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SPECIAL SECTION

Effect of hot deformation and isothermal holding temperature on retained austenite characteristics in 3–5% Mn multiphase steels

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Abstract. The paper presents stress-strain characteristics recorded during the four-step compression of axisymmetric samples in the Gleeble thermomechanical simulator. The hot deformability of three steels with Mn concentrations of 3%, 4% and 5% was compared. The analysis of the influence of plastic deformation and Mn content on the microstructure of alloys, and in particular, on a fraction and morphological features of the retained austenite, was performed. The proportion of the retained austenite was determined by the X-ray diffraction method. It was found that the content of Mn in the range from 3% to 5% does not have a significant impact on high-temperature resistance of the steel during compression tests, but it has a significant influence on the microstructure of the steel and the fraction of retained austenite. The optimal conditions for maximizing the proportion of retained austenite were obtained at the temperature of 400°C, and it decreased with increasing Mn concentration in the steel. It has been shown that this is related to the redistribution of carbon from the remaining austenite fraction with an increase in manganese content. The mechanical properties were determined on the basis of hardness measurements.

Key words: hot deformation; bainitic transformation; retained austenite; multi-phase structure; Mn effect.

1. INTRODUCTION

Environmental degradation and the impending energy crisis determine the strong drive to develop technologies enabling the production of ultra-durable structural elements for the automotive industry, allowing to reduce the weight of the vehicle and reduce CO_2 emissions to the environment while ensuring passenger safety [1–3]. The answer to these requirements are advanced steels with a multi-phase microstructure, in which the key component is retained austenite (RA) in amounts from 10% to 30%, ensuring the required level of ductility [4–7]. The main elements that determine stability of the retained austenite are carbon and manganese [8–11]. Since the C content is usually limited due to welding reasons, Mn plays an important role in TRIP (transformation induced plasticity) steels containing from 10% to 15% of retained austenite [12–16], and in the formation of the austenitic phase in high-manganese steels showing the TRIP and / or TWIP (twinning induced plasticity) effect [17, 18]. In this case, stabilization of homogeneous austenite requires the addition of Mn in the range from 15% to 30% [19–21].

The determinant of contemporary achievements of physical metallurgy of iron alloys in the field of designing steel for car bodies, combining high strength, plasticity and the ability to absorb energy by properly designed zones of controlled crushing, are medium-manganese steels with a multi-phase microstructure with RA [22]. Medium manganese steels, containing between 3% and 12% Mn, offer the best combination of strength and ductility and are significantly cheaper compared to high-Mn austenitic steels [23].

Cold-rolled medium manganese steels are produced by intercritical annealing after cold rolling [24–26]. This allows the microstructure to achieve an equilibrium of ultra-fine-grained ferrite and austenite, which by C and Mn stabilization can be maintained up to room temperature. Lowering the martensitic transformation temperature is favored by the diffusion of carbon from the ferrite / bainitic ferrite to the γ phase, increased manganese concentration and fragmentation of the microstructure [27, 28]. Alternatively, medium manganese steels can be hot-rolled and directly cooled from finishing rolling temperature [29–31]. Such thermomechanical rolling simulations (important e.g. for higher thickness sheets used for underbody elements) are investigated distinctly less often.

It is necessary to apply controlled, multi-step cooling immediately after the completion of hot rolling. Its main stage is the isothermal bainitic transformation (in industrial conditions, e.g. coiling sheets into coils), during which the formation of bainitic ferrite is accompanied by an increase in the carbon content in the remaining fraction of the γ phase [32, 33]. The physical phenomena taking place in such medium-Mn steels are the same as in conventional thermomechanically processed TRIP-aided steels, although the bainitic transformation kinetics is different (strongly delayed) due to increased Mn concentrations. Plastic deformation of austenite accelerates these diffusion processes, and the presence of silicon and/or aluminum in steel limits/prevents the precipitation of carbides in

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bainite [34]. High-temperature deformation (strain, strain rate, break times between deformations, temperature of the final deformation) and cooling conditions in the range of austenite to isothermal bainite transformation have a significant impact on the kinetics of this transformation and on the final microstructure and mechanical properties of multiphase steels [35]. The temperature and time of isothermal holding in the area of bainite transformation have a significant influence on the obtained microstructure and the stability of the retained austenite. If the isothermal holding time is too short, the austenite will have poor stability due to the low carbon content. Carbides will precipitate if the isothermal holding time is too long. In both cases, this leads to an unfavorable increase in the M_s temperature and the formation of the so-called fresh martensite with the final cooling of the steel to room temperature [33–35]. In [36], it was shown that the isothermal temperature during the bainite transformation of 0.2C-1.5Mn-1.5Si-0.04Al steel significantly influences the obtained amount of retained austenite and the concentration of carbon in this phase. It was found that the highest proportion of RA was obtained during isothermal holding at a temperature of about 430°C.

There is a limited amount of knowledge on the synergistic effect of hot deformation and the effect of isothermal temperature and time on the microstructure of medium manganese steels. The publications available on this subject concern steels with Mn concentrations below 2% [37–40]. Therefore, the aim of this study is to investigate the effect of high-temperature plastic deformation and isothermal holding parameters on the microstructure and microstructural characteristics of three newly developed multiphase steels containing 3%, 4% and 5% Mn.

2. METHODOLOGY

The tests were performed on three newly developed steels with the chemical composition given in Table 1. Manganese was used to stabilize retained austenite, while the combination of low Si and high Al contents was designed to ensure the formation of carbide-free bainite [8, 12]. Mo and Nb were added into the steel to increase the strength by grain size refinement of the structure and precipitation hardening.

Table 1
Chemical composition of investigated steels, in wt.%

Steel grade	C	Mn	Al	Si	Mo	Nb	S	P
3MnNb	0.17	3.1	1.6	0.22	0.22	0.04	0.005	0.008
4MnNb	0.18	3.6	1.7	0.20	0.20	0.04	0.004	0.008
5MnNb	0.17	5.0	1.5	0.21	0.20	0.03	0.005	0.008

Steel melts weighing 50 kg were produced in a laboratory vacuum induction furnace, type VSG-100S. The forging of ingots into flat bars with a cross-section of 22x170 mm was carried out using a hydraulic press. After austenitization for 2 hours at 1200°C, the ingots were forged in the temperature range from 1150°C to 900°C. The forged samples were air cooled to room temperature (RT) achieving coarse-grained bainitic-martensitic-austenitic microstructures [5]. This initial material formed the basis for further sample machining and performing thermomechanical tests.

In order to determine the influence of plastic deformation conditions on changes in flow stress and isothermal temperature on the microstructure of steel with different Mn contents, plastometric tests were carried out using the Gleeble 3800 simulator. Axisymmetric samples with a diameter of 10 mm and a length of 12 mm were used. A detailed program of thermomechanical treatment, i.e. heating, deformation and cooling conditions, are summarized in Table 2. The deformation schedule was designed based on the previous results of dilatometric and hot compression tests [41, 42]. The samples were heated in vacuum at the rate of 3°C/s to the temperature of 1200°C, in which they were annealed for 30 s. After austenitizing, the samples were cooled at the rate of 5°C/s to the temperature of 1150°C, which is the temperature of the beginning of plastic deformation. The samples were compressed in four stages, at 1150°C, 1050°C, 950°C and 850°C at a rate of 10 s^{-1}, respectively. In one case, the final deformation temperature of 750°C was also applied to 4MnNb. Then, the samples were cooled according to the cooling profiles listed in Table 2 to the isothermal holding temperature of 450°C, 400°C and 350°C. The isothermal holding of the samples was used to enrich the austenite with carbon. After isothermal holding at the mentioned temperatures for 300 s, the samples were cooled to room temperature at a rate of 0.5°C/s. The isothermal holding conditions were selected basing on B_s and M_s temperatures. In all the cases the holding temperature is lower than the calculated B_s temperature ($B_s = 543$, 526 and 477°C, respectively for 3MnNb, 4MnNb and 5MnNb steels). The 450–350°C range covers a temperature window in which the treatment is done below or above the dilatometrically determined M_s temperatures ($M_s = 389$, 349 and 314°C, respectively for 3MnNb, 4MnNb and 5MnNb steels) [8, 41, 42].

The aim of the metallographic tests was to assess the effect of high-temperature treatment conditions and isothermal holding parameters on the microstructure features of steels containing various Mn contents. Scanning electron microscopy and X-ray diffraction were used to analyze all constituents of the microstructure, especially the retained austenite. After 2% nital etching, metallographic observations were made with a SUPRA 25 scanning electron microscope using the backscattered electron technique at an accelerating voltage of 20 kV. X-ray examinations were carried out using an X'PERT PRO diffractometer, using a lamp with a cobalt anode, operating at a voltage of 40 kV and a current intensity of 30 mA. The test was made with the stepwise method, recording every 0.05°, as a function of the angle 2θ in the range from 40° to 115°. The proportion of retained austenite was determined by the Rietveld method. The angle positions of austenite peaks were used to determine a lattice constant and in this way the corresponding carbon content in the retained austenite. The following equation was applied [23]:

$$a\gamma = 3.578 + 0.033C\gamma, \tag{1}$$

where: $a\gamma$ – lattice parameter of the austenite (A), $C\gamma$ – carbon concentration in the austenite (wt. %).

Table 2
Detailed heating, deformation and cooling conditions for analyzed samples

Heating					
Soaking temperature, °C		Heating rate, °C/s	Soaking time, s	Cooling rate to deformation temperature, °C/s	
1200		3	30	5 (10 s)	
Deformation conditions					
No.	Deformation temperature °C	ε	$\dot{\varepsilon}$, s^{-1}	Cooling rate to a successive deformation step, °C/s	Time between successive deformation steps, s
1	1150	0.25	10	8	12.5
2	1050	0.25	10	10	10
3	950	0.25	10	20	5
4	850 (750)	0.25	10	below	–
Cooling conditions after deformation					
No.	Temperature range, °C		Cooling rate, °C/s		Isothermal holding time, s
1	850 → 700		40		–
2	700 → 650		5		–
3.1	650 → 450		40		
4.1	**450**		–		**300**
5.1	450 → RT		0.5		–
3.2	650 → 400		40		
4.2	**400**		–		**300**
5.2	400 → RT		0.5		–
3.3	650 → 350		40		
4.3	**350**		–		**300**
5.3	350 → RT		0.5		–

3. RESULTS AND DISCUSSION

3.1. Hot deformation behavior

The values of flow stresses registered in the four-step compression tests increase significantly with the reduction of the deformation temperature (Fig. 1). They are similar to the stresses obtained in the continuous and two-step compression tests [42, 43]. Dynamic recovery is the process controlling the strain hardening in the entire range of deformation temperatures. The influence of Mn in the range from 3% to 5% on the values of flow stress is negligible. The greatest differences in the value of flow stresses occur at the lowest temperature of plastic deformation, i.e. 850°C.

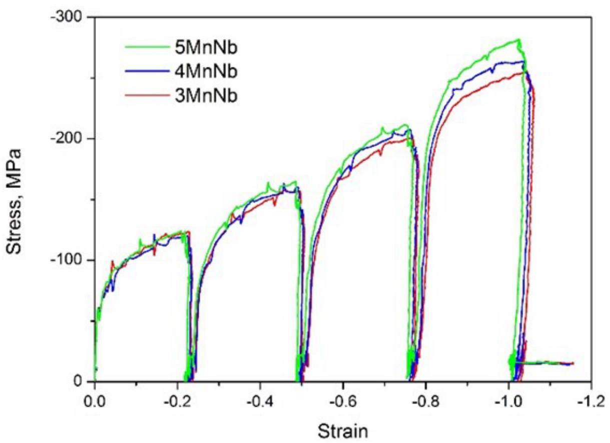

Fig. 1. Effect of Mn addition on the σ-ε curves in the four-step compression test of the tested steels

At this temperature, the stress values increase slightly with the increase in the Mn content in the steel. The steels contain increased Al addition. Its effect on flow stresses is relatively low (and the same, as the steels contain a similar Al content). Al forms AlN preferentially in the steels. It means that the driving force for NbN or Nb(C,N) formation decreases significantly. Therefore, a presence of NbC can be expected or more presumably Nb should dominate in the solid solution, which was revealed in our earlier work [42].

The influence of the final deformation temperature on the σ-ε curves is shown in Fig. 2, at the example of 4MnNb steel. As can be seen from the data presented in this figure, the temperature of the end of plastic deformation significantly influences the obtained values of flow stresses. As expected, a lower plastic deformation temperature generates a much higher level of stress values over the entire deformation temperature range. For the last deformation, the stress increases from 250 to 350 MPa along with the reduction of the compression temperature from 850 to 750°C.

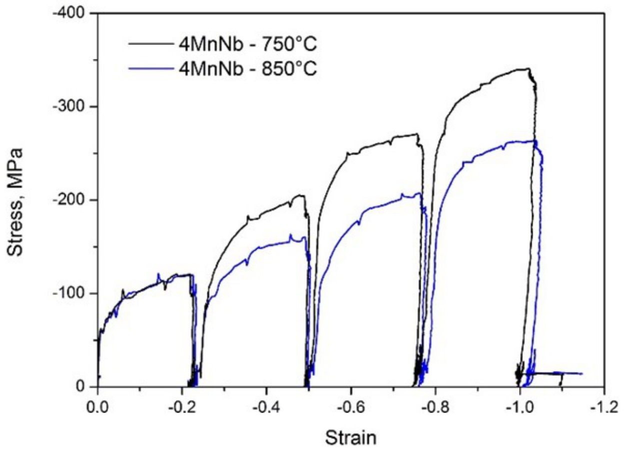

Fig. 2. Effect of final deformation temperature on $\sigma - \varepsilon$ curves of 4MnNb steel

3.2. Retained austenite fraction

The X-ray diffraction results confirm the presence of retained austenite, which is evidenced by the reflections from this phase on the diffraction patterns. It can be observed that the intensity of the reflections from the γ phase increases with the increase of isothermal temperature (Fig. 3a) and the decrease of Mn content in the steel (Fig. 3b).

The effect of the Mn content and isothermal temperature on the amount of RA in the tested steels is shown in Fig. 4. The error in measuring the RA amount by X-ray is ca. 2%, which can be seen when analyzing error bars in Fig. 4. After the isothermal holding of the samples at 350°C for 300 s, the fraction of retained austenite is similar and it is 7.1%, 6.3% and 5.9% for steels 3MnNb, 4MnNb and 5MnNb, respectively. Increasing the isothermal temperature in the case of 3MnNb steel to 400°C and 450°C results in an almost twofold increase in the proportion of retained austenite. At the temperatures mentioned, it is similar and amounts to approx. 14%. In the case of 4MnNb and 5MnNb steels, the increase in the isothermal holding temperature also resulted in an increase in the γ phase amount, but this was not as pronounced as in the case of 3MnNb steel. In 4MnNb steel, the fraction of this phase increased to approx. 10%, and in 5MnNb steel to approx. 9%. The increase in the amount of γ phase is the result of an increase in the rate of carbon diffusion along with an increase in isothermal temperature [23]. It is interesting that the amount of retained austenite at particular temperatures is the highest for steel with the lowest concentration of Mn. This is closely related to the changes in C concentration in this phase, as shown in Fig. 5. As shown in this figure, the highest values of C concentration – regardless of the Mn con-

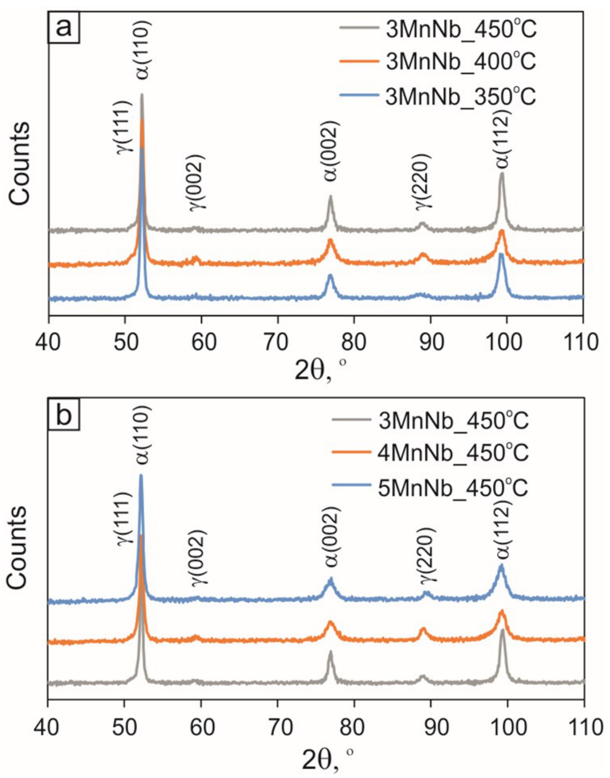

Fig. 3. Diffraction patterns of 3MnNb steel registered at different isothermal holding temperatures (a) and 3MnNb, 4MnNb, 5MnNb steels registered at 450°C (b)

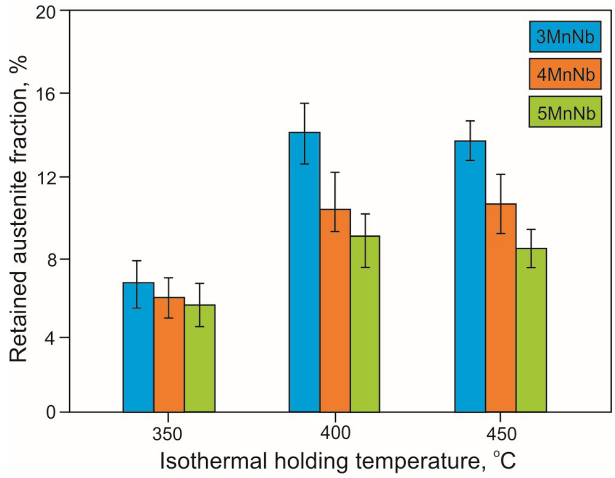

Fig. 4. Effect of isothermal holding temperature on the fraction of retained austenite in the tested steels

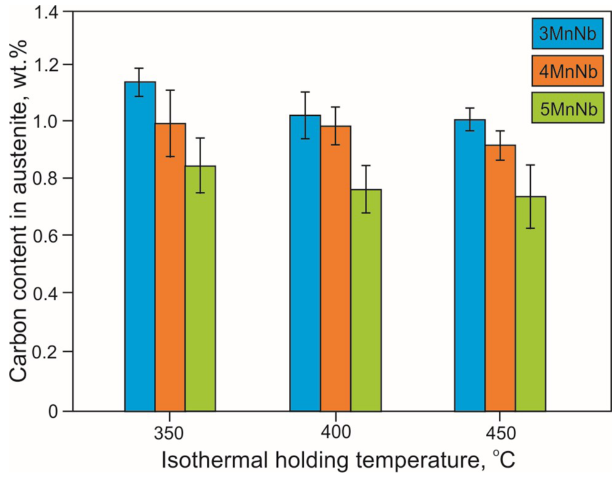

Fig. 5. Effect of isothermal holding temperature on carbon concentration in austenite

tent in steel – were recorded at 350°C. The carbon content of austenite after isothermal holding at this temperature is 1.17%, 0.96% and 0.82% for steels containing 3% Mn, 4% Mn and 5% Mn, respectively. It is related to the decreasing solubility of C in austenite with increasing Mn concentration, which results from thermodynamic calculations [25] and experimental data [41]. On the one hand, with increasing Mn content austenite stability should be increased. On the other hand, with increasing Mn content, the C content in this phase decreases. Therefore, it is a complex interaction of these two elements. Since the C effect is stronger, it appears that austenite stability is deteriorated in the steels containing 4 and 5% Mn. It should be noted that this is not a linear tendency because the austenite is stable with a further Mn content increase from ca. 6%. The C content still decreases in the austenite; however, such Mn content decreases the M_s temperature significantly enough, below room temperature [10].

As the temperature of isothermal holding increased for the same steel grade, the carbon content in the austenite of the tested steels decreased. This is due to the need to redistribute C to a larger volume of the γ phase [44–46]. Moreover, at the highest temperature of isothermal holding, the precipitation process of carbides may already begin, despite the increased concentration of Al in the tested steels [47, 48].

3.3. Microstructure evolution

The initial hot forged samples were subjected to the essential thermomechanical/multi-step cooling tests with applying deformation or without it (for comparison purposes). The comparison of the microstructure of the undeformed sample and the one subjected to the four-step compression, and isothermal holding at the temperature of 400°C, are all shown in Fig. 6. A reduction in the grain size of the prior austenite and the fragmentation of the microstructure of the steel subjected to plastic deformation are visible. The retained austenite in 3MnNb steel mainly takes the form of layers of different thickness arranged along the boundaries of the prior austenite and along individual packages of bainite laths. A comparison of the morphological features of the microstructure for 3MnNb and 5MnNb steels is shown in Fig. 7. Realization of phase transformations of supercooled austenite finally deformed at the temperature of 850°C results in a significant fragmentation of all structural constituents. This is true for both the bainite and retained austenite regions. The lath character of the structures increases with the increase in the concentration of Mn in the steel and with the decrease of isothermal temperature from 450 to 350°C. In general, steels are dominated by two-phase bainite-austenitic microstructures, but with some deviations. The RA can be identified because it a has a smooth topography and brighter contrast in SEM images [15, 23]. Moreover, it is usually located as layers between bainitic ferrite. In the case of 5MnNb steel this phase also forms MA constituents. Its presence was confirmed by X-ray techniques and EBSD [5].

Optimal microstructures in terms of RA stabilization can be observed at 400°C. For example, 3MnNb steel has granular bainite microstructure [37–40] with dominant RA layers (Fig. 7b). After increasing the temperature to 450°C, RA blocky grains dominate (Fig. 7c), which exhibit lower thermal and mechanical stability. The result is that in the largest austenite blocky grains (with a size exceeding approx. 2 μm) fresh martensite is formed. After the holding temperature has been reduced to 350°C, the interlath RA dominates, arranged in the direction of growth of the bainite laths (Fig. 7a). However, at this temperature, individual bainite laths merge locally, and the initial RA films are degraded into small blocks, which then, as a result of loss of thermal stability, initiate the release of cementite [12, 26, 49]. Al should prevent carbide precipitation during isothermal holding. However, its effectiveness is smaller compared to Si [4]. That is why fraction of carbides is partially precipitated, which is observed in Fig. 7 and 8. The precipitation process in 3MnNb steel occurs only at a temperature of 350°C, while in 5MnNb steel it can take place in the entire range of the holding temperature, and it is most intensive at 350°C. The tendency of carbide precipitation and the smallest enrichment of austenite with carbon determine that in the steel with the highest concentration of Mn, a large number of MA (martensite-austenite) islands are formed from blocky γ phase grains (Fig. 7d,e,f). The effect of the end of plastic deformation temperature, i.e. 750°C and 850°C, on the microstructure of 4MnNb steel is shown in Fig. 8.

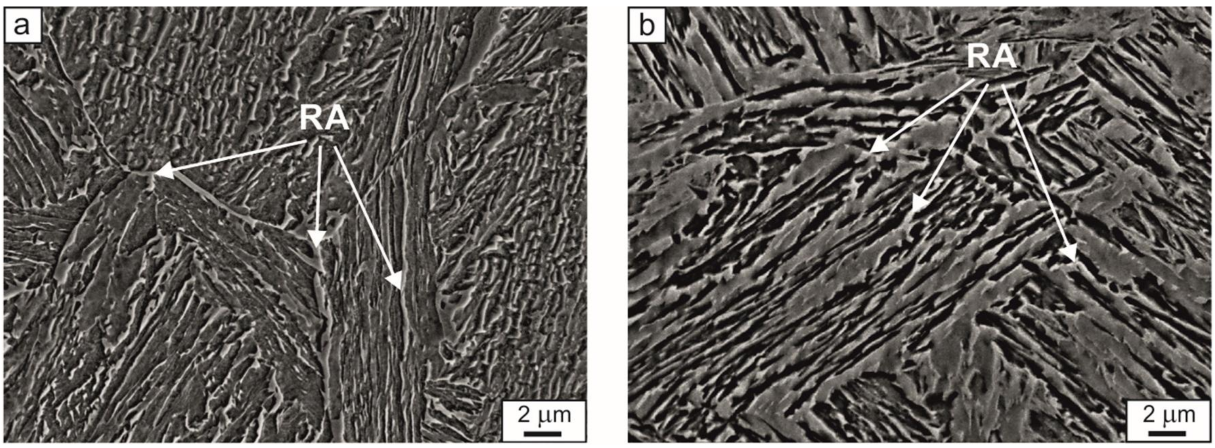

Fig. 6. Structure of 3MnNb steel undeformed (a) and subjected to four-step compression (b) after isothermal holding at 400°C

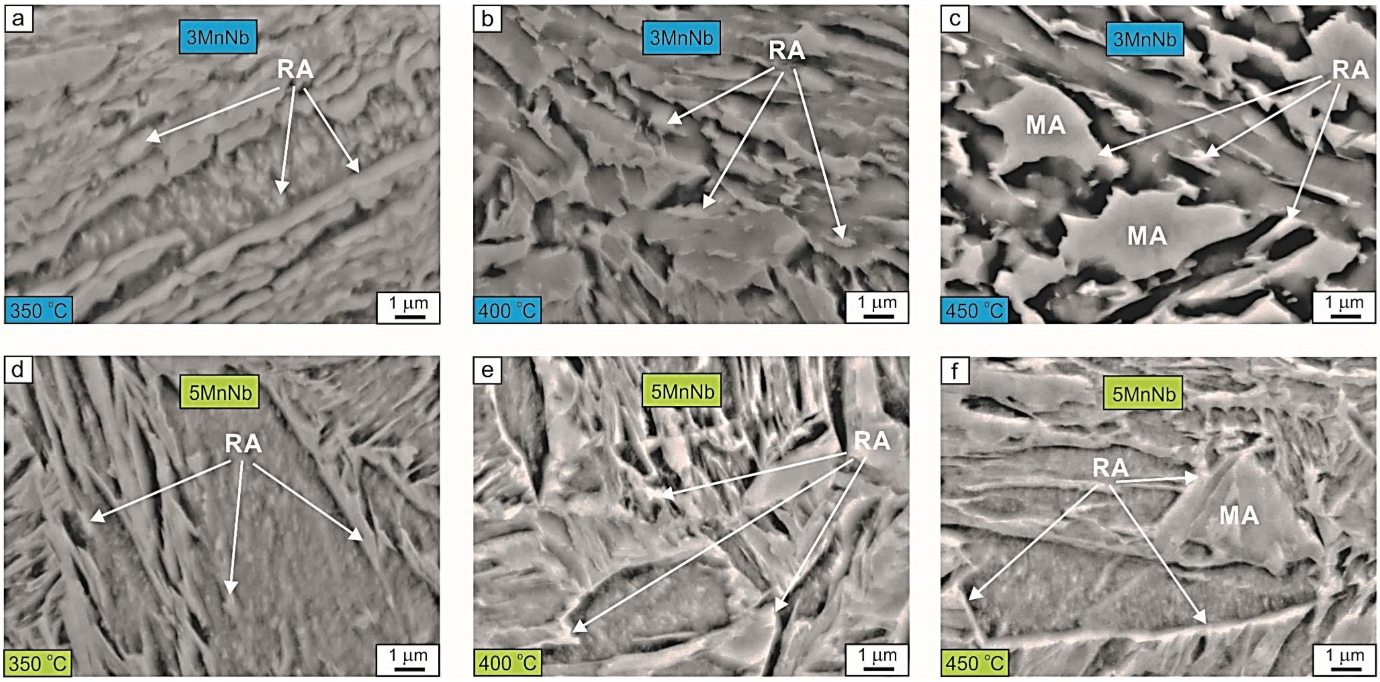

Fig. 7. SEM structures of 3MnNb (a, b, c) and 5MnNb (d, e, f) steels after isothermal holding at 350°C, 400°C and 450°C

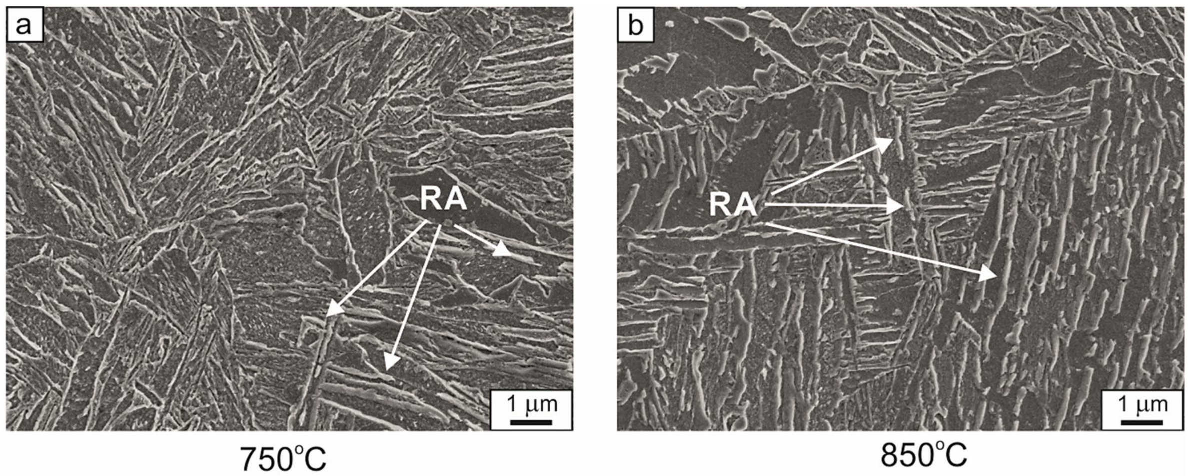

Fig. 8. Effect of final deformation temperature on the microstructure of 4MnNb steel held at 450°C

The microstructure shown in this figure fully corresponds to the σ-ε curves for this steel presented in Fig. 2. As expected, slightly greater fragmentation of the structure occurs in the case of a sample deformed at a lower temperature (Fig. 8a). The amount of austenite is comparable (approx. 11%), with the dominant fraction of layered retained austenite.

3.4. Hardness

The performed hardness measurements of the tested steels indicate that the Mn content and the microstructure have a significant impact on its value. It has been shown that the hardness of the samples decreases with the increase of isothermal temperature, and this applies to all the tested steels (Fig. 9). For example, the hardness of 3MnNb steel after isothermal holding at 350°C is approx. 469 HV10, at 400°C it is approx. 435 HV10, and it decreases to approx. 376 HV10 after isothermal holding at 450°C.

In the above-mentioned isothermal temperature range, the hardness of 4MnNb steel samples decreases from approx. 521 HV10 to approx. 472 HV10, and of 5MnNb steel from approx. 560 HV10 to approx. 463 HV10. It has also been shown that for a given isothermal temperature, the hardness increases with the increase in the Mn content in the steel. The changes in hardness correspond well with microstructural evolution, i.e. the hardness of the steel increases with the decreasing amount of retained austenite, which is a synergistic effect of lowering isothermal temperature and increasing the Mn content in the steel.

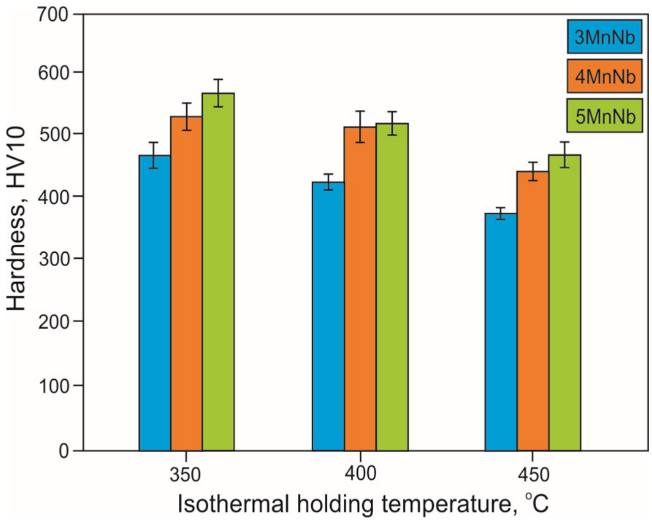

Fig. 9. Effect of isothermal holding temperature on hardness of the tested steels

The basic factor responsible for the increase in hardness along with a decrease in isothermal temperature is the increase in the density of structural defects in structural constituents combined with a decrease in bainite transformation temperature [39].

4. CONCLUSIONS

The content of Mn in the range of 3–5% does not significantly affect the value of flow stress, which only slightly increases during the last deformation at 850°C with the increase in the Mn content. Plastic deformation causes the phase transformations to be realized under conditions of strong austenite deformation. Isothermal holding conditions in the range from 350°C to 450°C favor the production of bainitic-austenitic structures for 3MnNb steels and bainitic-martensitic structures with retained austenite for 4MnNb and 5MnNb steels. The optimal amount of retained austenite for all steels was obtained after the samples holding at the temperature of 400°C. The highest fraction of RA (approx. 14%) with layered morphology was obtained for the steel with the lowest concentration of Mn, which is related to the highest carbon enrichment of this phase. The amount of this phase drops to approx. 9% for 5MnNb steel. At the holding temperature of 450°C, blocky austenite dominates, which favors partial formation of MA constituents by the formation of fresh martensite. At the temperature of 350°C, austenite laths partially decompose, which is particularly favored along with an increase in the concentration of Mn in the steel.

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