10.9. In addition, it can be assumed that due to the need to use higher tempering temperatures and a lower carbon content, products made of 32KhGMR steel will have higher service properties, including fatigue strength.

Table 6. Upper and lower limits of tempering temperatures at which mechanical properties corresponding to property classes 10.9 and 12.9 are achieved, as well as the arithmetic mean value of the found temperature range.

Steel	42CrMo4	32CrB4		40KhN2MA		34CrNiMo6	31CrMoB2		32KhGMR	
Property class	12.9	10.9	12.9	10.9	12.9	12.9	10.9	12.9	10.9	12.9
Tmin, °C	462 ± 5	467 ± 5	450 ± 5	450 ± 5	498 ± 5	476 ± 5	476 ± 5	450 ± 5	524 ± 5	450 ± 5
Tmax, °C	532 ± 5	523 ± 5	460 ± 5	485 ± 5	570 ± 5	546 ± 5	534 ± 5	465 ± 5	591 ± 5	509 ± 5
Tmean, °C	497	495	455	468	534	511	505	458	558	480

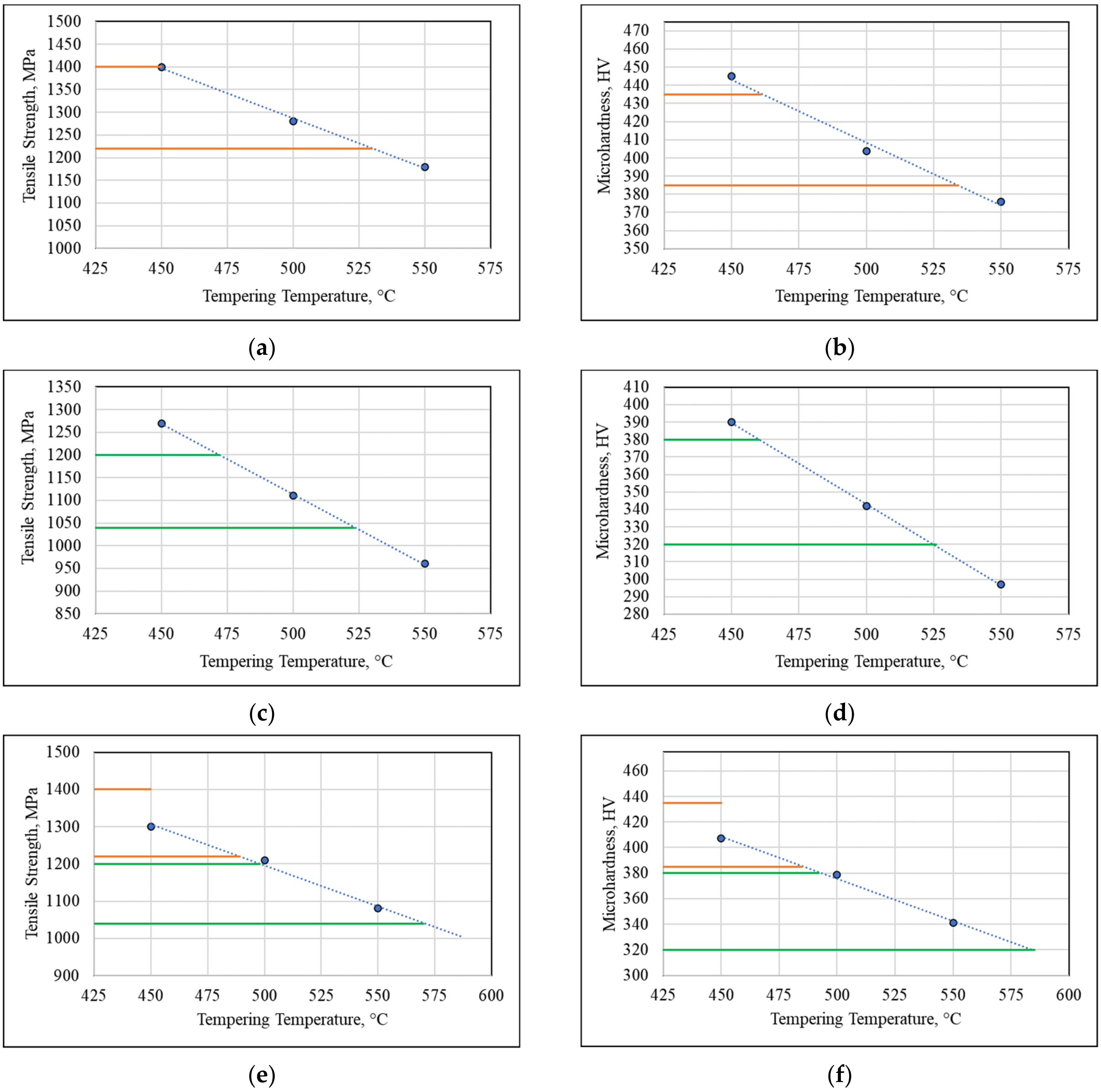


Figure 5. *Cont.*

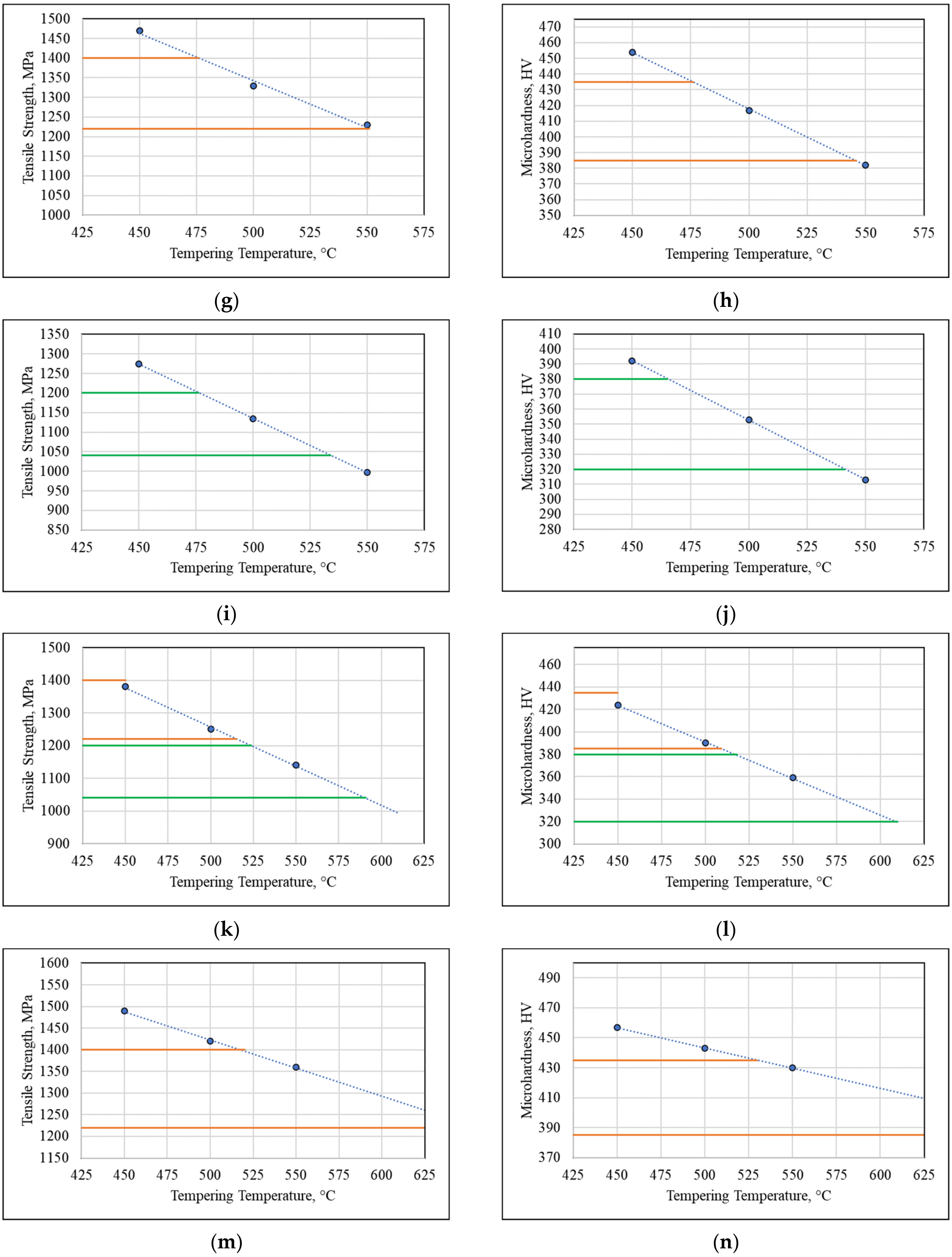


Figure 5. Dependences of the tensile strength and HV hardness on the tempering temperature of rolled products made of steel of grades: (**a**,**b**) 42CrMo4; (**c**,**d**) 32CrB4; (**e**,**f**) 40KhN2MA; (**g**,**h**) 34CrNiMo6; (**i**,**j**) 31CrMoB2; (**k**,**l**) 32KhGMR; and (**m**,**n**) 35KhNMFA. Orange horizontal lines correspond to the maximum and minimum values of properties for property class 12.9, green lines correspond to property class 10.9.

The results obtained indicate the possibility of manufacturing fasteners from 32CrB4 grade steel not only of property class 10.9, but also of lower property classes, including 8.8 and 9.8, by increasing the tempering temperature to 540–600 °C. Despite the possibility of achieving values of strength characteristics corresponding to property class 12.9, it is clear that it is necessary to use an extremely narrow range of tempering temperatures (Table 6). Therefore, it is advisable to use rolled steel of 32CrB4 grade for manufacturing fasteners of property class 10.9, and, if necessary, 8.8 and 9.8.

Currently, the required properties for fasteners of property class 14.9 are not yet included in ISO 898-1-2013 ($\sigma_B \geq 1400$ MPa, $\sigma_{0.2} \geq 1260$ MPa), so they are not shown in Figure 5m,n. The graphical analysis carried out by the method of linear interpolation showed that the achievement of the mechanical properties of 35KhNMFA rolled steel, corresponding to the property class 14.9, is ensured at a tempering temperature below 520 ± 5 °C. Thus, to obtain ultra-high-strength fasteners, the recommended tempering temperature is 480–520 °C. The obtained dependence of the decrease in strength characteristics on the tempering temperature allows us to make an assumption that, in order to obtain property class 12.9 from 35KhNMFA grade steel, it is possible to temper in a very wide range, up to 700 °C, which requires confirmation in further studies.

Thus, an analysis was made of the effect of tempering temperature on mechanical properties that meet the requirements of various property classes according to ISO 898-1-2013, for steels of various chemical compositions. Figure 6 shows a summary diagram for the studied steel grades.

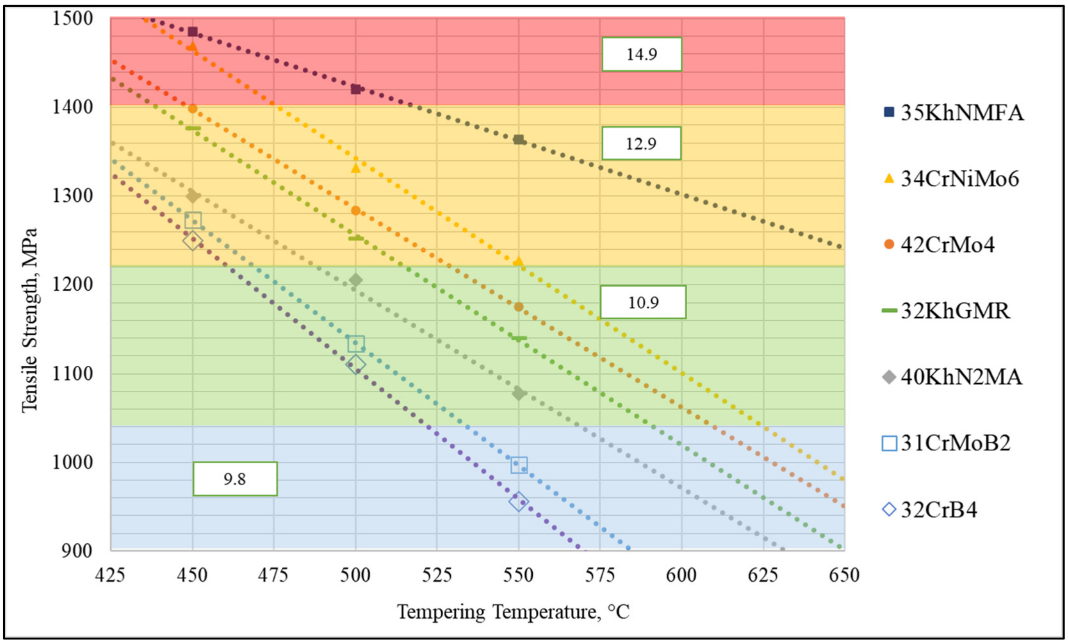


Figure 6. Dependences of the tensile strength of rolled steels of various grades on the tempering temperature.

From the data in Figure 6, it can be seen that for 35KhNMFA grade steel, the slope of the dependence of the ultimate strength on the tempering temperature is much less than for steel of all other grades. The difference between steel of grade 35KhNMFA and other grades is the presence of vanadium in its composition. An increase in tempering temperature leads to the formation of dispersed precipitates of vanadium carbonitride, which make a significant contribution to the dispersion strengthening of steel. Therefore, the loss of strength due to the tempering of martensite is largely compensated by the formation of vanadium carbonitride precipitates. This testifies to the great promise of using the highest strength categories of vanadium-containing steels for the manufacture of fasteners.

4. Conclusions

From the analysis of the data obtained, three main groups of steel grades can be distinguished.

- The first group of economically alloyed boron-containing steel grades (32CrB4 and 31CrMoB2), in a wide range of tempering temperatures, makes it possible to manufacture products of property classes 10.9 and 9.8, with the possibility of achieving properties corresponding to class 12.9 in a narrow tempering temperature range.
- The second group of steels, alloyed with chromium and molybdenum (32KhGMR, 42CrMo4) and with chromium, nickel, and molybdenum (40KhN2MA, 34CrNiMo6), makes it possible to consistently achieve mechanical properties corresponding to property class 12.9. For 32KhGMR steel grade, with an economical alloying system, it is possible to achieve high mechanical properties at a significantly lower carbon concentration.
- The third group consists of steel alloyed with chromium, nickel, molybdenum, and vanadium (35KhNMFA), the use of which makes it possible to manufacture ultra-high-strength fasteners with a tensile strength of more than 1400 MPa.

The results of studying the effect of the tempering duration of 42CrMo4 rolled steel for 30, 45, 60, 75, and 90 min at a temperature of 500 °C showed that the required mechanical properties for property class 12.9 are achieved in the entire tempering duration interval studied at a temperature of 500 °C.

The results of studying the microstructure of round-rolled samples made of 40KhN2MA grade steel after quenching and tempering at temperatures from 400 to 550 °C showed that it is advisable to restrict the lower limit of the tempering temperatures used at 450 °C.

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