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## STRAIN-INDUCED AUSTENITIC STRUCTURE IN MICROALLOYED STEELS

### INDUKOWANA ODKSZTAŁCENIEM STRUKTURA AUSTENITYCZNA W STALACH MIKROSTOPOWYCH

Austenite morphology is one of the main factors determining austenite-ferrite transformation kinetic and effectively affects the final microstructure and properties. The basic criteria for proper assessment of the austenite transformation products, their refinement, is the relation between the nucleation to growth rates. The main factor accelerating both, the nucleation rate of austenite during heating, and ferrite during cooling is the presence of accumulated deformation energy. The primary aim of this work is to increase our knowledge of the effects of deformation - its accumulated energy on the austenite structure and properties. Two specific steel grades were selected for the present investigation, i.e. microalloyed and IF steel, essentially different in equilibrium transformation temperatures. Obtained austenitic microstructures were analyzed, first of all as a start point for the austenite-to-ferrite transformation. Specific case of this transformation was considered i.e. Strain Induced Dynamic Transformation SIDT. The characteristic feature of the SIDT is the strong dependence of their kinetic on the austenite morphology, especially grain size. Thermomechanical processing, that utilize the SIDT, is one of the most effective ways to produce ultrafine-grained steel. One of the main benefits of the austenite refinement, just before the  $\gamma \rightarrow \alpha$  transformation, is its significant effect on the microstructure evolution during subsequent thermomechanical processing. Experimental results clearly show how direct and positive influence the austenite grain refinement has on the composition and refinement of transformation products. Presented study was focused on Strain Induced Dynamic Reverse Transformation. It is proved that this kind of transformation is very efficient way to intensify thermomechanical processing of microalloyed steels. Dynamic transformation kinetics were analyzed based upon flow curves recorded during the SIDT process. The main effect of presented research is analyze of influence of prior microstructure on dynamically formed austenite morphology.

*Keywords:* grain refinement, strain-induced dynamic transformation, microalloyed steel

Morfologia struktury austenitycznej jest jednym z podstawowych czynników rzutujących na przebieg przemiany austenit-ferryt, a co za tym idzie na własności końcowe struktury ferrytycznej. Podstawowym kryterium oceny przemiany austenitu pod kątem stopnia rozdrobnienia powstających produktów jego przemiany jest stosunek prędkości zarodkowania nowej fazy do prędkości jej wzrostu. Kluczowym czynnikiem przyspieszającym zarodkowanie zarówno fazy austenitycznej przy nagrzewaniu oraz ferrytu przy chłodzeniu jest obecność zakumulowanej energii odkształcenia. Podstawowym celem prezentowanych badań była ocena wpływu odkształcenia oraz wynikającej z niego energii zgromadzonej na właściwości struktury austenitycznej. Badania wykonano z wykorzystaniem dwóch charakterystycznych gatunków stali – mikrostopowej i typu IF, istotnie różniących się równowagowymi temperaturami przemian. Otrzymana struktura austenityczna była badana przede wszystkim pod kątem wpływu jej morfologii na kinetykę i produkty przemiany austenit-ferryt. Rozważany był szczególny przypadek tej przemiany – Indukowana Odształceniem Przemiana Dynamiczna (ang. Strain Induced Dynamic Transformation SIDT). Cechą charakterystyczną SIDT jest silna zależność jej kinetyki od morfologii austenitu. Zastosowanie przeróbki termomechanicznej z wykorzystaniem SIDT jest bardzo efektywnym sposobem wytwarzania stali ultra drobnoziarnistych. Podstawowym kryterium oceny jakościowej struktury austenitycznej jest jej stan przed przemianą, co bezpośrednio kształtuje skład mikrostruktury w wyrobie gotowym oraz stopień jej rozdrobnienia. W prezentowanej pracy skupiono się na indukowanej odkształceniem przemianie odwrotnej. Stwierdzono, że tą drogą można zintensyfikować procesy mikrostrukturalne wykorzystywane w przeróbce termomechanicznej stali mikrostopowych. Kinetykę przemian dynamicznych oceniano na podstawie analiz krzywych płynięcia zarejestrowanych podczas odkształcania w warunkach sprzyjających intensyfikacji przemian fazowych. Efektem głównym wykonanych badań jest analiza wpływu mikrostruktury wyjściowej na morfologię austenitu powstałego w wyniku przemiany dynamicznej.

### 1. Introduction

Grain refinement of metals still remains an essential mechanism that simultaneously improves mechanical proper-

ties without a loss in toughness. In recent years, a variety of methods have been developed to produce ultrafine grained (UFG) materials with a ferrite grain size of around  $1 \mu\text{m}$  [1,2,3,4]. One of the methods to produce steels containing ul-

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Chemical composition and equilibrium temperatures of investigated steels

Material	C	Si	Mn	Nb	Ti	Al	P	S	N	B	A <sub>e1</sub> , °C	A <sub>e3</sub> , °C
<b>Microalloyed</b>	0.07	0.29	1.36	0.06	0.03	0.02	0.015	0.006	0.0098	0.003	<b>670</b>	<b>843</b>
<b>IF steel</b>	0.004	0.003	0.164		0.08	0.039	0.0019	0.016	0.0019	0.0001	<b>893</b>	<b>912</b>

trafine grained ferrite is advanced thermomechanical processing (ATP) route that involves – among others – utilisation of ultrafine grained austenite as an initial structure [1]. Presented work focuses on one of the ways to produce this kind of microstructures i.e. Spontaneous Reverse Transformation (SRT)[5] and Deformation-Induced Ferrite Transformation (DIFT). First approach – SRT is a way of austenite formation during deformation above the Ae1 but below the Ac1 from martensitic starting structure. In the presented study, initial overheating above the Ae1 temperature and deformation heat is utilized to maximise volume fraction of austenite formed during transformation.

To fully highlight advantages of high refinement of austenitic structure, its influence on the second kind of Strain Induced Dynamic Transformation (SIDT) i.e. Deformation Induced Ferrite Transformation (DIFT) was investigated.

## 2. Research description

### 2.1. Material and samples

Two different steel grades were selected for the present investigation, with chemical composition shown in table 1. One of the main factors differentiating these two grades are equilibrium transformation temperatures, that in the present work were calculated using JMat Pro software and are also presented in table 1. As it was expected, in the case of IF steel, because of low carbon content, calculated values of Ae1 and Ae3 were higher and  $\alpha$ - $\gamma$  two-phase region was narrower than in the case of microalloyed steel. Another factor with great impact on thermomechanical processing possibilities is the presence of microalloying elements, especially Nb [4,6,7,8]. Content of this element in solid solution delays phase transformation during cooling, from the other hand, however, Nb(C,N) are potential nucleation sites for the  $\alpha \rightarrow \gamma$  transformation. Additionally, as it is well known, presence of B decreases the transformation temperature during cooling, what also results in increased SIDT process window [9]. These two factors significantly accelerate the phase transformation process, first of all due to a higher mobility of atoms, and easiness of overcooling below the two phase region.

### 2.2. Thermomechanical processing

The torsion tests were performed using servohydraulic testing rig. Solid bar torsion specimens were machined from as-hot rolled plate in perpendicular direction to the rolling direction (RD) [10]. Dilatation measurements were also taken during these tests. Two groups of thermomechanical tests were performed. The aim of the first part of the study was

to characterize austenite microstructure just after SIDT, and the purpose of the second test was to analyze an influence of austenite refinement on further thermomechanical processing. Additionally, in order to find processing parameters determining the kinetics of the dynamic austenite formation, two kinds of primary material microstructures were considered: as-received ferrite microstructure (Figure 1.a) and austenitized and water quenched (WQ) to fully martensitic microstructure (Figure 1.b)).

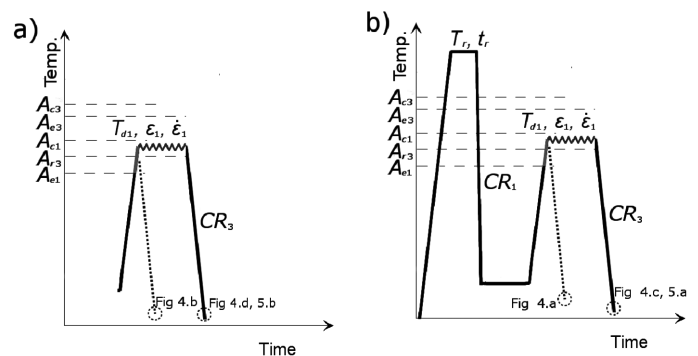


Fig. 1. Thermomechanical schedule designed to reveal morphology of dynamically formed austenite

In the second group of the tests, after first deformation, sample was reheated to fully austenitic range. Prepared in such a way microstructure has undergone thermomechanical treatment focused on the  $\gamma \rightarrow \alpha$  transformation including its special case i.e. DIFT. Main reason for choosing this kind of thermomechanical processing was its sensitivity to the initial austenite grain size. Table 2 shows process parameters applied during these tests. Test M1 was conducted to examine structure of the “strain-induced” austenite formed from as-received ferritic structure; M2 was similar, but with heat treatment applied to investigate the influence of martensitic structure on SRT; in schedule M3, static  $\alpha \rightarrow \gamma$  was applied. After austenitisation sample was cooled with air and deformed to induce DIFT; as opposed to the previous schedule M4 considers influence of UFG austenite (created in SRT) on static  $\gamma \rightarrow \alpha$  transformation; M5 is combination of two previous and contains SIDT (SRT and DIFT). Last two schedules were prepared to investigate DIFT in IF steels. In IF 1 schedule prior austenite structure was formed during static transformation and in schedule IF 2 UFG austenite was produced during SRT.

From the torsion tests, torque vs. twist angle data were recorded and calculated into equivalent plastic strain vs. equivalent stress data using standard equations. Additionally, temperature change and dilatation data were recorded during heating and cooling and – on that basis – critical temperatures were determined.

TABLE 2

Process parameters applied in the thermomechanical processing (Fig. 1)

Mat.	Sched.	$T_r$ , °C	$t_r$ , s	$CR_1$ , °C/s	$T_{d1}$ , °C	$\varepsilon_1$	$\dot{\varepsilon}_1$ , s <sup>-1</sup>	$T_{d2}$ , °C	$t$ , s	$CR_2$ , °C/s	$T_{d3}$ , °C	$\varepsilon_3$	$\dot{\varepsilon}_3$ , s <sup>-1</sup>	$CR_3$ , °C/s
Microalloyed	M1	-	-	-	800	1	1	-	-	-	-	-	-	-
	M2	1200	900	160										
	M3				-	-	-	900	300	30	765	1	1	160
	M4											-	-	
	M5				1	1								
IF	IF 1	1025	945	160	-	-	-	950	60	30	892	1	1	
IF 2	912				1	1								

where:

 $T_r$ –austenitization temperature, °C; $t_r$ –austenitization time, s; $CR_1$ – cooling rate after austenitization; $T_{d1}$ – temperature of first deformation, ( $A_{c1}-20$ ), °C; $\varepsilon_1$ – strain of first deformation; $\dot{\varepsilon}_1$ – strain rate during first deformation, s<sup>-1</sup>; $T_{d2}$ – temperature of the second deformation and

holding for strain-induced precipitation, °C;

 $\varepsilon_2$ – strain of second deformation; $\dot{\varepsilon}_2$ – strain rate during second deformation, s<sup>-1</sup>; $t$ – holding time for strain-induced precipitation; $CR_2$ – cooling rate before deformation of overcooled

austenite, °C/s;

 $T_{d3}$ – temperature of third deformation, ( $A_{r3}+20$ ), °C; $\varepsilon_3$ – strain of third deformation; $\dot{\varepsilon}_3$ – strain rate in third deformation, s<sup>-1</sup>; $CR_3$ – cooling rate after third deformation, °C/s.

### 2.3. Microstructure characterisation

Microstructural analysis was carried out by the means of both optical (OM) and scanning microscopy (SEM). Samples were prepared by the standard mechanical grinding and polishing procedures. For the optical microscopy samples were etched by a mixture of a solution of 1 vol%  $Na_2S_2O_3$  and the solution of 4 vol% of picric acid to identify a phase composition. Before the analysis of colored metallography, 1% nitric acid alcohol was used to erode samples to reveal the morphology of the microstructure [11]. Colored metallography was applied to distinguish microstructure components in order to make quantitative analysis of phase composition. To reveal the microstructure for SEM observations, the samples were etched in 1% Nital for 5 s.

## 3. Experiments results and their discussion

### 3.1. Identification of processing window

The first task, in order to obtain expected microstructural developments, was to define the process window for SIDT. It exists between equilibrium transformation temperature and static transformation start temperature. The latter temperature is determined by the morphology of initial phase and by the cooling rate. To determine deformation temperatures for SIDT and to take into account influence of ultrafine austenite, thermomechanical schedule presented in Figure 2.a was applied in the present study. In order to locate the start of static transformation, temperatures marked with dark points were measured. For precise determination of transformation start temperature, differential curve was superimposed on a dilatation graph (Figure 2.b). This curve determines the rate of the transformation and can be used to precise identification of transformation start with is represented by drop on this curve [12],[13].

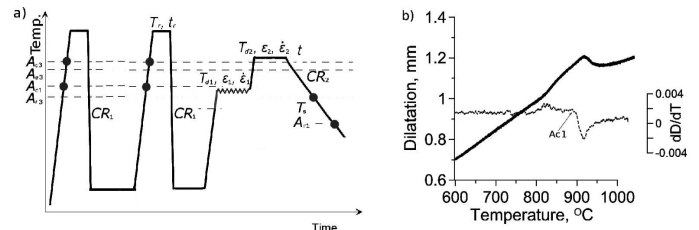


Fig. 2. Thermomechanical schedule a), Example of dilatation curve and differential curve for IF steel b)

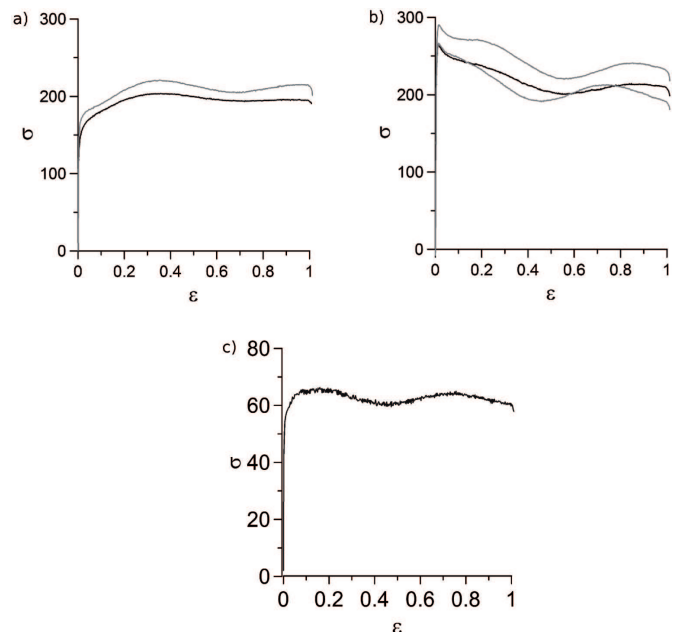


Fig. 3. Flow curves for transformation during heating from ferritic structure a) martensitic b) IF steel c)

### 3.2. Transformation kinetics

Presented in Figure 3 stress-strain curves show mechanical response of material during SIDT. Because this transformation is a kind of softening mechanism, presence of the transfor-

mation can be seen as a stress drop on this curve. Intensity of this drop is attributed to accumulated energy dissipation what, in turn, is directly connected with transformation kinetics. It can be seen that for both kinds of initial microstructure, flow stress stabilizes at a similar level. This phenomenon can be explained by similar accumulated deformation energy needed to induce a formation of austenite. Initial microstructure was substantially different what was confirmed by metallographic study and initial flow stress for both groups of samples: ferritic a) and martensitic b). At this moment authors have no information about similar results from other researchers, and thus, more tests are needed to confirm this thesis. It is also observed that in case of IF steel flow stress stabilized at much lower strain Figure 3.c. It can be explained by much higher intensity of transformation connected with higher transformation temperature. Black curves on Fig. 3.a, 3.b and 3.c correspond to deformation which resulted in microstructures presented on Fig. 5.a, 5.b and 6.a.

**3.3. Dynamic Reverse Transformation products**

The microstructures presented in Figure 4 clearly indicate influence of prior microstructure on austenite morphology. On Fig. 4.a structure of tempered martensite can be seen. On Fig. 4.b as-delivered ferritic microstructure is presented. Fig. 4.c presents frozen microstructure after deformation of martensitic microstructure above Ae1 temperature (M2). Fig. 4.d represents effects of similar treatment applied on initial ferritic phase (M1). In two last cases dark areas represent martensitic phase formed during water quenching from dynamically formed austenite. White phase is ferrite. It can be observed that arrangement of grain boundaries in prior structures is similar to locations of martensite (represents “strain-induced” austenite).

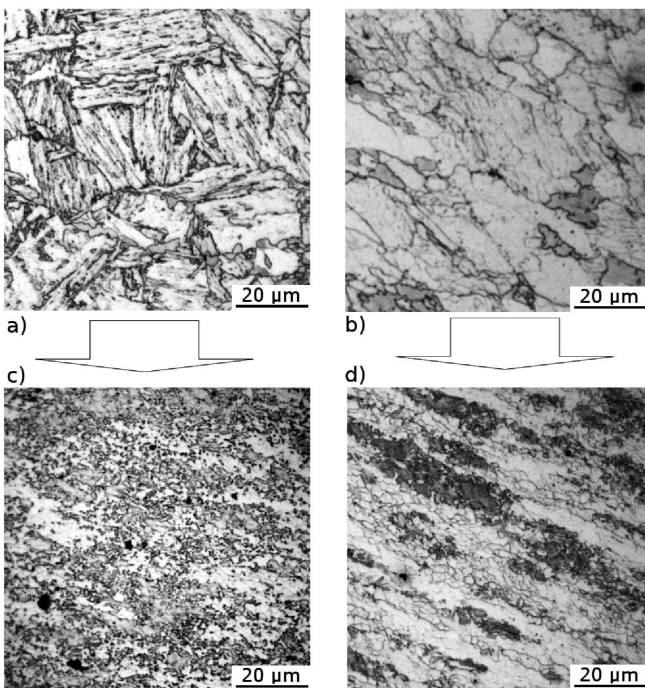


Fig. 4. Microstructures before a), b) and after SIDT +WQ c), d) from initial microstructure of ferrite a), c) and martensite b), d)

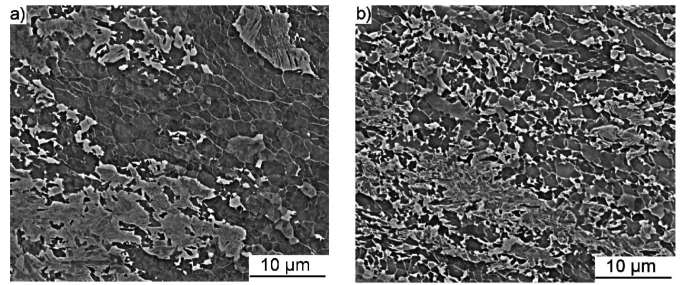


Fig. 5. SEM micrographs showing differences in the distribution of austenite, produced by the dynamic transformation from ferrite a) and martensite b)

Figure 5 shows differences in the distribution of austenite, produced by the dynamic transformation in microalloyed steel. Both microstructures contain martensitic phase. Throughout the analysis of this structure, prior austenite grain boundaries can be deduced. Detailed observation of these boundaries allows one to say that before water quenching if was an UFG austenitic structure. SEM observation confirms that austenite nucleating on tempered martensite was more disperse while austenite transformed from ferritic phase formed large separated areas. It can be explained by a higher number of potential nucleation sides in martensite. Another factor accelerating phase transformation is higher energy accumulated during plastic strain, caused by higher flow stress. Second phase that can be found on the micrograph is recrystallized ferrite. In schedule M1 ferrite grain size is significantly smaller from initial, what can be observed on figure 4.b and 4.d. On microstructure after schedule M2 prior martensitic phase during deformation passed in ultrafine grained austenite and fine grained ferrite. Based upon microstructural analysis it can be concluded that two softening mechanisms are present during deformation of presented material between Ae1 and Ac1 temperature. SRT, where the product is UFG austenite, and dynamic recrystallisation, where the effect is a large degree of ferrite grain refinement.

**3.4. Effects of the use of refined austenite**

The primary criterion for evaluation the effect of SIDT during heating of steels was its influence on further thermo-mechanical processing and microstructure of the final product.

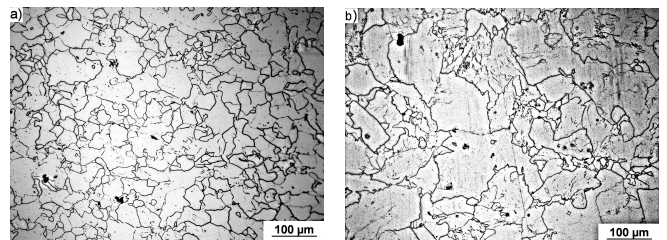


Fig. 6. Microstructure after dynamic transformation from refined a) and large grain austenite b)

Figure 6. shows influence of prior austenitic microstructure on final ferrite microstructure after thermomechanical processing of IF steel. Great difference in grain size can be observed. It can be explained that, because SIDT is a process controlled mainly by nucleation rate, volume fraction of austenite grain boundaries (main ferrite nucleation sites) is

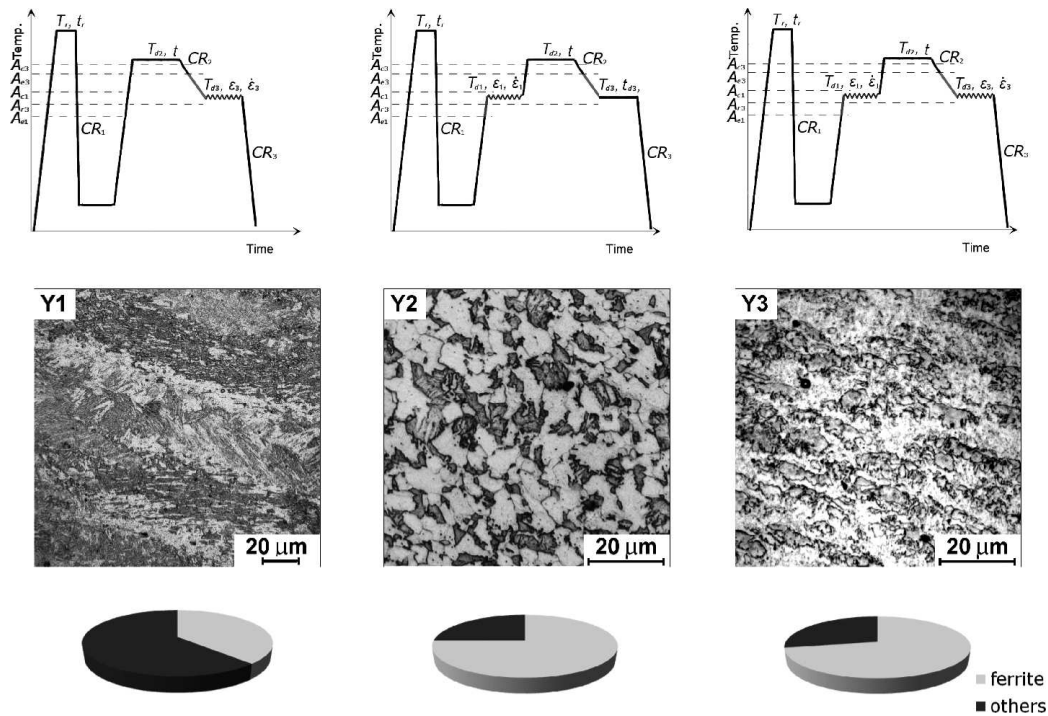


Fig. 7. Influence of prior phase composition on austenitic microstructure after transformation

critical for final microstructure refinement. Severely deformed ferrite grains can be observed in both cases. It proved presence of large deformation in the newly formed ferritic phase. Because these deformation effects appear in most of ferrite grains conclusion can be drawn that phase transformation was rapid, and occurred in most of the material volume at the beginning of deformation process, and further strain proceeded in mostly ferritic structure. At this point it is worth noting that observation of dilatation curve shows no signs of static transformation before start of the deformation.

The microstructures presented in Figure 7 clearly indicate influence of refined austenite microstructure on significant refinement of microstructure of the final product and transformation degree after  $\gamma \rightarrow \alpha$  transformation. It is also observed in the present study that the morphology of the prior austenite affects the final microstructures in various ways. For example, after SIDT of coarse grain austenite transformation degree is relatively low and most of ferritic phase is grouped close to the prior austenite grain boundaries. On the contrary, after SIDT of ultrafine austenite, martensite/bainite islands are "separated" while coarse-grained austenite produces "connected" areas. Morphology after only one dynamic transformation during cooling is closer to this after two dynamic transformations. However, in the case where transformation took place during cooling, the grain size of ferritic phase is larger, and morphology of ferritic/bainitic phase is significantly different. Such differences in microstructural features could influence the mechanical behavior of the final structural material. In general, it was observed in the present study that the progress of austenite transformation was significantly different for particular thermomechanical schedules. For microalloyed steel transformation degree was: M3 -43%, M4 -67%, M5 - 67%, respectively.

#### 4. Summary and conclusions

The following conclusions can be formulated as a result of the present study:

In case of microalloyed steels:

1. Ultrafine austenitic structure was obtained after SRT from both ferrite and martensite. Morphology of austenite formed from each phase was different.
2. Flow stress during transformation stabilizes at similar level for the initial martensitic and ferritic microstructure.
3. Ferrite recrystallisation was observed together with SRT.
4. Strong austenite grain refinement can intensify the microstructure development observed during thermomechanical processing of microalloyed steels.

In case of IF steel:

5. Substructure in ferrite grains in IF steel indicates that DIFT was very rapid and localized at the beginning of deformation.

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