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# Structural and mechanical behaviour of TRIP-type microalloyed steel in hot-working conditions

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**Properties**

## ABSTRACT

**Purpose:** The aim of the paper is to investigate the influence of various deformation conditions on microstructure evolution and flow curves of TRIP-type steel.
**Design/methodology/approach:** In order to determine the influence of MX-type interstitial phases on limiting the grain growth of primary austenite, samples were quenched in water from a temperature range, from 900 to 1200°C. Determination of processes controlling strain hardening was carried out in compression test using Gleeble 3800 simulator. The σ-ε curves were defined in a temperature range from 850 to 1150°C, for 0.1, 1 and $10s^{-1}$ of strain rate. To determine the progress of recrystallization samples were isothermally held for up to 60 s at 900 and 1000°C.
**Findings:** Profitable impact of TiN and NbC particles on austenite grain growth limitation is present up to 1050°C. The values of flow stress are equal from 120 to 270 MPa. The steel is characterized by quite high values of deformation, $\varepsilon_{max}$=0.4-0.65, corresponding to maximum stress on σ-ε curves. Beneficial grain refinement of primary austenite microstructure can be obtained due to static recrystallization. In temperature of 1000°C, $t_{0.5}$ is equal 35 s and elongates to 43 s after decreasing deformation temperature to 900°C. The σ-ε curves obtained during multi-stage compression tests confirmed that a process controlling the strain hardening is a dynamical recovery.
**Research limitations/implications:** To design hot-rolling conditions, the analysis of the primary austenite microstructure evolution during successive deformation cycles should be carried out.
**Practical implications:** The obtained precipitation kinetics of MX-type phases and σ-ε curves are useful in determining hot-rolling conditions ensuring the fine-grained microstructure of primary austenite.
**Originality/value:** The determined true stress–true strain curves were obtained for the TRIP-type microalloyed steel containing decreased Si concentration.
**Keywords:** Metallic alloys; TRIP-steel; Hot-compression test; Dynamic recovery; Dynamic recrystallization; Static recrystallization

## 1. Introduction

High-strength steels with Nb, Ti and V microadditions have been applied with success for diverse car parts and other means of transport for many years. Depending on ferritic, ferritic-pearlitic, ferritic-bainitic, bainitic or tempered martensite microstructure formed in the manufacturing process, parts made of these steels are characterized by diversified yield strength, tensile strength and formability in processes of bending, forming and others [1]. In the

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period of last twenty years, new groups of steels applied more commonly in the automotive industry have been developed. They meet the requirements in the aspect of particular formability during sheet-metal forming (Interstitial Free, Bake Hardenable, IS isotopic type steels) [2-4], combination of high strength, ductility and susceptibility to strong work hardening (Dual Phase type steels) [5,6] or ability to absorb considerable amounts of energy in the conditions of collision (TRIP and Complex Phase type steels) [7-12]. Hardening of these steels is induced by the impact of few mechanisms, which can be used together or separately. They are, among others: solution hardening, precipitation strengthening, strain hardening, grain refinement, and formation of desired texture or material with characteristics of composite material.

Mechanical and technological properties achieved by dual-phase ferritic-martensitic microstructure appeared to be particularly useful for automotive industry. They are: good combination of high strength and ductility, lack of clear yield point, low value of $YS_{0.2}$/UTS proportion and connected with it strain hardening [5, 13]. Presence of hard martensite islands in these steels decides about their strong strain hardening in the initial stage of technological forming [6]. This feature can be disadvantageous when a ready element has a complex shape and requires application of higher technological strain, e.g. in the process of sheet-metal forming. Technologically unintentional localization of strain in the soft ferritic matrix is, in such conditions, a fundamental problem making impossible to obtain a good drawpiece, i.e. without folds, cracks or excessive thinning of the sheet [14].

Different course of strain hardening is present for multiphase steel strengthened through TRIP (Transformation Induced Plasticity) effect [15-18]. After cold rolling, sheets made of these steels are annealed in a range of temperatures between $A_{c1}$ and $A_{c3}$ with successive isothermal holding in a range of bainitic transformation or they're cooled in a controlled way directly from the temperature of hot rolling finish. Microstructure of these steels is ferritic-bainitic with retained austenite, which delays thinning of sheets when transforming into martensite during sheet-metal forming. Gradual transformation of retained austenite into martensite preventing localization of plastic strain creates a possibility to apply higher technological deformations in the manufacturing process of ready parts. Obtaining 10-15% of retained austenite is possible due to the presence of approximately 1.5%Si in these steels, effectively delaying precipitation of cementite during isothermal holding of steels in the bainitic range [10, 12, 18]. However, such concentration of silicon is the cause of lack of adequate wettability by liquid zinc in the process of hot galvanizing, making impossible to assure a proper corrosion resistance of these sheets [16, 19]. It was found [19] that the concentration of silicon effectively preventing cementite precipitation should be equal around 0.8% and the rest, necessary for conserving required portion of retained austenite in the microstructure, can be complemented by Al or P [12, 20]. Aluminium, likewise silicon, is insoluble in cementite and delays its precipitation in the conditions of isothermal bainitic transformation. Additionally, this element accelerates formation of bainite, which creates chances to adopt existing technological lines of galvanizing originally designed for necessities of IF-type steels [3, 19].

It was found [21], that the arrangement and grain size of $\gamma$ phase has an influence on stability of retained austenite. The size of bainitic-austenitic islands can be beneficially decreased by production of TRIP-type steels using thermo-mechanical processing method [7, 8]. In this case, microadditions introduced into the steel, which connecting in dispersive particles of carbonitrides additionally increase mechanical properties of the steel through precipitation hardening and grain refinement, play an essential role [22-24]. Obtaining assumed multiphase microstructure of hot-rolled sheets requires determination of power-force parameters of hot-working. These problems, relating to TRIP-type steels, are rarely the subject of investigations, which for this steel group focus mainly on optimalization of heat treatment conditions and determination of retained austenite into martensite transformation kinetics in the conditions of cold plastic deformation [15, 16, 21]. Determination of TRIP-type steels behaviour in the conditions of hot-working, apart from technological features, should take into consideration the development of microstructure in the successive stages of hot-working. Applied approach should be similar as in case of commonly used C-Mn [25, 26], microalloyed, IF-type [27, 28] and DP-type [29] steels. Conditions of physical simulation of rolling process, apart from defining its power-force parameters, allow determining processes controlling the course of strain hardening as well as processes removing strain hardening in the intervals between successive sequences of deformations and after rolling is finished, leading to formation of fine-grained microstructure of the steel. These problems are the subject of physical and mathematical modelling taking into account the influence of variable deformation parameters on development of microstructure of the steel [29, 30]. Assurance of fine-grained microstructure of primary austenite before initiation of transformations during cooling, guarantees the fine-grained microstructure of products of supercooled austenite transformations [7, 8].

## 2. Experimental procedure

The investigation was performed on C-Mn steel with microadditions and chemical composition presented in Table 1. In comparison to TRIP-type steels used most often, elaborated steel is characterized by decreased to 0.87% concentration of silicon, which decrement was compensated by addition of Al as well as Nb and Ti microadditions, for which a positive impact on thermal stabilization of retained austenite was stated [7, 19]. Moreover, the steel is characterized by high metallurgical purity connected to low concentration of phosphorus (P=0.01%) and sulphur (S=0.004). The melts together with modification of non-metallic inclusions by rare-earth elements were done in Balzers VSG-50 vacuum induction furnace. Liquid metal was cast into ingot moulds with 25 kg capacity in argon atmosphere. Ingots, after cutting off the top and base, were subjected to forging into 220 mm wide and 20 mm thick flat bars, from which 10 mm diameter and 12 mm in length samples were prepared.

Table 1.
Chemical composition of the investigated steel

| Mass contents, (%) | | | | | | | |
|---|---|---|---|---|---|---|---|
| C | Mn | Si | Al | P | S | Nb | Ti |
| 0.24 | 1.55 | 0.87 | 0.40 | 0.010 | 0.004 | 0.034 | 0.023 |

The preliminary examinations were performed in order to design several-stage hot compression simulating final roll passes. Obtaining fine-grained austenite microstructure requires a proper selection of plastic working conditions adjusted to the precipitation kinetics of nitrides and carbides of microadditions introduced into the steel. Decisive meaning for impeding the grain growth of primary austenite in successive stages of hot-working should belong to TiN, TiC and NbC, which precipitation kinetics in austenite are described by kinetic equations (1-3):

$$\log [Ti] [N] = 0.32 - 8000/T \tag{1}$$

$$\log [Nb] [C] = 3.04 - 7290/T \tag{2}$$

$$\log [Ti] [C] = 5.33 - 10745/T \tag{3}$$

where: [Ti], [Nb], [N], [C] – mass fractions of Ti and Nb as well as N and C dissolved in austenite at the T temperature (in K), the constants: A=8000, B=0.32 for TiN, A=7290, B=3.04 for NbC and A=10745, B=5.33 for TiC, given in [24] were used.

In order to determine the influence of MX-type interstitial phases on limiting the grain growth of primary austenite, samples were quenched in water from a range of austenitizing temperature, from 900 to 1200°C. Determination of processes controlling strain hardening was carried out in continuous compression test using Gleeble 3800 thermo-mechanical simulator. In order to eliminate welding of sample with a die and decrease the friction on die – sample contact surface, very thin tantalic foil covered with lubricant based on nickel was introduced between the contact surfaces. The σ-ε curves were defined in a temperature range from 850 to 1150°C, for 0.1, 1 and $10s^{-1}$ of strain rate. Identification of thermally activated processes controlling the course of strain hardening was performed through quenching of samples in water from the temperature of 950 and 850°C after applying true strain equal 0.2, 0.4 and 0.6. In order to determine the progress of recrystallization in the conditions simulating intervals between roll passes, part of samples was subjected to the heat treatment presented in Fig. 1. After true strain equal 0.29 in the temperature of 1000 and 900°C, samples were isothermally held for up to 60 s and then cooled in water for microstructure freezing.

Determined data was used for elaboration of controlled thermo-mechanical processing conditions simulating final roll passes. Processing was also conducted on Gleeble 3800 thermo-mechanical simulator, implementing axisymmetrical samples. Degrees of reduction, strain rates and intervals between successive deformations (Fig. 2) were selected taking into account the conditions of planned hot-rolling of flat bars with initial thickness equal 5 mm into 2 mm thick sheets.

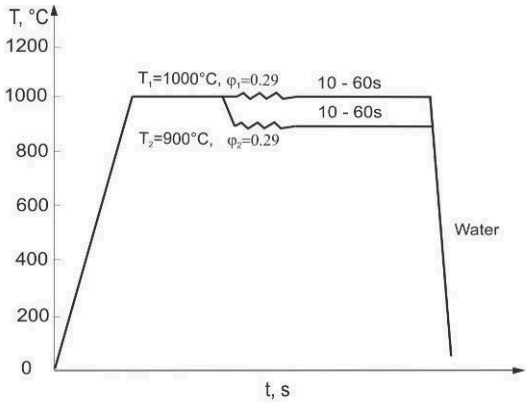


Fig. 1. Schematic representation of plastic deformation conditions for specimens isothermally held at 900 or 1000°C after plastic deformation to a true strain of 0.29

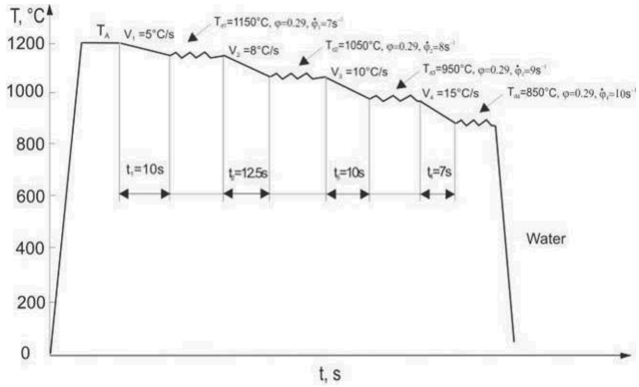


Fig. 2. Schematic representation of multi-stage compression test for specimens four times deformed to a true strain of 0.29

Metallographic tests of samples along with determination of grain size of primary austenite were performed on LEICA MEF4A optical microscope, equipped with Leica Qwin image analyzer. Grain boundaries of primary austenite were revealed after etching in saturated water solution of picric acid with addition of $CuCl_2$ in the temperature of 70°C. The participation of statically recrystallized austenite fractions was metallographically defined in the distance of 1/3 of radius from centre of the sample. In order to reveal real microstructure etching in Nital was applied.

## 3. Results and discussion

The change of grain size in the function of austenitizing temperature indicates that the steel is characterized by fine-grained microstructure of primary austenite up to the temperature of about 1050°C. In a temperature range from 900 to 1050°C, grain size of this phase slightly changes from 13 to 20μm (Fig. 3). It's confirmed by fine-grained microstructure of primary austenite of steel quenched from 1000°C, presented in Fig. 4. Further increase of temperature causes more intense increase of γ phase grain size, up to 48μm for temperature of 1200°C. Figs. 5 and 6 present microstructures of primary austenite, corresponding to this temperature range, of samples quenched from 1100°C and 1200°C temperatures. Thickened grain boundaries of primary austenite as well as regions between packets of martensite laths indicate the presence of retained austenite in microstructure of quenched specimens. It's confirmed in Fig. 7 showing microstructure of lath martensite of steel quenched from the temperature of 1200°C. Retained austenite, in a form of characteristic polygonal grains, is located along previous grain boundaries of γ phase and also between laths of martensite. It confirms the effectiveness of chemical composition selection in the aspect of thermal stabilization of retained austenite, what is the essential factor for TRIP-type steels manufacturing.

Fine-grained microstructure of the steel is probably the result of influence of dispersive particles of interstitial phases in a form of titanium and niobium carbonitrides. It arises from calculations performed in accordance with equations (1-3), that TiN will be the first phase precipitating in austenite. The beginning of TiN precipitation occurs in the temperature equal around 1480°C.

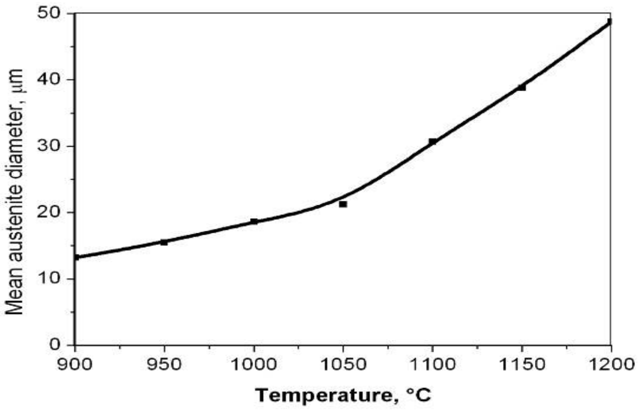


Fig. 3. Influence of austenitizing temperature on a grain size of primary austenite


Fig. 4. Structure of primary austenite of the specimen water-quenched from a temperature of 1000°C

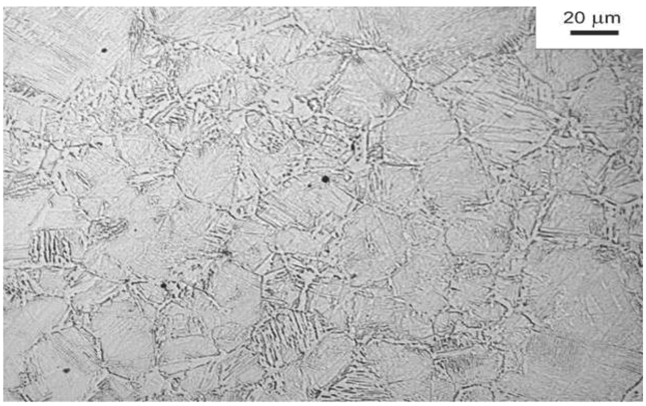


Fig. 5. Structure of primary austenite of the specimen water-quenched from a temperature of 1100°C

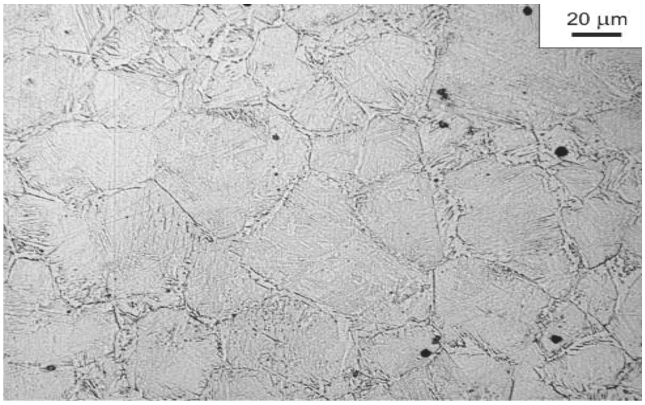


Fig. 6. Structure of primary austenite of the specimen water-quenched from a temperature of 1200°C


Fig. 7. Lath martensite structure containing retained austenite of the specimen water-quenched from 1200°C

Along with decrease of temperature, gradual decreases of participation of Ti dissolved in solid solution (Fig. 8a) as well as the increase of TiN participation occur (Fig. 8b). Complete binding of titanium in TiN is present at the temperature of approximately 1100°C. It arises from Fig. 9 that the initiation of NbC precipitation, which takes place at temperature of around 1180°C, imposes on TiN precipitation finish. Decrease of participation of niobium dissolved in austenite occurs together with decreasing the temperature, which in consequence causes the increase of NbC fraction (Fig. 9b). TiC is a next phase precipitating in the investigated steel. Assuming that the phase can dispose of total titanium concentration in the steel (0.023% wt.) - the beginning of TiC precipitation will occur at the temperature equal around 1150°C. In reality, the concentration of titanium should be decreased by concentration needed for binding the whole nitrogen in TiN. Considering atomic weights of Ti and N, concentration of titanium necessary for binding whole nitrogen into TiN is equal: 3.4x% wt. N = 0.0095% wt. Taking for calculations, in accordance with dependence (3), concentration of Ti = 0.0135% wt., the initiation of TiC precipitation decreases from a temperature 1150°C to 1100°C. In practice, temperature sequence of MX-type phases' precipitation in austenite, presented in Fig. 10, has greater meaning.

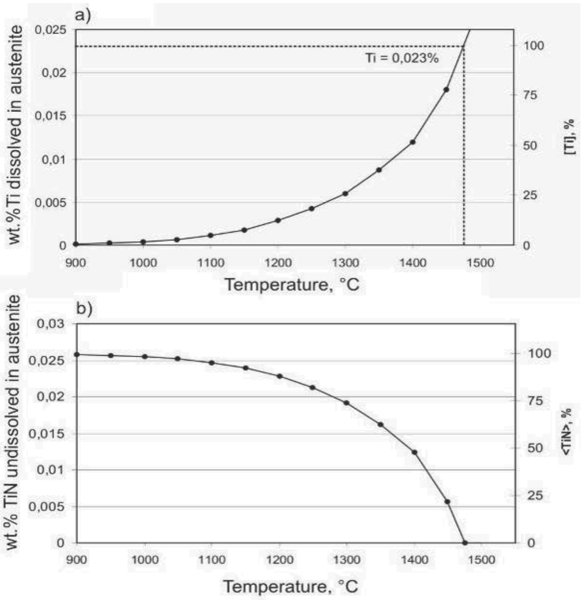


Fig. 8. Dissolution kinetics of TiN in austenite as a function of temperature; a) dissolution of Ti in solid solution, b) dissolution of TiN in solid solution

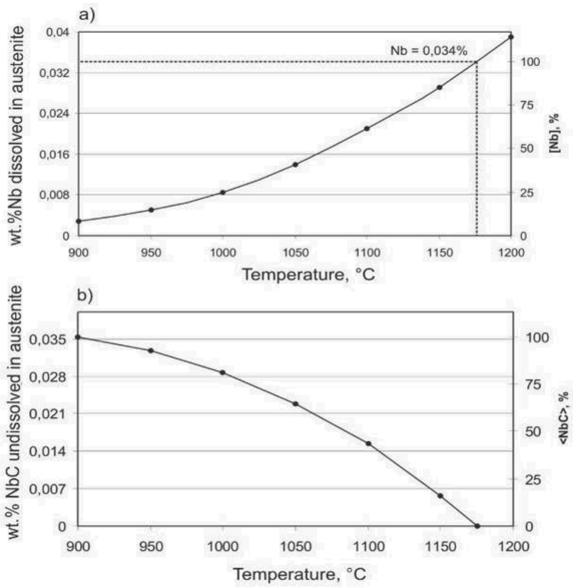


Fig. 9. Dissolution kinetics of NbC in austenite as a function of temperature; a) dissolution of Nb in solid solution, b) dissolution of NbC in solid solution

It derives from Fig. 10, that above 1200°C, TiN particles have an exclusive impeding influence on the grain growth of austenite. Additionally, starting from the temperature of 1180°C, NbC particles will precipitate and from 1150°C – precipitations of TiC. In reality, apart from TiN and NbC particles, (Ti,Nb)(C,N)-type complex carbonitrides should be expected, what was observed in steel with similar chemical composition [7]. These problems will be also the subject of further investigations of elaborated steel.

The flow curves presented in Fig. 11 indicate that the strain rate has great influence on the value of flow stress, which varies from 200 to 270 MPa for applied deformation conditions. However, no meaningful influence of strain rate in a range from 0.1 to $10s^{-1}$ on $\varepsilon_{max}$ deformation, corresponding with maximum flow stress, was observed.

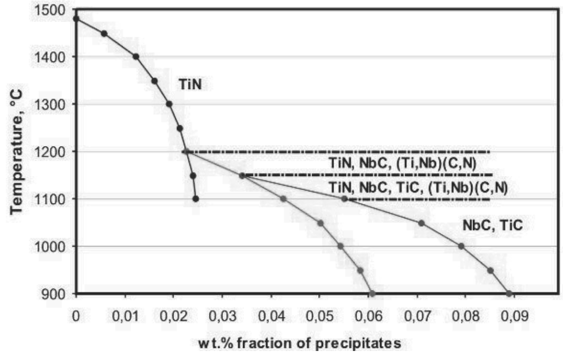


Fig. 10. Temperature sequence of MX-type phases precipitation in austenite of the investigated steel

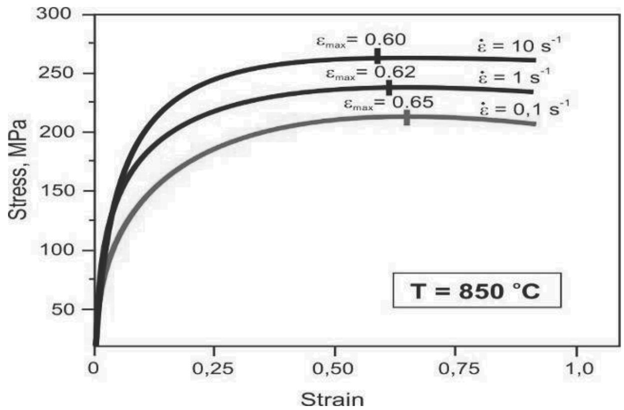


Fig. 11. Influence of strain rate on stress-strain curves of the specimens compressed at a temperature of 850°C

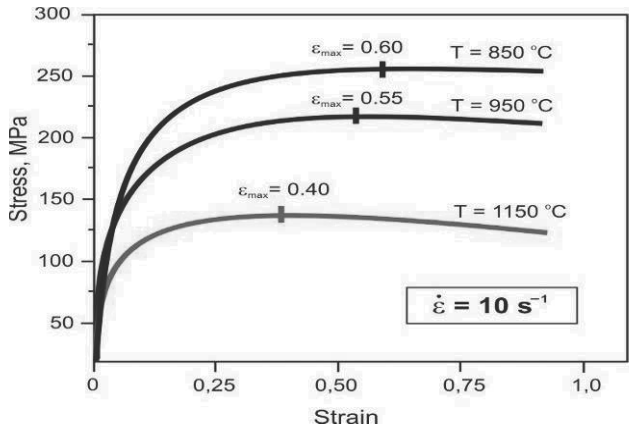


Fig. 12. Influence of temperature on stress-strain curves of the specimens compressed at a strain rate of $10s^{-1}$

It's characteristic that a state of dynamic equilibrium between the increment of dislocations generated during deformation and their decrement in the process of dynamic recovery is maintained in a quite wide range of strain. More meaningful influence has the deformation temperature - both on the value of $\varepsilon_{max}$ deformation and on increasing the slope of σ-ε curve after reaching the maximum value of flow stress (Fig. 12). For the temperature of 1150°C, deformation $\varepsilon_{max}$ is equal 0.4 and increases up to approximately 0.6 along with decreasing the temperature to 850°C. Example microstructures of primary austenite of samples quenched in water from the temperature of 850°C, after applying true deformation of 0.2, 0.4 and 0.6 with strain rate of $10s^{-1}$ are presented in Fig. 13a-c. Deformation of steel to true strain of 0.2 results in obtaining elongated grains of dynamically recovered austenite (Fig. 13a). Increase of true strain up to 0.4 leads to further elongation of grains of dynamically recovered austenite and initiation of dynamic recrystallization process (Fig. 13b). It's in accordance with data presented in [24], that the initiation of dynamic recrystallization can be started already at deformation $\varepsilon_{cd}$=(0.5-0.85)$\varepsilon_m$. Increase of true strain to 0.6 leads to formation of fine-grained microstructure of recrystallized grains (Fig. 13c).

In the industrial conditions forming fine-grained microstructure, in the consequence of dynamic recrystallization during hot rolling, is impossible due to the limitations coming from possible reduction that can be applied. It arises from Fig. 12 that dynamic recrystallization can partially proceed in the initial passes realized in a temperature range from 1200 to 1100°C. In the lower temperature range of rolling, refinement of microstructure should proceed with application of static, thermally activated processes. Development of microstructure of steel deformed at the temperature of 1000°C with reduction of 25% in the function of time of isothermal holding in this temperature is presented in Fig. 14.

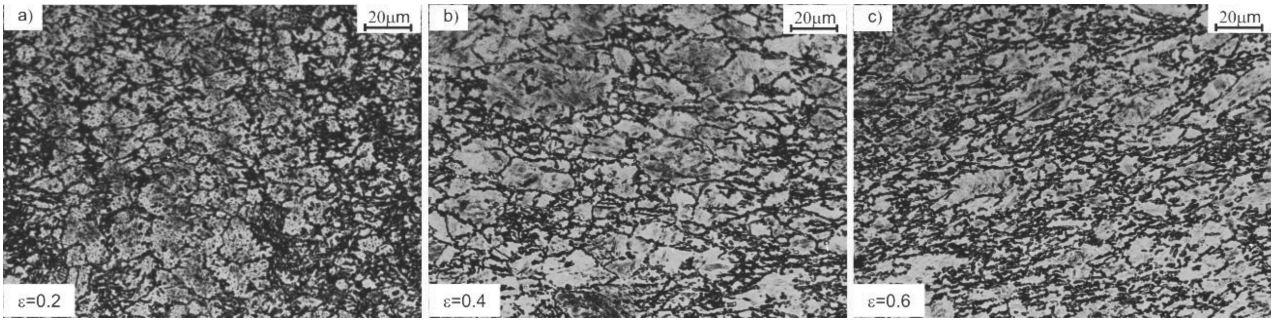


Fig. 13. Primary austenite microstructure evolution of the specimens quenched from 850°C after plastic deformation to a true strain of 0.2 (a), 0.4 (b), 0.6 (c)

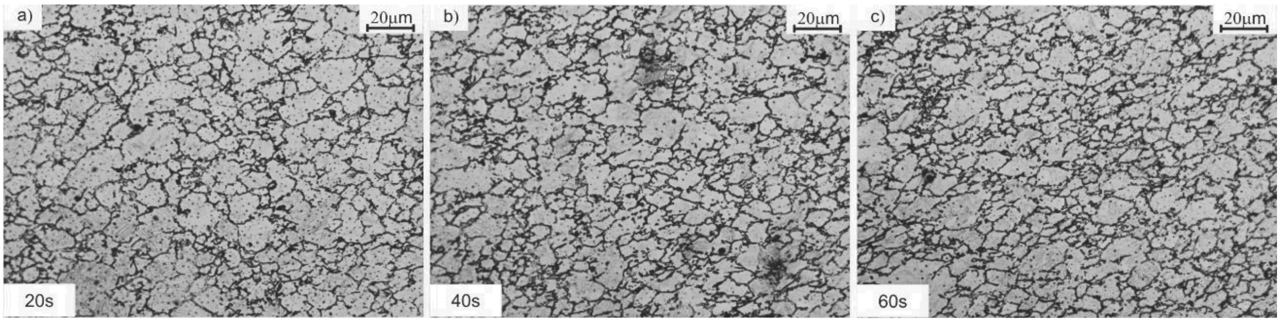


Fig. 14. Primary austenite microstructure evolution of the specimens quenched from 1000°C after plastic deformation to a true strain of 0.29 and isothermal holding for 20s (a), 40s (b), 60s (c)

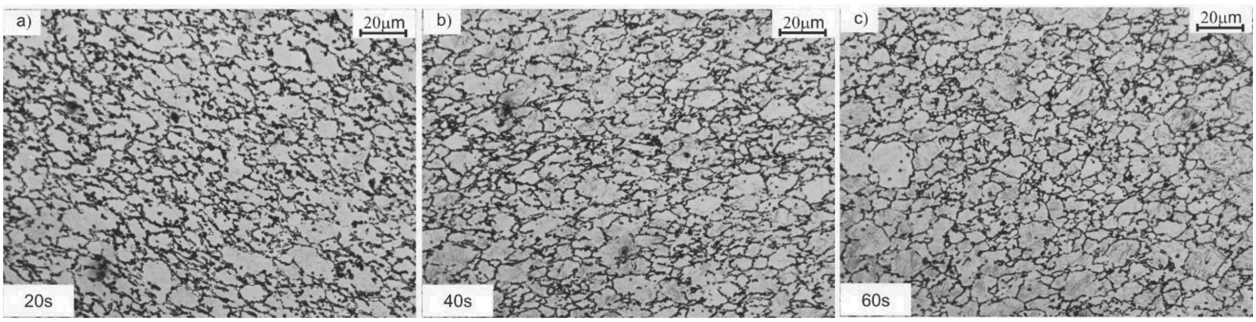


Fig. 15. Primary austenite microstructure evolution of the specimens quenched from 900°C after plastic deformation to a true strain of 0.29 and isothermal holding for 20s (a), 40s (b), 60s (c)

After 20 s of isothermal holding in the temperature of 1000°C, participation of recrystallized grains in relatively low. They are located along previous grains of primary dynamically recovered austenite with size of approximately 20μm (Fig. 14a). Elongation of holding time of specimens up to 40 s results in increase of statically recrystallized grains fraction up to around 55%, with average size of approximately 7μm (Fig. 14b). Apart from statically recrystallized grains, fine grains of retained austenite arranged uniformly in microstructure of the steel can be observed. Further time increase to 60 s leads to increase of fine recrystallized grains fraction, at simultaneous size decrease of grains in which recrystallization haven't occurred yet (Fig. 14c). Successive stages of primary austenite microstructure evolution after decreasing deformation temperature to 900°C are presented in Fig. 15a-c. Fine recrystallized grains, located solely along dynamically recovered austenite are visible after holding the samples for 20 s (Fig. 15a). Fine recrystallized grains located uniformly in the matrix of non-recrystallized grains can be observed after increasing the time of isothermal holding to 40 s (Fig. 15b). Increasing the time to 60 s results in obtaining remarkably fine-grained microstructure of primary austenite with fraction of recrystallized grains equal approximately 60% (Fig. 15c). The curves of recrystallization progress in the function of isothermal holding time are shown in Fig. 16. They allow concluding that the whole course of recrystallization of austenite requires long times, that are not accepted in rolling lines. In technical conditions, time necessary for the course of half-recrystallization of austenite is often applied. It arises from Fig. 16 that for 1000°C, it's equal $t_{0.5}$=35 s and elongates to $t_{0.5}$=43 s together with decreasing the temperature to 900°C.

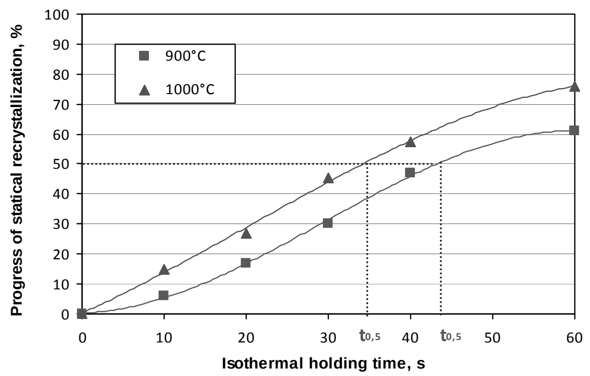


Fig. 16. Influence of the isothermal holding time on a fraction of recrystallized primary austenite of the specimens 25% deformed (true strain = 0.29) at 900 and 1000°C

Determined dependences between the influence of microadditions on kinetics of interstitial phases' precipitation in austenite as well as dependences between hot-working parameters (T, t, $\varepsilon$, $\dot{\varepsilon}$) and development of microstructure of primary austenite, served for elaboration of thermo-mechanical processing conditions presented in Fig. 2. The results of performed processing are $\sigma$-$\varepsilon$ curves shown in Fig. 17. According to the predictions, dynamic recovery is the process controlling strain hardening in a whole range of plastic working. Initially, gradual increase of flow stress occurs - from around 100 MPa to 180 MPa for the temperature equal 950°C. Applied intervals between passes allow partial course of static recrystallization. More intense increase of flow stress occurs for the temperature of last strain, equal 850°C, what is connected both with lower temperature and short time of the interval between third and fourth cycle of deformation. However, limitation of time results from adjusting the time of the interval to the time simulating cooling of 2.5 mm thick sheets between third and fourth deformation. Nevertheless, implemented conditions of cyclic deformation should result in fine-grained microstructure of primary austenite prior to further several-stage sheet cooling, typical for TRIP-type steels. It'll be the subject of further part of investigation.

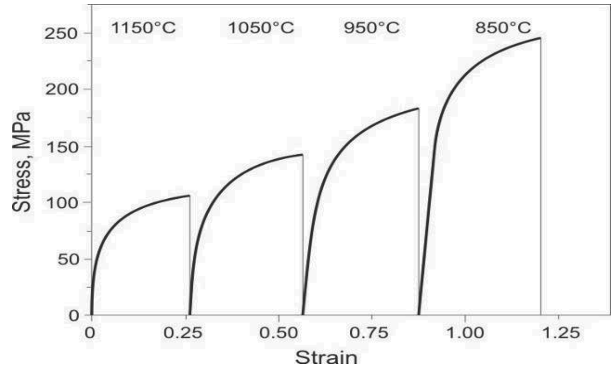


Fig. 17. Stress-strain curves corresponding to hot-working conditions in Fig. 2

## 4. Conclusions

Designing the conditions of hot-working assuring fine-grained microstructure of primary austenite prior the initiation of cooling directly from the temperature of plastic deformation finish should take into account many factors, which are among others:

- influence of austenitizing temperature on primary austenite grain size,
- influence of microadditions made to steel on their precipitation kinetics in austenite,
- influence of deformation temperature, reduction and a strain rate on the course of processes controlling strain hardening,
- influence of holding time between successive deformation cycles on the course of static processes that remove strain hardening,
- the possibility of conducting complex deformation cycles in the industrial conditions together with giving consideration to the thickness of a ready sheet.

Beneficial influence of TiN on primary austenite grain size causes that the stock can be austenitized in the temperature of 1200°C without a danger of excessive grain growth of $\gamma$ phase. Temperature range of hot-working was adjusted to the kinetics of TiC and NbC precipitation in austenite. Profitable impact of these particles on austenite grain growth limitation is present up to 1050°C. The values of flow stress in a temperature range from 850-1150°C are equal from 120 to 270 MPa and are comparable to the values noted for DP-type steels commonly used in automotive industry. Quite high values of deformation, $\varepsilon_{max}$=0.4-0.65, corresponding to maximum stress on $\sigma$-$\varepsilon$ curves, lead to the fact that the state of dynamic equilibrium between the increment of dislocations and their decrement in result of dynamic recovery is maintained in a quite wide range of strain. Beneficial grain refinement of primary austenite can be obtained during the intervals between successive cycles of deformation thanks to static recrystallization. At 1000°C, half-time of recrystallization of austenite is equal 35 s and elongates to 43 s after decreasing deformation temperature to 900°C. Affirmative effects of static recrystallization course in a temperature range of hot-working, from 1150 to 950°C, confirms a slight increase of flow stress on curves of several-stage compression, where during plastic deformation only dynamic recovery is occurring. The value of flow stress for last deformation realized in the temperature of 850°C reaches 250 MPa.

## Acknowledgements

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