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INFLUENCE OF ISOTHERMAL HEATING TIME ON THE DISAPPEARANCE OF STRAIN HARDENING IN HIGH-MANGANESE TRIPLEX TYPE STEELS

The paper presents the results of the effect of isothermal heating time on the disappearance of strain hardening (the softening degree) of the studied high-manganese TRIPLEX type steels at a temperature of 900 and 1000°C. In order to determine the kinetics of recrystallization of austenite plastically deformed for selected steels, hot compression tests with draft $\varepsilon = 0.2$ were made. The presented results reveal that the complete recrystallization of austenite needs long isothermal heating times. In industrial conditions, such long times are not used, therefore in the initial rolling passages, the time required for half recrystallization of austenite $t_{0.5}$ is often used. The total disappearance of the strain hardening, completion of the recrystallization of austenite tested high-manganese X98 and X105 TRIPLEX type steels isothermal heating time requires far more than 200 s. The increase of the deformation temperature is a factor influencing the acceleration of the disappearance of strain hardening.

Keywords: Fe-Mn-Al-C steels, TRIPLEX steel, thermo-mechanical processing, softening degree

1. Introduction

Promising materials for applications in various types of construction elements are Fe-Mn-Al-C steels, characterized by low density and very good strength properties. The mechanical properties and structure of these steels determine such alloy components as carbon, aluminium and manganese. On the basis of the share of elements stabilizing austenite such as manganese and carbon and forming ferrite – aluminium and silicon steels Fe-Mn-Al-C can be divided into four groups: ferritic steels, ferrite based duplex steels, austenite based duplex steels and austenitic steels [1-7,12,16-19].

An important aspect when designing thermo-mechanical treatment is to determine the kinetics of austenite recrystallization, which can be determined on the basis of changes in flow stress as a function of isothermal heating time between successive stages of plastic deformation. As an indicator of the share of static austenite recrystallization, the degree of disappearance of strain hardening is assumed, otherwise known as softening degree. condition determining strain hardening [1,9,10,13,20-29].

Hamada et al. used a two-stage deformation method to determine the degree of softening. In their research, they compared high-manganese steels (25Mn and 25Mn3Al) with low-carbon steel. They noticed that the degree of disappearance of strain hardening in high manganese steels occurs much later than in the case of low carbon steel. They found that high manganese content affects the delay of the static recrystallization, and the impact of aluminium is minimal. In connection with the above, it was found that the activation energy of static recrystallization in high manganese steels is about 330kJ/mol [11].

Opiela and Ozgowicz presented research on the disappearance of strain hardening of austenitic steels with micro-additives Nb, Ti and V. Just like Hamada et al. they used the method of two-stage hot compression, and also by the analytical method, calculated the kinetics of recrystallization of plastically deformed austenite using the Johnson-Mehl-Avrami equation. On the basis of the obtained results, it was found that lowering the temperature of plastic deformation from 1100 to 900°C causes a significant increase in the recovery time and a decrease in the recrystallization rate of austenite. The alloying elements dissolved in a solid solution and the presence of dispersion precipitates in the MX type phases significantly affect of static recrystallization. Dispersed precipitates have a significant impact on the prolongation of recovery and recrystallization. The disappearance of strain hardening at 900°C and the deformation rate of $10\ s^{-1}$ is caused by static recovery and recrystallization, while at 1000 and 1100°C – by static recovery, metadynamic and static recrystallization [8,10].

Dynamic recovery and dynamic recrystallization processes that are controlling the plastic deformation does not guarantee receipt of the equilibrium structure of the deformed material. During slow cooling or isothermal heating between the plastic deformation stages, static processes occur that affect the disappearance of strain hardening, and are conditioned by the speed and size of the deformation. These processes include: static recovery, metadynamic recovery, static recrystallization and metadynamic

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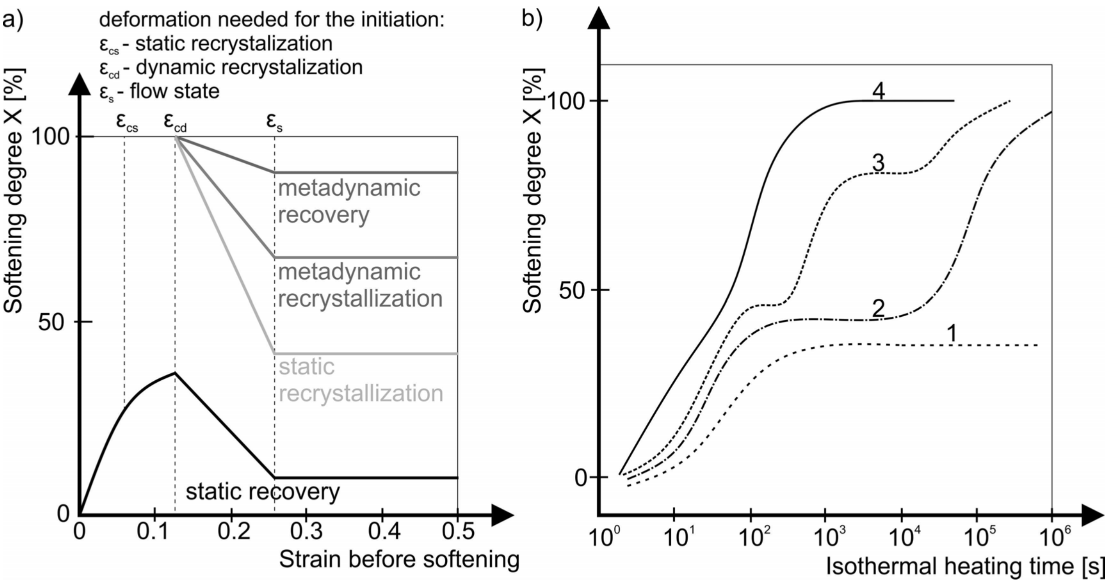

Fig. 1. a) Scheme effects of static processes thermally activated on the disappearance of strain hardening after hot deformation, b) a diagram of the mechanisms of removing the effects of strain hardening during isothermal heating after hot deformation [9,30]

recrystallization. The main mechanisms removing the effects of strain hardening during isothermal heating are static recovery, metadynamic and static recrystallization. Figure 1 shows a diagram of the impact of the above mentioned mechanisms [9,22,26-27,30]. While the deformation is smaller than the critical value needed to initiate a static recrystallization ($\varepsilon < \varepsilon_{cs}$), the disappearance of strain hardening occurs through static recovery (Fig. 1b – curve 1). Curve 2 corresponds to the course of static recovery on the horizontal section and static recrystallization in its further part ($\varepsilon > \varepsilon_{cs}$). In the case of curve 3, when $\varepsilon_{cd} > \varepsilon < \varepsilon_s$, the disappearance of strain hardening initially occurs through metatynamic recovery and recrystallization, followed by static recovery and static recrystallization. Curve 4 corresponds to the course of metadynamic recovery ($\varepsilon > \varepsilon_s$) [8,9,25-30].

2. Research material and methodology

The subject of the research were two high-strength structural, high manganese Fe-Mn-Al-C TRIPLEX type steels with the chemical composition shown in Table 1. These steels are characterized by austenitic-ferritic structure with carbide precipitates of different composition and morphology, as described in other publications [14,15]. The casts were modified with rare earth elements: cerium, lanthanum and neodymium. One of the casts of the tested steels was also introduced micro-additives of niobium and titanium with a strong chemical affinity for nitrogen and carbon.

The casts of the tested steels with a mass of 25 kg were made in a VSG-50 laboratory vacuum induction furnace from Balzers. The casting of the tested steels was carried out under argon to a round hot-topped mould, converging downwardly with internal dimensions: bottom – Φ122 mm, top – Φ145 mm, *h* = 200 mm – without a hot top (with a hot top – 300 mm) (Fig. 2). After casting, about 60 minutes were needed to solidify the head of the ingot, and then the furnace chamber was opened and the ingot in the ingot mold cooled down in the air. Samples for testing were prepared from a pre-forging ingot.

TABLE 1

The chemical composition of the examined steels

C	Mn	Al	Si	Nb	Ti	Ce	La	Nd	P_{max}	S_{max}
[wt. %]										
Steel X98MnAlNbTi24-11 (steel X98)										
0.98	23.83	10.76	0.20	0.048	0.019	0.029	0.006	0.018	0.002	0.002
Steel X105MnAlSi24-11 (steel X105)										
1.05	23.83	10.76	0.10	—	—	0.037	0.011	0.015	0.005	0.005

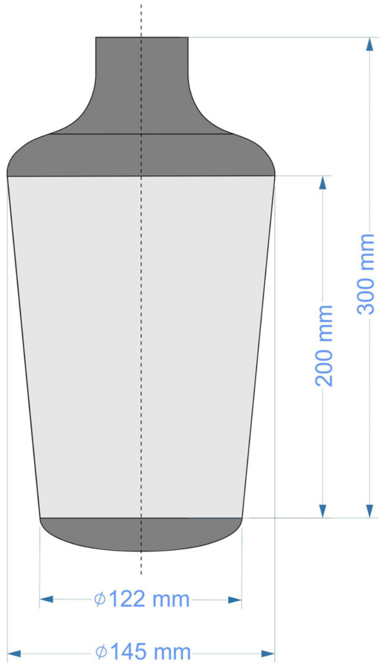

Fig. 2. Scheme of the cast ingot with dimensions

The initial hot plastic processing of ingots, for flat bars with a cross-section of 20×210 mm, was made using the forging method on Kawazoe's high-speed hydraulic press with a pressure of 300 tons. The ingots were heated in a gas forging furnace. The forging temperature ranged from 1200 to 900°C with in-process heat so that the material did not cool down below 900°C. Bodies of ingots without a head of an ingot – cut off at a height to which the shrinkage cavity and an ingot foot reached – cut at a height of about 3 cm were subjected to forging.

Cylindrical samples with dimensions ∅10×12 mm were made for plastometric tests. The tests were carried out using the Gleeble 3800 thermo-mechanical treatment simulator on axiomymetric samples. The aim of this study was to analyse the disappearance of strain hardening, i.e. the softening degree of the tested steels at 900 and 1000°C. In order to determine the kinetics of recrystallization of austenite plastically deformed for the investigated steels, hot compression tests were carried out in two stages with a draft $\varepsilon = 0.2$ assuming that $\varepsilon < \varepsilon_m$ (ε_m – maximum strain), with isothermal heating between the first and second stage of deformation by time from 1 to 200 s (Fig. 3). The deformation temperature (T_o) was 900°C and 1000°C. The softening degree was determined from the following relationship (Fig. 4):

$$X = \frac{\sigma_1 - \sigma_2}{\sigma_1 - \sigma_0} \qquad (1)$$

where, respectively:

σ_0 – stress of the beginning of plastic deformation,
σ_1 – stress at the moment of its interruption in the first stage of deformation,
σ_2 – stress initiating plastic deformation in the second stage of deformation after the time Δt between these stages.

The above mentioned test stand used for plastometric tests is equipped with a direct resistance heating system, maintaining the set temperature with an accuracy of ±1°C. Temperature control of samples takes place via a signal transmitted by means of thermocouples. The samples are deformed using tungsten carbide anvils. The Gleeble 3800 system, equipped with a hydraulic mechanical system, allows the pressure to be applied in the order of 200 kN during compression and allows testing at a strain rate in the range of 0.0001 to 200 s^{-1}. Linear variable differential transformer and strain gauges for measuring the pressure allowed obtaining to obtain feedback, which allows for accurate execution and repeatability of the specified mechanical quantities of the assumed technological process. In order to reduce the friction introduced very thin graphite and tantalum foils, between the surface of the sample and the surfaces of the anvils. Both surfaces were covered with a nickel-based lubricant (Fig. 5).

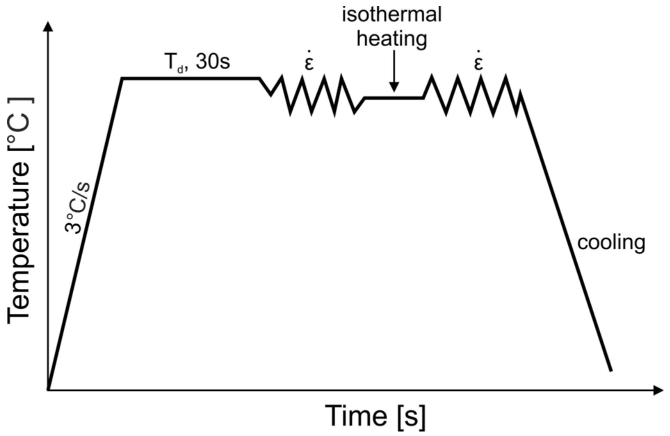

Fig. 3. Diagram of a two-stage compression process with isothermal heating

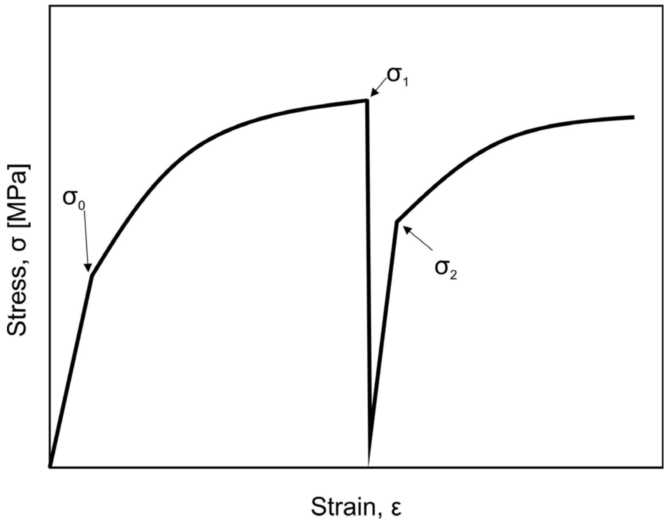

Fig. 4. Scheme for determining the progress of austenite recrystallization (softening degree)

Samples for structural tests were ground and mechanically polished on abrasive papers and discs moistened with diamond suspension. To reveal the structure as a reagent, a 5% solution of HNO$_3$ in ethyl alcohol was used. The etching time was 30 s. Observations of the structure of the investigated steels were carried out using the Axio Observer light microscope from Zeiss. The pictures of the structures of tested steels were made at 200× and 500× magnifications.

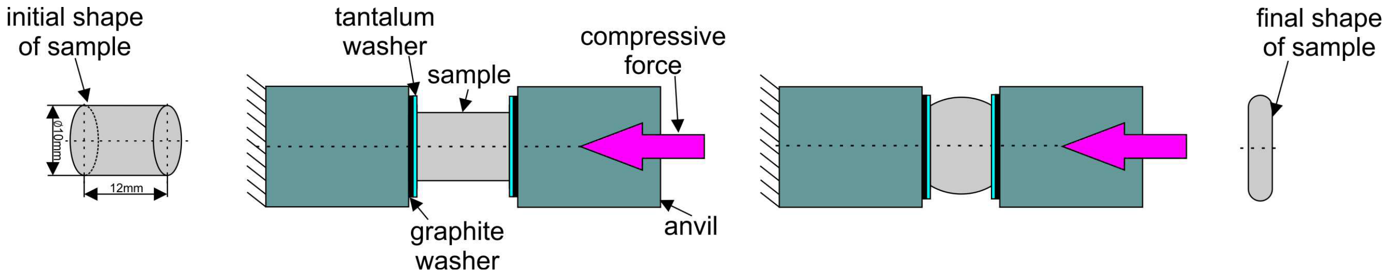

Fig. 5. Diagram showing the compression of axisymmetric samples in the Gleeble 3800 thermo-mechanical simulator

3. Results and discussion of results

The two-stage compression tests of the axisymmetric samples in the Gleeble 3800 thermo-mechanical treatment simulator allowed to state that during the isothermal heating between the first and the second stage of the deformation, partial or even complete disappearance of strain hardening takes place. This process is dependent on three main factors: the deformation temperature, strain rate and above all of the isothermal heating time between successive stages. The disappearance of strain hardening is a consequence of the course of static recovery and static recrystallization. The results of investigations of the disappearance of strain hardening in the intervals between the first and the second stage of deformation at the speed of 10 s^{-1}, for X98 steel are presented in the table 2, while for X105 steel in the table 3 and graphically depicted in figures 5 and 6 respectively. Based on the obtained results, a trend line was established. Achieving the total disappearance of the strain hardening requires a recrystallization time of well over 200 s for both tested steels (Figs. 6,8). In turn, the time $t_{0.5}$ needed to produce 50% of the recrystallized fraction for X98 steel is about 3 seconds at the test temperature of 1000°C and 10s at 900°C (Tab. 2, Figs. 6,7). However, the time $t_{0.5}$ needed to produce 50% recrystallized fraction is longer for steel X105 and is 8 s at the test temperature of 1000°C and increases to 26 s with decreasing deformation temperature to 900°C (Tab. 3, Figs. 8,9) as determined using the approximation shown in figure 8. Micro-additives Nb and Ti in X98 steel affect the grain refinement, which means that the time required for the half-recrystallization of austenite is shorter than in X105 steel. As a result of the increase of the plastic deformation temperature from 900°C to 1000°C, the static recovery time is shortened as well as the acceleration of the softening kinetics of the deformed austenite. At a higher temperature of plastic deformation there is a greater mobility of the grain boundaries, thereby increasing the rate of recrystallization [9,10]. The obtained results show that the total course of austenite recrystallization needs long isothermal heating times, which are not used in industrial rolling lines for economic or technological reasons. In industrial conditions, the time required for half-recrystallisation of austenite $t_{0.5}$ is often used in initial rolling passages. Determination of the half-time value of recrystallization is important in order to properly design individual stages of thermo-mechanical treatment and, as a consequence, stages and intervals between individual culverts of hot rolling in industrial conditions.

Figures 7a and 9a show the structures of tested steels in the initial state, these steels are characterized by austenitic-ferritic structure with participation of carbides as confirmed by detailed microscopic studies using light microscopy, scanning electron

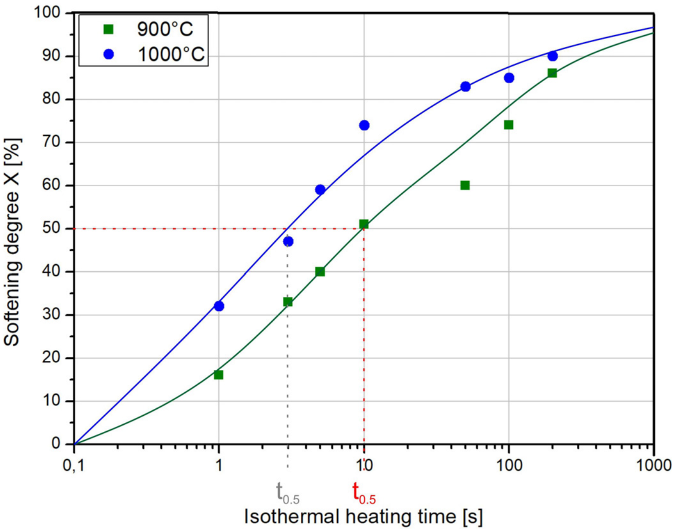

Fig. 6. Influence of isothermal heating time between the first and second strain on the kinetics of austenite recrystallization deformed in a hot compression test ($\dot{\varepsilon}$=10s^{-1}, ε = 0.2, T_o = 900°C and 1000°C) in a Gleeble thermo-mechanical simulator for X98 steel

TABLE 2

The results of research on the disappearance of austenite strain hardening in the intervals between the first and the second stage of hot plastic deformation of X98 steel at 900 and 1000°C

Deformation temperature T_o [°C]	ε_1	$\dot{\varepsilon}$ [s^{-1}]	Isothermal heating time, [s]	ε_2	$\dot{\varepsilon}$ [s^{-1}]	σ_0	σ_1	σ_2	Softening degree X, [%]
900	0.2	10	1	0.2	10	329	419	405	16
			3			322	425	391	33
			5			315	421	378	40
			10			334	414	374	51
			50			324	397	353	60
			100			332	411	353	74
			200			328	421	341	86
1000	0.2	10	1	0.2	10	254	303	287	32
			3			203	302	255	47
			5			200	300	241	59
			10			226	302	246	74
			50			192	310	213	83
			100			234	300	244	85
			200			220	289	227	90

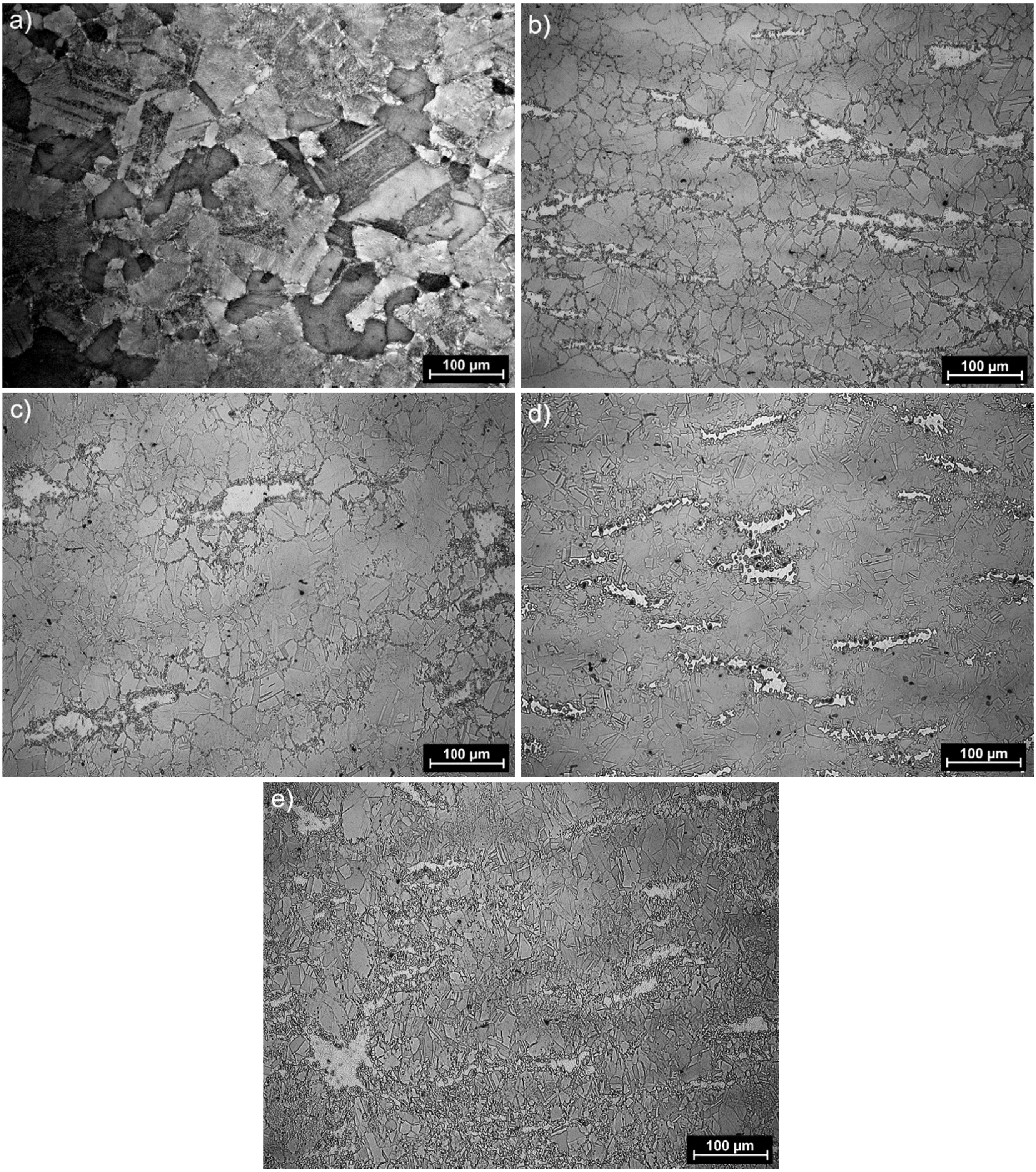

Fig. 7. Evolution of the structure obtained during two-stage hot deformation tests for X98 steel with a true strain equal 0.2 and isothermally held for the time from 1 to 200 s between deformations steps: ($\dot{\varepsilon}$ =10 s^{-1}, ε = 0.2, T_o = 900°C): a) initial state, b) after 1 s of isothermal heating, c) after 10 s of isothermal heating, d) after 200 s of isothermal heating, e) after second compression step with 200 s of isothermal heating between deformations steps and then finally all specimens were cooled with water under pressure

microscopy including EBSD research and high resolution transmission electron microscopy of steel in the initial state and described in detail in the publications [14,15]. In the next figures 7b-e and 9b-d, evolution of the structure of the investigated steels as a function of isothermal holding time are shown after the first true strain equal to 0.2 at 900°C for X98 steel, figures 7b-d, at 1000°C for X105 steel figures 9b-c and after the second deformation with a true strain of 0.2 (Figs. 7e,9d). True strain value equal 0.2 at both 900°C for X98 steel and 1000°C for X105 steel is enough to initiate the dynamic recrystallization process, while the use of isothermal holding between individual stages of deformation was to determine the time required to

TABLE 3

The results of research on the disappearance of austenite strain hardening in the intervals between the first and the second stage of hot plastic deformation of X105 steel at 900 and 1000°C

Deformation temperature T_o [°C]	ε_1	$\dot{\varepsilon}$ [s^{-1}]	**Isothermal heating time, [s]**	ε_2	$\dot{\varepsilon}$ [s^{-1}]	σ_0	σ_1	σ_2	**Softening degree X, [%]**
900	0.2	10	1	0.2	10	353	400	393	18
			3			345	381	373	28
			5			339	394	375	35
			50			334	399	365	58
			100			332	398	355	75
			200			326	376	332	90
1000	0.2	10	1	0.2	10	255	275	270	25
			3			220	265	251	35
			5			229	277	255	46
			50			248	274	256	75
			100			247	274	254	80
			200			246	264	247	94

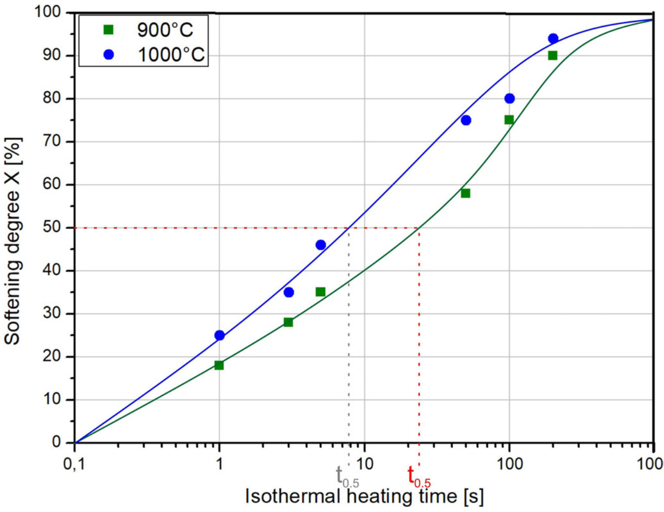

Fig. 8. Influence of isothermal heating time between the first and second strain on the kinetics of austenite recrystallization deformed in a hot compression test ($\dot{\varepsilon} = 10\text{s}^{-1}$, $\varepsilon = 0.2$, $T_o = 900$°C and 1000°C) in a Gleeble thermo-mechanical simulator for X105 steel

obtain 50% fraction recrystallized as a result of static processes, it is static and metadynamic recrystallization occurring during this isothermal holding. Isothermal holding of X98 steel after the first deformation at 900°C for 1 second is too short time to initiate static recrystallization. Based on previous studies for this type of steel, it was found that the incubation time necessary to initiate static recrystallization is from a few to several seconds, so the only process that removes the effects of strain hardening taking place during such short time is the metadynamic recrystallization [8,10,19,20,24]. The recrystallized ferrite grains are distributed along the boundaries of the austenite grains (Fig. 7b). Extending the isothermal heating time of samples to 10 s results in an increasing in the proportion of recrystallized grains to approximately 50% (Fig. 7c). A further increase in the time of isothermal holding of X98 steel to 200 s results in an increase in the proportion of fine recrystallized grains, however, with the participation of grains in which no recrystallization has occurred (Figs. 7d-e). A second deformation at 900°C with a true strain of 0.2 and automatic cooling with water under pressure causes refinement of the structure due to dynamic recrystallization occurring during the hot plastic deformation steps, and also isothermal holding between these deformation stages causes that effects of strengthening is removed by static and metadynamic recrystallization. However, in the case of X105 steel, after 5 seconds of isothermal strength at 1000°C, the proportion of recrystallized grains is almost 50% (Fig. 9b). Increasing the isothermal endurance time to 100s results in an increase in the proportion of fine grains recrystallized statically (Figs. 9c-d).

4. Summary

The temperature of deformation, the strain rate and the isothermal heating time between successive passes are factors influencing the disappearance of strain hardening, which is the result of the course of static recovery and static recrystallization. The results shown reveal that long isothermal heating times are needed to obtain 100% recrystallization of austenite. In industrial conditions, such long heating times for technological and economic reasons are not used, therefore the heating time needed for the so-called half-recrystallization of austenite $t_{0.5}$ is often used in the rolling passages. In order to design an appropriate and correct thermo-mechanical treatment, it is important to determine the half-time recrystallization which in this work was done for two experimental high-manganese X98 and X105 steels, which on the basis of other tests performed in the analysis of their structure and properties have the potential for practical application in real conditions or they may interest producers of modern construction materials.

The total disappearance of the strain hardening i.e. the total course of austenite recrystallization, of the X98 and X105 high-manganese TRIPLEX type steels, requires an isothermal heating time of well over 200 s. Half-recrystallization time of

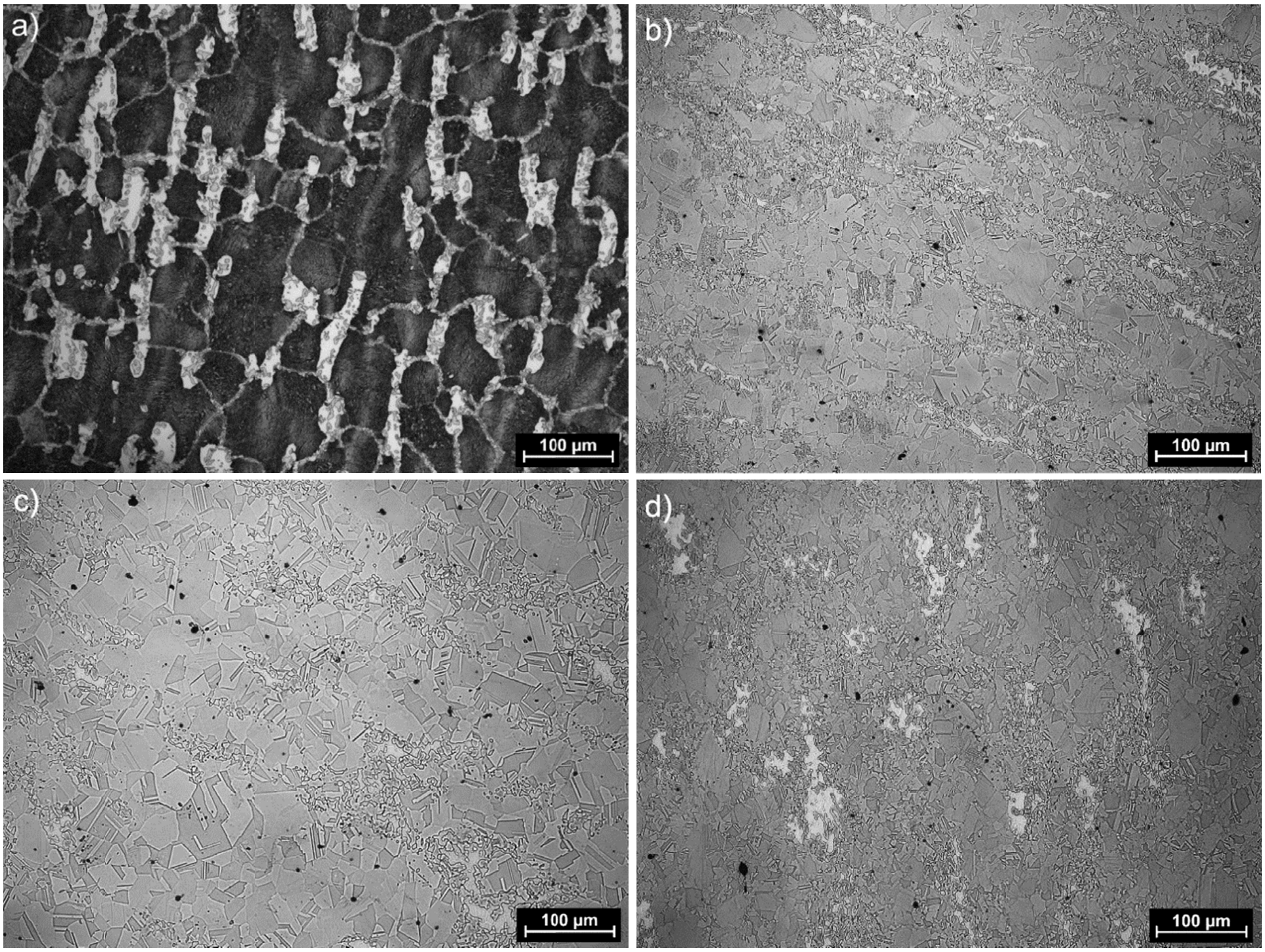

Fig. 9. Evolution of the structure obtained during two-stage hot deformation tests for X105 steel with a true strain equal 0.2 and isothermally held for the time from 1 to 100s between deformations steps: ($\dot{\varepsilon} = 10$ s^{-1}, $\varepsilon = 0.2$, $T_0 = 1000$°C): a) initial state, b) after 5 s of isothermal heating, c) after 100 s of isothermal heating, d) after second compression step with 100 s of isothermal heating between deformations steps and then finally all specimens were cooled with water under pressure

X98 steel at 900°C is 10s, for X105 steel this time is almost three times longer and amounts to 26 s. To produce 50% of the fraction recrystallized at 1000°C in the case of X98 steel, only slightly more than 3 s are needed, while in X105 steel, similarly to 900°C, the time is much longer and amounts to 8 s. The increase in deformation temperature is a factor affecting the acceleration of disappearance of the strain hardening, this is caused by the greater mobility of the grain boundaries at a higher temperature of plastic deformation.

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