www.czasopisma.pan.pl PAN www.journals.pan.pl
POLSKA AKADEMIA NAUK

ARCHIVES OF METALLURGY AND MATERIALS
Volume 59 2014 Issue 3
DOI: 10.2478/amm-2014-0205

M. OPIELA*

ELABORATION OF THERMOMECHANICAL TREATMENT CONDITIONS OF Ti-V AND Ti-Nb-V MICROALLOYED FORGING STEELS

OPRACOWANIE WARUNKÓW OBRÓBKI CIEPLNO-PLASTYCZNEJ STALI MIKROSTOPOWYCH DO KUCIA TYPU Ti-V I Ti-Nb-V

The goal of the work was to describe the forging conditions of thermomechanical treatment for Ti-V and Ti-Nb-V microalloyed steels. Conditions of hot-working allowing to obtain both the desired microstructure and mechanical properties of forgings were selected taking into consideration: precipitation analysis of MX-type (M-Nb, Ti, V; X-N, C) interstitial phases in austenite; research on the influence of the austenitizing temperature on the γ-phase grain size; investigation of the continuous compression of specimens; and examination of the kinetics of recrystallization of plastically deformed austenite. The precipitation analysis of MX-type interstitial phases in austenite was conducted on the basis of a simplified thermodynamic model for equilibrium conditions as proposed by Adrian, assuming that individual MX phases are soluble in austenite. The effect of the austenitizing temperature in a range from 900 to 1200°C on the prior austenite grain size was investigated to verify the precipitation analysis of MX-type phases. The work also presents the results of the effect of Nb, Ti and V microadditions on flow stress, recrystallization kinetics and microstructure. Plastometric tests were carried out using the Gleeble 3800 thermomechanical test simulator. The studies provide the basis for a proper design of the manufacturing process for thermomechanical treatment of forged machine parts obtained from high-strength microalloyed steels.

Keywords: microalloyed steels, thermomechanical treatment, dynamic recrystallization, forgings

Celem pracy było opracowanie warunków kucia metodą obróbki cieplno-plastycznej stali mikrostopowych typu Ti-V i Ti-Nb-V. Warunki obróbki plastycznej na gorąco, zapewniające pożądaną mikrostrukturę i własności mechaniczne odkuwek, dobrano z uwzględnieniem: analizy wydzielania się w austenicie faz międzywęzłowych typu MX (M-Nb, Ti, V; X-N, C), badań wpływu temperatury austenityzowania na wielkość ziarn fazy γ, badań ciągłego ściskania próbek oraz badań kinetyki rekrystalizacji austenitu odkształconego plastycznie. Analizę wydzielania faz międzywęzłowych typu MX w austenicie przeprowadzono na podstawie modelu zaproponowanego przez Adriana, zakładającego rozpuszczalność w warunkach równowagi termodynamicznej, indywidualnych faz MX w austenicie. Badania wpływu temperatury austenityzowania w zakresie od 900 do 1200°C na wielkość ziarn austenitu pierwotnego przeprowadzono w celu weryfikacji analizy wydzielania faz typu MX. W pracy przedstawiono także wyniki badań wpływu mikrododatków Nb, Ti i V na krzywe płynięcia, kinetykę rekrystalizacji i mikrostrukturę. Badania plastometryczne przeprowadzono przy użyciu symulatora termomechanicznego Gleeble 3800. Przeprowadzone badania stanowią podstawę prawidłowego projektowania procesu technologicznego obróbki cieplno-plastycznej kutych elementów maszyn o wysokiej wytrzymałości ze stali mikrostopowych.

1. Introduction

The technical and economic benefits that result from using microalloyed structural high-strength, HSLA-type steels with microadditions of Nb, Ti and V determine a steady increase in the demand for and development of this group of materials in many fields of technology. Products that are made from such steels with the use of thermomechanical treatment have mechanical properties that are comparable with those that are obtained from much more expensive heat-treatable alloy structural steels, and there is no need to use expensive heat treatment, or such treatment can be limited to tempering [1-3].

The chemical composition of and microstructural changes in these steels during hot working constitute the primary source of differences between the mechanical properties of the finished products. Mechanical properties in this group of materials are developed by both invoking proper strengthening mechanisms and properly designing the steel's phase composition. Due to the ever-increasing performance requirements, microalloyed steels must be characterized by high strength and at the same time by satisfactory plasticity and toughness as well as guaranteed crack resistance. In order to ensure that the product possesses the above-mentioned mechanical properties, the desired structural state of plastically deformed

---

* INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS, SILESIAN UNIVERSITY OF TECHNOLOGY, 18A KONARSKIEGO STR., 44-100 GLIWICE, POLAND

austenite must be created before the phase change takes place in the presence of dispersive particles of MX-type interstitial phases of microadditions that have been introduced into the steel [4,5].

A commonly used method of producing forgings from the steels in question has been through drop-hammer forging or press forging at a controlled temperature of charge heating and plastic deformation, followed by cooling the forgings in air. Forgings with a ferritic-pearlitic microstructure that have been made under these conditions obtain an YS of 400-750 MPa [6-8], but also a significantly lower impact strength as compared to steels that have been quenched and tempered. This may sometimes limit the possible range of their applications due to requirements regarding certain production standards. The toughness of forged products with this structure can be improved by lowering the temperature of charge heating and plastic deformation to a value that would cause only partial dissolution in a solid solution of dispersive particles of the interstitial phases. Although this slightly reduces the contribution of precipitation hardening to the strengthening of steel, it also clearly increases its impact strength, which is related to its fine-grained microstructure [9].

Elements forged in dies using the method of thermomechanical treatment by toughening low-alloy steels with microadditions of Ti, Nb and V and N or B gain higher mechanical properties, especially crack resistance, as compared with forgings with a ferritic-pearlitic microstructure. This method involves the plastic deformation of steel in conditions of controlled forging with successive quenching of forgings directly from the temperature of the forging finish, preferably after $t_{0.5}$ time that is necessary to produce a 50% fraction of statically recrystallized austenite. Direct hardening after $t_{0.5}$ time at the temperature of forging finish limits heat treatment of the forged parts only to high-temperature tempering [10,11].

Another solution which makes it possible to manufacture high-strength forgings with good toughness at lower production costs as compared to steels that have been quenched and tempered is to water-quench them directly after forging without the need for subsequent tempering. High strength (YS from 1000 to 1200 MPa) and good toughness are obtained as a result of creating a dislocation lath martensite microstructure with inter-lath films of the retained austenite in phase α [12,13].

In this study, thermomechanical treatment conditions were described for two (Ti-V and Ti-Nb-V) microalloyed forging steels.

2. Material and experimental procedure

The research was conducted on two newly elaborated microalloyed steels (Ti-V – steel A, and Ti-Nb-V – steel B). The chemical composition of the steels (Table 1) was designed by taking into consideration the production of forged elements by thermomechanical processing.

Calculations of the solubility products of MX-type phases in austenite are the first step in correctly designing the thermomechanical treatment conditions for microalloyed steels. The analysis of the precipitation of MX-type phases was done based on the simplified thermodynamic model as proposed by Adrian [14], assuming that individual MX phases are soluble in austenite. The analysis is based mainly on the equation:

$$\log\,[M]\cdot[X] = B - \frac{A}{T} \tag{1}$$

where: [M] and [X] – mass fraction of microalloy addition and interstitial atom dissolved in austenite at T temperature, respectively, A and B – constants connected to free enthalpy of MX phase formation. In order to verify the performed analysis, the effect of the austenitizing temperature on the grain size of prior austenite was investigated. Samples 25×20×32 mm in size were austenitized at a temperature of 900, 1000, 1100 and 1200°C for 30 min and then water-quenched. In order to reveal the grain boundaries of prior austenite, the samples were etched in a saturated water solution of picric acid with the addition of $CuCl_2$ at a temperature of 60°C.

TABLE 1

Chemical composition of investigated steels

Chemical composition, (%wt)							
Ti-V (steel A)							
C	Mn	Si	P	S	Cr	Ni	Mo
0.31	1.45	0.30	0.006	0.004	0.26	0.11	0.22
Nb	Ti	V	B	Cu	Al	N	O
–	0.033	0.008	0.003	0.20	0.040	0.0043	0.0006
Ti-Nb-V (steel B)							
C	Mn	Si	P	S	Cr	Ni	Mo
0.28	1.41	0.29	0.008	0.004	0.26	0.11	0.22
Nb	Ti	V	B	Cu	Al	N	O
0.027	0.028	0.019	0.003	0.20	0.025	0.0039	0.0006

Plastometric tests were carried out using the thermomechanical test simulator Gleeble 3800. Continuous compression tests of samples up to true strain $\varepsilon = 1$ were conducted in order to obtain $\sigma - \varepsilon$ curves and activation energy of plastic deformation. Specimens (φ10 x 12 mm) were resistance-heated in a vacuum at a rate of 3°C/s to a temperature of 1150°C. The samples were held at 1150°C for 30 s and cooled to a deformation temperature of 1100, 1050, 1000, 950, and 900°C. Compression of specimens was done at a strain rate of 1, 10, and 50 s^{-1}. Tantalum foils were used to prevent sticking, and graphite foils were used as a lubricant to minimize the effect of friction on the flow curves. Additionally, both surfaces were covered with a nickel-based substance. A smoothing procedure using Origin 8.0 software was used to obtain the σ-ε curves. Activation energy of plastic deformation was calculated using Energy 4.0 software, basing on the following dependence:

$$\dot{\varepsilon} = A\,[\sinh(\alpha\sigma)]^{n}\, exp\left(\frac{-Q}{RT}\right) \tag{2}$$

where: A, α, n – constants, $\dot{\varepsilon}$ – strain rate, T – strain temperature, Q – activation energy of the process of plastic deformation, R = 8.314 J $mol^{-1}\cdot K^{-1}$ – gas constant, σ – value of stress corresponding with the maximum value of flow stress.

Discontinuous compression tests of samples were conducted in a temperature range from 900 to 1100°C with isothermal holding of specimens between successive deformation steps for 2 to 100 s in order to determine the kinetics of recrystallization of plastically deformed austenite. The softening faction X, was determined according to the following dependence:

$$X = \frac{(\sigma_1 - \sigma_2)}{(\sigma_1 - \sigma_0)} \tag{3}$$

where: σ_0 and σ_1 – the stress necessary to initiate plastic deformation and its value at the moment of its finish in the first stage of deformation, respectively, σ_2 – the stress necessary to initiate plastic deformation in the second stage of compression after Δt time between those stages. Time $t_{0.5}$ corresponding to 50% recrystallized austenite was assessed on the basis of experimentally Avrami-type relationship.

3. Results and discussion

3.1. The analysis of the precipitation of MX-type phases

The concentration of Ti in the examined steel was selected so as to bond the whole nitrogen. Thus, VN, NbN and AlN were not included in the conducted thermodynamic analysis of single interstitial phases. In addition, AlN does not dissolve in carbonitrides because it is characterized by a different, hexagonal crystal structure. The solubility of particular phases in austenite of microalloyed steels is determined by a logarithmic dependence (Eq. 1). The calculated solubility temperatures for the phases investigated here, formed in the examined A and B microalloyed steels, are presented in Table 2. Taking into consideration the temperature factor of the solubility products of the analyzed phases, which can potentially be formed in the investigated microalloyed steels, it can be found that TiN, TiC and VC type phases precipitate in steel A and in steel B – also NbC. The maximum temperatures of dissolution for TiN-type interstitial phases in γ solution are equal to 1350 and 1331°C – for steel A and B, respectively. It can be assumed that the use of a high austenitizing temperature – close to 1200°C – will not cause significant grain growth of austenite through the undissolved TiN fraction. This creates the possibility of using high temperature of charge heating and forging, which has a beneficial influence on the durability of the forging tools.

TABLE 2
Summary of solubility temperatures of individual interstitial phases

No	Type of MX phase	Constants in Eq. (1)		Solubility temperature, °C	
		A	B	Ti-V (steel A)	Ti-Nb-V (steel B)
1.	TiN	15490	5.19	1350	1331
2.	TiC	10745	5.33	1188	1167
3.	NbC	7900	3.42	–	1137
4.	VC	9500	6.72	734	776

The NbC carbide, precipitating in austenite of steel B with a maximum solubility temperature equal to 1137°C will essentially contribute to steel strengthening through grain refinement and precipitation hardening with a simultaneous decrease in the ductile-to-brittle transition temperature. In turn, the VC carbide will be completely dissolved in austenite of the examined steels in the range of the hot-working temperature.

The effect of the austenitizing temperature in a range from 900 to 1200°C on prior austenite grain size was investigated to verify the precipitation analysis of MX-type particles. The results of the research are presented in Fig. 1. The size of prior austenite grains at an austenitizing temperature from 900 to 1000°C is very small and ranges from 11 to 18 μm for Ti-V steel and from 8 to 12 μm for Ti-Nb-V steel. The increase in the austenitizing temperature causes gradual growth of the austenite grains, which is more distinct for the Nb-free steel. The average diameter of the austenite grains after austenitizing of steels at a temperature of 1200°C increases to about 66 and 62 μm, for steel A and B, respectively. The observed grain sizes of austenite for the investigated steels are also similar to those reported for other Ti-V or Ti-Nb steels [15,16]. A change in the austenite grain size as a function of the austenitizing temperature is typical for microalloyed steels. The changes are parabolical for both steels, especially for the Ti-Nb steel (Fig. 1).

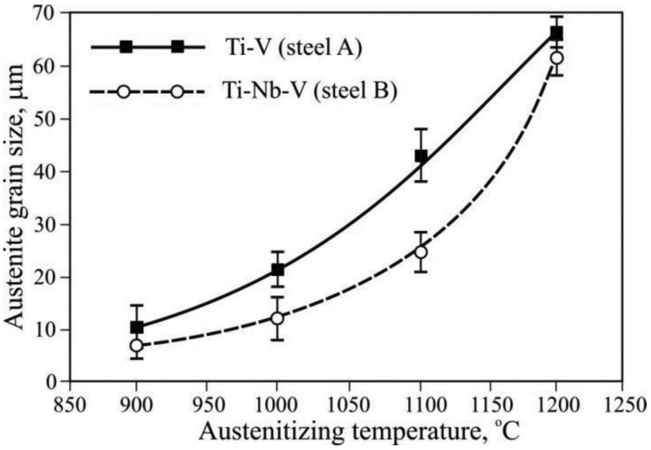

Fig. 1. Influence of the austenitizing temperature on the grain size of the austenite

3.2. Stress-strain curves and calculation of activation energy of deformation

The analysis of the process of hot plastic deformation of steels A and B that was conducted in a temperature range from 900 to 1100°C and at a strain rate of 1, 10 and 50 s^{-1} allowed to define the impact of compression parameters at a constant austenitizing temperature equal to 1150°C on the shape of the work-hardening curves, as determined in the function of flow stress σ – strain ε.

In the considered range of temperature and strain rate, compression curves of steel B are characterized by slightly higher values of maximum flow stress σ_m in comparison to steel A (Fig. 2). This is a result of the deformation of prior austenite grains of steel A and B, diversified with respect to the chemical composition and size after austenitizing at a temperature of 1150°C. For a strain rate of 1 s^{-1}, a decrease in the temperature of deformation from 1100 to 900°C leads to an increase in the maximum flow stress from 98 MPa to 186 MPa and their movement from ε_m =0.26 to ε_m =0.42 for steel A, and an increase of σ_m from 103 MPa to 192 MPa and their movement towards higher deformations – from ε_m =0.27

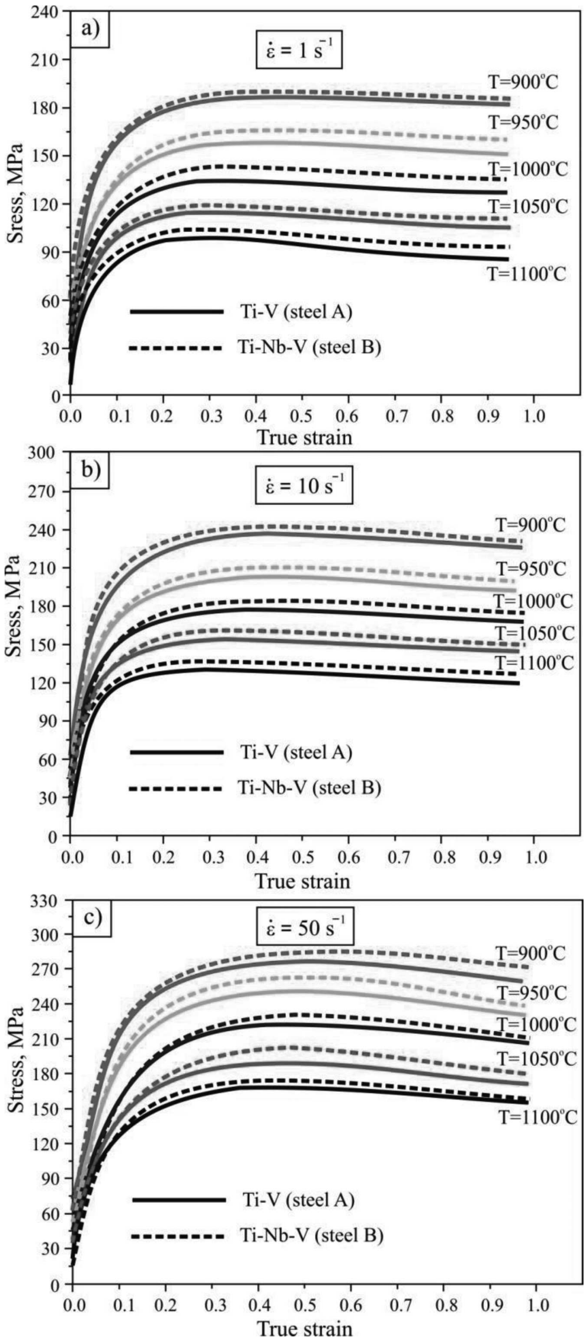

Fig. 2. Influence of the plastic deformation temperature and strain rate (a-c) on a shape of $\sigma-\varepsilon$ curves for investigated steels

to ε_m =0.43 for steel B. A decrease in the strain temperature in the same range during compression at a rate of 50 s^{-1} influences the increase of σ_m from 169 MPa to 285 MPa for steel A and from 173 MPa to 285 MPa for steel B. For the described parameters of compression, the ε_m strain increases from ε_m =0.37 to ε_m =0.57 for steel A and from ε_m =0.44 to ε_m =0.59 for steel B. During compression of steel A at a rate of 10 s^{-1} at a temperature of 900°C the value of σ_m is equal to 245 MPa and takes place at a strain equal to ε_m =0.46; for steel B the value of σ_m = 246 MPa at ε_m = 0.53. An increase in the strain rate up to a temperature of 1100°C at a strain rate of 10 s^{-1} causes a decrease of σ_m to a value of 124 MPa for steel A and to a value of 134 MPa for steel B. The values of ε_m corresponding to the maximum flow stress are equal to 0.30 and 0.38 – for steel A and B, respectively. An analysis of the form and shape of curves obtained in the compression test allows to state that in the studied range of hot plastic deformation parameters the decrease in strain hardening, both for steel A and B, is caused by the process of continuous dynamic recrystallization.

An analysis of the shape of curves obtained in the compression test allows to state that in the studied range of hot deformation parameters the decrease in strain hardening was caused by the process of continuous dynamic recrystallization. This is also confirmed by the results of evaluating the activation energy of the examined steel's plastic deformation process. The determined activation energy of the plastic deformation process of Ti-V steel is equal to Q = 382 kJ·mol^{-1}, while the activation energy of the plastic deformation process of Ti-Nb-V steel is equal to Q = 398 kJ·mol^{-1}, wherein the values of constants in (Eq. 2) for stress corresponding with ε_{max} deformations are equal: A = 3.43·10^{15}, α = 0.00628, n = 6.93 and A = 7.42·10^{15}, α = 0.00649, n = 7.17 – for Ti-V and Ti-Nb-V steel, respectively. The obtained values of activation energy are substantially higher than the activation energy of self-diffusion, i.e. when the processes which control the course of plastic deformation are dislocation climbing and form subgrains. This means that the process of plastic deformation of the studied steels is controlled by dynamic recrystallization.

3.3. Kinetics of recrystallization

The research conducted here on the recrystallization of Ti-V and Ti-Nb-V steel after two-stage hot compression allowed to determine the influence of the testing temperature on the kinetics of thermally activated processes. Discontinuous compression tests of the specimens at a given strain revealed, according to expectations, that there is a partial and even complete decay of strain hardening between the two stages of deformation depending on the strain temperature and the time of isothermal holding. The research results of strain hardening kinetics of the studied steels are presented in Table 3.

The impact of alloying elements dissolved in a solid solution and the presence of dispersive particles of MX-type interstitial phases on the rate of recovery and mobility of the recrystallization front significantly influence the kinetics of static recrystallization of the steels studied here. This interaction is illustrated as the strain hardening curves of steel A austenite containing 0.31% C, 1.45% Mn, 0.22% Mo and microadditions of Ti and V in an amount of 0.033% and 0.008%, respectively (Fig. 3). A comparison of the course of softening kinetics curves for steel A indicates that the decrease in the plastic deformation temperature and the decrease in the isothermal holding temperature from 1100 to 900°C causes considerable extension of the recovery time and a decrease in the austenite recrystallization rate.

This is a result of the decreasing value of the self- diffusion coefficient of Fe and the coefficients of volume diffusion of the alloying constituents along with a lowering of the temperature as well as the influence of the atoms and dispersive particles of the interstitial phases of microadditions introduced into the steel on the migration rate of the recrystallization fronts. For steel A, Mo and V and partially Ti can be found in austenite at a temperature of 1100°C in dissolved state.

TABLE 3

Results of the discontinuous compression of specimens

Steel designation	Temperature of plastic deformation, °C	$\dot{\varepsilon}$ s^{-1}	ε_1	Isothermal holding time, s	ε_2	σ_0 MPa	σ_1 MPa	σ_2 MPa	Softening fraction X
Ti-V (steel A)	900	10	0.2	2	0.2	143.22	224.02	212.20	0.15
			0.2	5	0.2	153.91	214.74	201.18	0.22
			0.2	10	0.2	154.71	215.06	195.30	0.32
			0.2	50	0.2	153.99	217.34	177.43	0.63
			0.2	100	0.2	157.40	211.46	166.60	0.83
	1000	10	0.2	2	0.2	109.51	165.62	152.52	0.23
			0.2	5	0.2	110.30	164.32	147.04	0.32
			0.2	10	0.2	110.01	165.55	137.35	0.50
			0.2	50	0.2	109.50	163.32	117.84	0.84
			0.2	100	0.2	112.11	166.23	114.99	0.95
	1100	10	0.2	2	0.2	87.64	122.20	112.54	0.28
			0.2	5	0.2	87.36	123.25	105.31	0.49
			0.2	10	0.2	87.24	123.74	98.65	0.68
			0.2	50	0.2	79.91	115.97	82.73	0.92
			0.2	100	0.2	79.29	119.32	79.29	1.00
Ti-Nb-V (steel B)	900	10	0.2	2	0.2	138.64	219.11	214.34	0.06
			0.2	5	0.2	139.32	220.46	210.19	0.12
			0.2	10	0.2	138.68	218.70	205.63	0.16
			0.2	50	0.2	141.49	220.51	187.80	0.41
			0.2	100	0.2	161.81	221.77	180.55	0.68
	1000	10	0.2	2	0.2	108.76	165.05	156.00	0.17
			0.2	5	0.2	108.00	167.01	150.15	0.28
			0.2	10	0.2	109.93	161.27	142.39	0.37
			0.2	50	0.2	108.40	161.76	123.51	0.72
			0.2	100	0.2	108.42	162.85	117.41	0.83
	1100	10	0.2	2	0.2	95.81	125.14	117.86	0.24
			0.2	5	0.2	95.86	123.79	112.55	0.40
			0.2	10	0.2	95.42	124.24	106.08	0.63
			0.2	50	0.2	95.69	123.77	100.49	0.83
			0.2	100	0.2	93.98	121.94	95.04	0.96

Interaction of the dissolved elements leads to an extension of the recovery time and a decrease in the recrystallization rate due to segregation of those atoms in the deformation field of dislocations and on the recrystallization fronts, thus causing a decrease in their mobility. The greatest influence on extending the times of recovery and recrystallization is exerted by the contribution of segregation of dissolved atoms and dispersive particles of the interstitial phases. As shown in Fig. 3, time $t_{0.5}$ that is necessary to form a 50% fraction of the recrystallized austenite at a temperature of 1100°C is equal to approximately 6 s and increases to about 30 s together with a decrease in the compression temperature to 900°C. The time of total recrystallization of austenite, t_R, which varies from 100 to about 600 s in the investigated temperature range, drags even more.

Strain hardening curves for steel B containing 0.28% C, 1.41% Mn, 0.22% Mo and microadditions of Nb, Ti and V in the amount of 0.027%, 0.028% and 0.019%, respectively, are presented in Fig. 3. Data shown in the figure indicate that inhibiting the interaction of the alloying elements introduced into the steel during the course of recovery and static recrystallization of austenite is particularly effectively noted after decreasing the temperature of plastic deformation and the temperature of isothermal holding to 900°C. At this temperature, Mo and the microaddition of vanadium are present in a solid solution in dissolved state. In turn, microadditions of Nb and Ti are completely bound into NbC, TiN and TiC at this temperature.

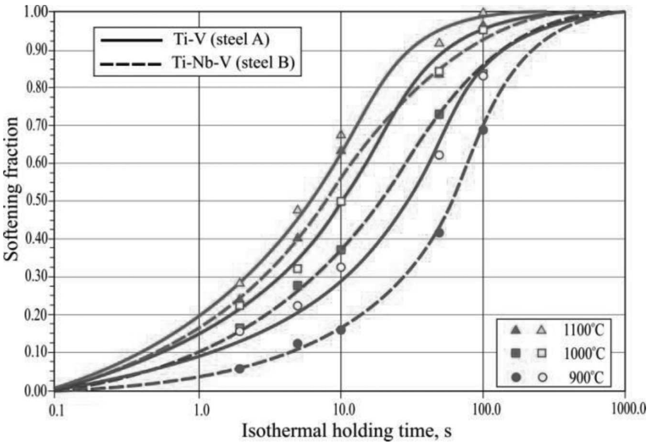

Fig. 3. Effect of the test temperature on the softening fraction of deformed austenite for steel A and steel B

The shape of the analyzed kinetic curves indicates the possibility of describing them in accordance with the model of recrystallization after hot deformation by taking into consideration the process of static recovery, metadynamic recrystallization and static recrystallization. The contribution of metadynamic recrystallization to the decrease in strain hardening after deformation of steel A, done at a temperature of 1100°C and at a rate of 1 s^{-1}, is possible because of the applied value of strain equal to ε =0.2, which is close to the ε_{cd} value that is required for initiation of dynamic recrystallization. The applied degree of deformation at lower strain temperature and at higher strain rates was lower than that required for initiation of the process of dynamic recrystallization. Nevertheless, after deformation of steel A at a temperature of 900°C and at a rate of 10 s^{-1}, the decrease in strain hardening is caused by the process of static recovery and static recrystallization, and at a temperature of 1000 and 1100°C it is caused by the process of static recovery, metadynamic recrystallization and static recrystallization. In steel B, on the other hand, deformed at a temperature of 900 and 1000°C, the decrease in strain hardening is a result of the course of static recovery and static recrystallization, and deformed at 1100°C it is controlled by the course of metadynamic and static recrystallization.

Furthermore, the discontinuous compression tests conducted on the investigated steels in the two-stage deformation by applying isothermal holding for a time period ranging from 2 to 100 s revealed that the kinetics curves of recrystallization of plastically deformed austenite of steel B are clearly displaced towards the right in relation to the kinetics curves of plastically deformed austenite of steel A. This should be explained by the interaction of the Nb microaddition, whose presence in steel B has a great influence on the decrease in the rate of thermally activated processes. The beginning of precipitation of the NbC carbides for equilibrium conditions occurs at a temperature of about 1137°C. This means that a certain portion of niobium is bound into the dispersive particles of the NbC carbides, formed on dislocations in plastically deformed austenite, already at a temperature of 1100°C. The fraction of NbC carbides in solid solution increases along with the temperature decrease, and niobium is bound into NbC at a temperature of 900°C.

3.4. Effect of isothermal holding time on the austenite grain size

Large austenite grains with an average diameter of 64 μm, after static recovery occurring in a time period equal to 2 s, were revealed in the microstructure of steel A, deformed in two stages at a temperature of 900°C. After isothermal holding at a temperature of 900°C for 5 s there is a decrease in the rate of the course of thermally activated processes, connected most likely to the formation of nuclei of static recrystallization. Isothermal holding of steel A specimens at such a temperature for 10 s leads to the initial stage of static recrystallization, whereas in these conditions the degree of softening is equal to X=0.32 (Table 3). An increase in the isothermal holding time up to 100 s (X=0.83) results in grain growth of austenite. Distinct refinement of the primary austenite grains in comparison to the initial state, probably caused by partial course of dynamic recrystallization during deformation and the course of metadynamic and static recrystallization after deformation, was observed after two-stage compression of steel A at a rate of 10 s^{-1}, and at a temperature of 1000 and 1100°C. Isothermal holding after deformation of steel A specimens at a temperature of 1000°C for 2 and 5 s leads to obtaining a 23% and 32% fraction of recrystallized austenite, respectively, and the size of the phase is 32 μm and 22 μm, respectively. Slowing down the process of decay of hardening is observed after isothermal holding at a temperature of 1000°C for 10 s, which is connected with the formation of nuclei of static recrystallization. Austenite grain size after isothermal holding of specimens for 10 s is equal to approximately 16 μm, and the recrystallized fraction is equal to 50%. The progress of the recrystallization process and growth of γ-phase grains is observed for the time of isothermal holding equal to 50 and 100 s. Isothermal holding of samples for 50 s leads to obtaining a 84% fraction of recrystallization and a mean diameter of austenite grains of approx. 35 μm. An increase in time up to 100 s results in obtaining a microstructure characterized by a 95% fraction of recrystallization and an average diameter of γ-phase grain size equal to 78 μm. A decrease in the hardening of steel A after double-hit compression at a temperature of 1100°C is determined by the course of dynamic recrystallization after isothermal holding of the samples at this temperature for 2 and 5 s and, successively, by the course of static recrystallization for the time of holding equal to 10, 50 and 100 s.

The size of the primary grains of austenite varies from about 27 μm for a holding time of 2 s to about 63 μm for a holding time equal to 100 s. As a result of two-stage compression of steel B at a temperature of 900°C, at a rate of 10 s^{-1}, diversified grain size of primary austenite, in a range from 25 μm to 70 μm, was obtained as a consequence of static recovery and static recrystallization. Two-stage deformation of steel B at a temperature of 1000°C with the use of isothermal holding for a time equal to 2 s to 100 s caused a decrease in strain hardening as a result of the course of static recovery (Fig. 4a) and static recrystallization (Figs. 4b-d). In the examined range of isothermal holding, the average diameter of the austenite grains varied from 14 μm to 60 μm, and the obtained decrease in hardening was equal to X=0.83. Isothermal holding of specimens at a temperature of 1100°C for 2 and 5 s after the first stage of deformation, equal to ε =0.2, caused a decrease in the hardening of steel as a result of the course of static recovery and static recrystallization, which determined that primary austenite grains with a mean diameter of 20 μm and 33 μm, respectively, were obtained. The recrystallized fraction of austenite for an isothermal holding time of 10, 50 and 100 s is equal to 63%, 83% and 96%, respectively.

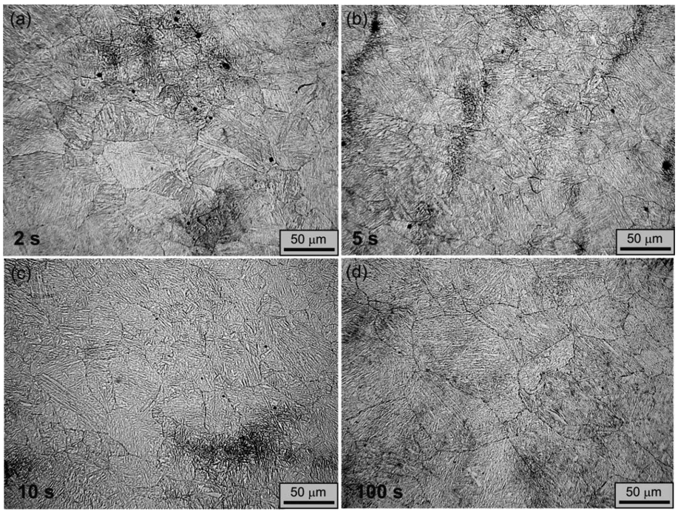

Fig. 4. Prior austenite grains of steel B revealed after two-stage compression test at the temperature of 1000°C at the rate of 10 s^{-1} applying breaks between deformation steps in a range from 2 to 100 s (a-d)

4. Conclusions

An investigation of the influence of austenitizing temperature on the grain size of prior austenite confirmed the correctness of the precipitation analysis of interstitial phases in the studied steels. The steels possess a fine-grained microstructure of austenite in the whole austenitizing temperature range that was investigated. Austenite grain size grows parabolically with the increasing temperature for both steels and is several times lower as compared to C-Mn steels without microadditions. Faster grain growth was also observed for the Ti-V steel.

Continuous compression tests of the investigated steels conducted at a strain rate of 1, 10 and 50 s^{-1} allowed to determine the influence of the plastic deformation temperature in a range from 1100 to 900°C on the shape of the $\sigma - \varepsilon$ curves and the ε_m strain corresponding to the maximum value of flow stress and, hence, allowed to estimate the strain that is necessary to initiate dynamic recrystallization of austenite. Analysis of the flow curves of the studied steels indicates that the increase in the strain rate from 1 to 50 s^{-1} causes an increase in the maximum value of flow stress by an average of 40 MPa regardless of the temperature applied. The values of activation energy obtained here indicate that the process of plastic deformation of the studied steels is controlled by dynamic recrystallization.

The two-stage compression tests conducted in this study revealed that, apart from the temperature, considerable influence on the kinetics of recovery and static recrystallization is exerted by the chemical and phase composition of steel after hot working has been concluded and that the t_R time of total recrystallization of steel containing microadditions of elements with high chemical affinity for carbon and nitrogen, bound into the dispersive particles of carbides or nitrides at the recrystallization temperature, is long. The determined time for total recrystallization of austenite, t_R, in a temperature range from 1100°C to 900°C changes from 100 to 600 s and from 300 to 800 s – for Ti-V steel and Ti-Nb-V steel, respectively. This means that the complete recrystallization of austenite requires long periods of time, which is unacceptable in the production process of forgings. Hence, forgings should be isothermally held at a temperature of forging finish prior to hardening for the time that is necessary to form about a 50% fraction of recrystallized austenite, which for steel A is equal to 30 s, and is 60 s for Nb-containing steel. The lower rate of static, thermally activated processes in steel B in relation to steel A is the result of the presence of the Nb microaddition which, formed on dislocations during plastic deformation in the form of dispersive NbC carbides, slows down the course of recovery and dynamic recrystallization and, after hot working is done, decreases the rate of recovery and static recrystallization and limits the grain growth of recrystallized austenite.

The research results obtained here allow to develop an industrial technology of forgings with high mechanical properties by using the method of thermomechanical treatment.

REFERENCES

[1] T. Gladman, The Physical Metallurgy of Microalloyed Steels, The Institute of Materials, University Press, Cambridge 1997.

[2] D.K. Matlock, G. Krauss, J.G. Speer, Microstructures and properties of direct-cooled microalloy forging steels, J. Mater. Process. Technol. **117**, 324 (2001).

[3] P. Skubisz, H. Adrian, J. Sińczak, Controlled cooling of drop forged microalloyed steel automotive crankshaft, Arch. Metall. Mater. **56**, 93 (2011).

[4] J. Zrnik, T. Kvackaj, A. Pongpaybul, P. Sricharoenchai, J. Vilk, V. Vrchovinsky, Effect of thermomechanical processing on the microstructure and mechanical properties of Nb-Ti microalloyed steel, Mater. Sci. Eng. A **319-321**, 321 (2001).

[5] M. Opiela, A. Grajcar, Elaboration of forging conditions on the basis of the precipitation analysis of MX-type phases in microalloyed steels, Arch. Civ. Mech. Eng. **4**, 427 (2012).

[6] J. Zhao, Z. Jiang, C.S. Lee, Effects of tungsten addition and heat treatment conditions on microstructure and mechanical properties of microalloyed forging steel, Mater. Sci. Eng. A **562**, 144 (2013).

[7] M.J. Balart, C.L. Davis, M. Strangwood, Fracture behavior in medium-carbon Ti-V-N and V-N microalloyed ferritic-pearlitic and bainitic forging steels with enhanced machinability, Mater. Sci. Eng. A **328**, 48 (2002).

[8] P.R. Spena, D. Firrao, Thermomechanical warm forging of Ti-V, Ti-Nb, and Ti-B microalloyed medium carbon steels, Mater. Sci. Eng. A **560**, 208 (2013).

[9] J. Zhao, Z. Jiang, C.S. Lee, Enhancing impact fracture toughness and tensile properties of a microalloyed cast steel by hot forging and post-forging heat treatment processes, Mater. Des. **47**, 227 (2013).

[10] M. Opiela, A. Grajcar, Hot deformation behavior and softening kinetics of Ti-V-B microalloyed steels, Arch. Civ. Mech. Eng. **3**, 327 (2012).

[11] M. Opiela, Thermomechanical treatment of Ti-Nb-V-B micro-alloyed steel forgings, Mater. Technol. **4**, 37 (2014).

[12] G. Thomas, Microalloyed Vanadium Steels, Proc. Symp., Kraków, Poland (1990).

[13] G. Krauss, S.K. Banerji (Eds.), Fundamentals of Microalloying Forging Steels, TMS, Warrendale, PA, USA (1987).

[14] H. Adrian, Thermodynamic model for precipitation of carbonitrides in high strength low alloy steels containing up to three microalloying elements with or without addition of aluminium, Mater. Sci. Technol. **8**, 406 (1992).

[15] A. Nowotnik, T. Siwecki, The effect of TMCP parameters on the microstructure and mechanical properties of Ti-Nb microalloyed steel, Journal of Microscopy **237**, 258 (2008).

[16] P.S. Bandyopadhyay, S.K. Ghosh, S. Kundu, S. Chatterjee, Evolution of microstructure and mechanical properties of thermomechanically processed ultrahigh-strength steel, Metall. Mater. Trans. A. **42**, 2742 (2011).

Received: 20 March 2014.