

## DEVELOPMENT OF STRUCTURAL STEEL CONTAINING 3÷5 WT% Al WITH MICROLAMINATED MICROSTRUCTURE

The aim of this work was to develop basic parameters of hot rolling and controlled cooling technology allowing to obtain the microlaminated (lamellar) microstructure in a lean-alloy structural steel containing 3÷5 wt % Al. Thermo-mechanical rolling tests of two experimental steels were carried out in a semi-industrial line comprising a one – stand reversing rolling mill. The final microstructure of the specimens subjected to rolling in the  $\gamma + \alpha$  stability region characterised with the microlaminated morphology composed of lamellae of ferrite with thickness down to 1  $\mu\text{m}$  or less and lamellae or grains of phases developed during transformation of the austenite. Determined parameters of the thermo-mechanical processing allowed to achieve very attractive mechanical properties of the experimental steels: tensile strength over 1.0 GPa and ductility level (total elongation) better than 15%.

*Keywords:* high strength steel, alloying with aluminium, microlaminated microstructure

### 1. Introduction

Manufacturing of steel products with ultrafine equiaxed grains (of size close to 1  $\mu\text{m}$ ) or fine equiaxed grains (of size about 2-3  $\mu\text{m}$ ) in industrial conditions is technologically difficult and could be successful only after applying very stringent production parameters [1,2]. For certain steel compositions allowing for the existence of two-phase  $\gamma + \alpha$  field at temperatures of hot rolling, fine laminated (lamellar) microstructure could be produced using typical processing parameters. The microlaminated microstructure allows to obtain values of the yield strength comparable with properties of the products with fine grained equiaxed microstructure. This results from the relationship between the yield strength and the morphological parameters representing the microstructure. The contribution of the matrix unit (grain) size to the yield strength for the plate – like or the lath – like microstructure is represented by the following equation [3]:

$$\Delta\sigma_L = A \cdot (\bar{L})^{-n} \quad (1)$$

where  $\Delta\sigma_L$  is the contribution to the yield strength from plates or laths boundaries,  $\bar{L}$  is the mean slip plane length in plate (lath) – like microstructure and  $A$ ,  $n$  are material constants. For the fine plate or lath morphology  $n$  was found to be equal to 1. The mean slip plane length  $\bar{L}$  is about twice the mean true thickness of plates or laths  $\bar{t}$  [4] and therefore Eq. (1) could be written as:

$$\Delta\sigma_L = A \cdot (2\bar{t})^{-1} \quad (2)$$

In Fig. 1 schematic illustration of the laminated (lamellar) microstructure is shown.

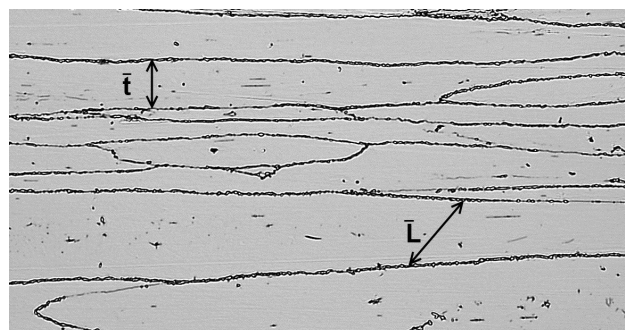


Fig. 1. Schematic drawings presenting laminated (lamellar) microstructure characterised by the mean slip plane length  $\bar{L}$  and the mean thickness of lamellae  $\bar{t}$

An addition of aluminium as alloying element to steel narrows the field of austenite stability and widens the two-phase  $\gamma + \alpha$  area [5,6], what facilitates producing laminated (lamellar) microstructure by means of hot rolling. In addition, aluminium is a significant solid solution hardener, producing for each 1 weight % of addition a 70-80 MPa increase in the yield strength [7-9]. A possibility of obtaining a laminated microstructure in hot rolled and controlled cooled dual phase steels alloyed with aluminium, containing 0.05-0.15% C, 5% Mn and 3% Al, has been recently proved experimentally by Zhang et al [10-12] (contents of the elements are given in weight % throughout this paper).

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Alloying of structural steels with aluminium (in amounts exceeding content needed for grain refinement) is not widely used and till now only automotive TRIP grades have been commercialised. Because the addition of aluminium, except for promoting ferrite formation at high temperatures and producing substantial solid solution strengthening also reduces steel density, many research activities have been undertaken aiming at development of structural steels based on Fe-Al-C [13-15] and Fe-Mn-Al-C systems [16-18]. In Fig. 2 approximate ranges of aluminium and carbon contents in three classes of Al – alloyed structural steels are shown.

The main aim of this work was to investigate the possibility of developing a hot rolling technology for production of microlaminated microstructure in lean-alloy structural steel based on Fe-(0.3% C)-(1.5÷2.5% Mn)-(3÷5% Al) composition. Phase transformations in the experimental steels were characterised by dilatometric methods and hot deformation behaviour was determined using the results of heating – deformation – cooling tests carried out in a laboratory (semi-industrial) hot rolling line. Microstructure of specimens subjected to various sequences of heat treatment and / or hot working operations was investigated by light microscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). Specimens subjected to specific processing variants were tested for mechanical properties.

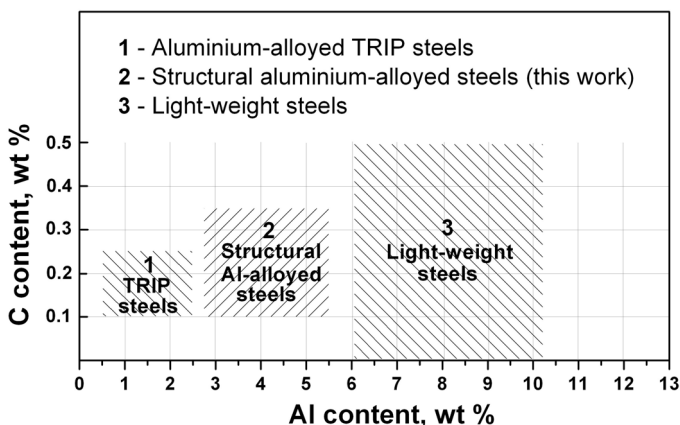


Fig. 2. Ranges of aluminium and carbon contents in Al-alloyed structural steels: range 2 comprises experimental steels investigated in this work

## 2. Experimental procedures

### 2.1. Preparation of experimental material

Chemical compositions of the experimental steels intended for investigation in the present work were designed taking into account results of preliminary tests and analyses, in that an experimentally developed metastable pseudobinary phase diagram for alloys Fe-(0-8%A1) with additions up to 0.35% C and up to 2.7% Mn, shown in Fig. 3. The diagram is of metastable character because the phases shown in the particular field of the diagram were identified after a short time of isothermal heating (10 minutes) to approximate parameters of the temperature cycles used in industrial thermo – mechanical processing.

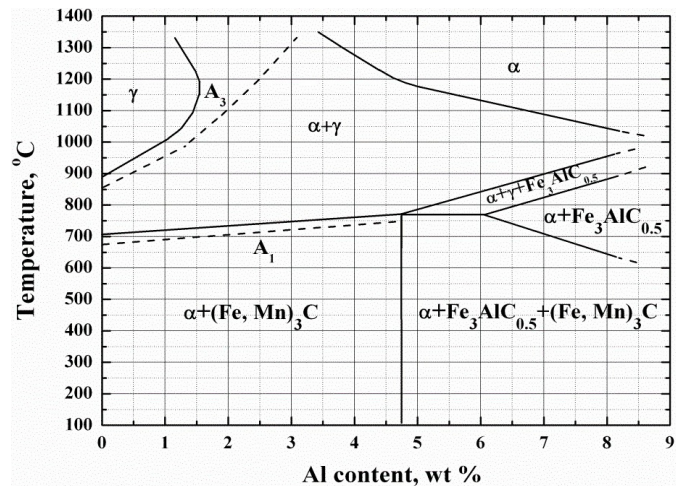


Fig. 3. Experimentally established metastable pseudobinary phase diagram (Fe+0.1% C+1.4% Mn)-Al, shown by the continuous lines; displacements of  $A_1$  and  $A_3$  temperatures in effect of increasing C content from 0.1% to 0.35% and Mn content from 1.4% to 2.7%, shown by the dashed lines

Because data concerning the effect of melting parameters on the metallurgical cleanliness of steels alloyed with Al are scarce (examples are [19,20]), one of the experimental heat was melted in a vacuum furnace and the other in an open furnace. Chemical compositions of the experimental steels are given in Table 1. Steel V was melted in a laboratory vacuum induction furnace of capacity 100 kg and cast in vacuum into ingot of 140 mm × 165 mm cross section, whilst steel A was melted in an open laboratory induction furnace of capacity 30 kg and cast into round ingot of  $\varnothing 122$  mm /  $\varnothing 145$  mm cross section. Next, the ingots were hot forged into bars of various cross sections suitable for preparation of particular kind of specimens used in the work, in that the bars for hot rolling experiments. Values of the austenite formation start temperature ( $Ac_{1S}$ ) and information on austenite formation finish temperature ( $Ac_3$ ), as determined by dilatometric method during the standard heating cycle, are also shown in Table 1.

TABLE 1

Content of alloying and residual elements in the experimental steels used in this work, wt %

Steel	C	Mn	Si	P	S	Al	N ppm	O ppm	$Ac_{1S}$ °C	$Ac_3$ °C
V	0.30	1.72	0.30	0.009	0.010	3.0	24	7	756	*
A	0.35	2.70	0.42	0.008	0.013	5.3	26	12	749	*

\* transformation to austenite was not completed during the heating cycle in the dilatometer (till 1100°C)

### 2.2. Microstructure examination and dilatometry

Microstructure was examined by optical microscopy (OM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). A scanning electron microscope FEI-Inspect F and an X-ray diffractometer Empyrean PANalytical were used. The X-ray

diffraction analysis was applied for measurement of the content of retained austenite (RA). The retained austenite measurement was conducted according to certified methodology using database of International Centre for Diffraction Data PDF-4+ (2011). A quantitative analysis of morphological parameters of microstructure was performed using a MetFlo 12.1 image analyzer.

Dilatometric investigation was carried out using a Bähr DIL 805 dilatometer to determine the critical temperatures of phase transformations and to perform experimental heat treatment cycles. Barrel – type specimens with 4 mm outside diameter, 2 mm inside diameter and 10 mm length were used. The critical temperatures were read out from the dilatometric curves by applying the tangent line and determining the deviation point using a computer programme.

### 2.3. Hot rolling experiments

The hot rolling trials were carried out in the laboratory using a semi – industrial line (LPS) consisting of: electric reheating furnace, descaler, one-stand reversing hot rolling mill, equipment for accelerated air and / or water cooling in the carry – away roller table area and electric furnace for heat treatment of the rolled products. The main difference between parameters of industrial rolling of plates and semi-industrial simulation in LPS concerns the values of strain rate. Strain rate during industrial rolling is typically in the range of  $8\div 15\text{ s}^{-1}$  while that of semi-industrial simulation in LPS is in the range of  $3.5\div 6.5\text{ s}^{-1}$ , depending on dimensions and properties of the rolled material and parameters of the rolling. In the present work the strain rate value was kept close to  $6.0\text{ s}^{-1}$ . Detailed description of the LPS facilities used in the semi-industrial rolling experiments was given in [21].

### 2.4. Determination of mechanical properties

Tensile tests were conducted at room temperature at the crosshead speed of  $10\text{ mm} \cdot \text{min}^{-1}$  using flat specimens machined along the rolling direction from the hot rolled in LPS flat bars

of thickness in the range of 3-12 mm. The tensile specimens fulfilled the geometrical similarity condition:

$$L_o / (A_o)^{1/2} = 5 \quad (3)$$

where  $L_o$  is the gage length of the specimen and  $A_o$  is the cross section area of the specimen.

Ultimate tensile strength (UTS) calculated as the ratio of the maximum load to the initial cross-section area, yield strength (YS) calculated as the ratio of the stress read out at 0.2% of plastic deformation to the initial cross-section area and the total percentage elongation of the specimen gage (TEL) were determined.

## 3. Results and discussion

### 3.1. Hot rolling tests

To determine the phase composition of the investigated steels V and A after reheating before hot rolling tests, volume fractions of ferrite and austenite in the specimens annealed at temperatures between  $800^\circ\text{C}$  and  $1300^\circ\text{C}$  were measured using a quantitative metallographic method. The specimens were held during 10 minutes at a specific temperature and then quenched with cooling rate over  $300^\circ\text{C}\cdot\text{s}^{-1}$ . Volume fraction of all the phases except for ferrite (i.e. martensite, bainite and retained austenite) was considered to be equal to volume fraction of the austenite existing at the annealing temperature. Results of the metallographic analysis are shown in Fig. 4.

Controlled hot rolling tests were carried out using specimens of dimensions  $18\text{ mm} \times 25\text{ mm} \times 110\text{ mm}$  or  $13\text{ mm} \times 90\text{ mm} \times 500\text{ mm}$ , applying one or several passes followed by controlled cooling. Typical microstructures of the specimens of steels V and A prepared for the rolling experiments are shown in Fig. 5.

Parameters of selected tests of controlled hot rolling and final cooling of the specimens of steels V and A carried out in the semi-industrial line LPS are listed in columns 3-12 in Table 2. In Table 2 only variants which produced satisfying mechanical properties are shown.

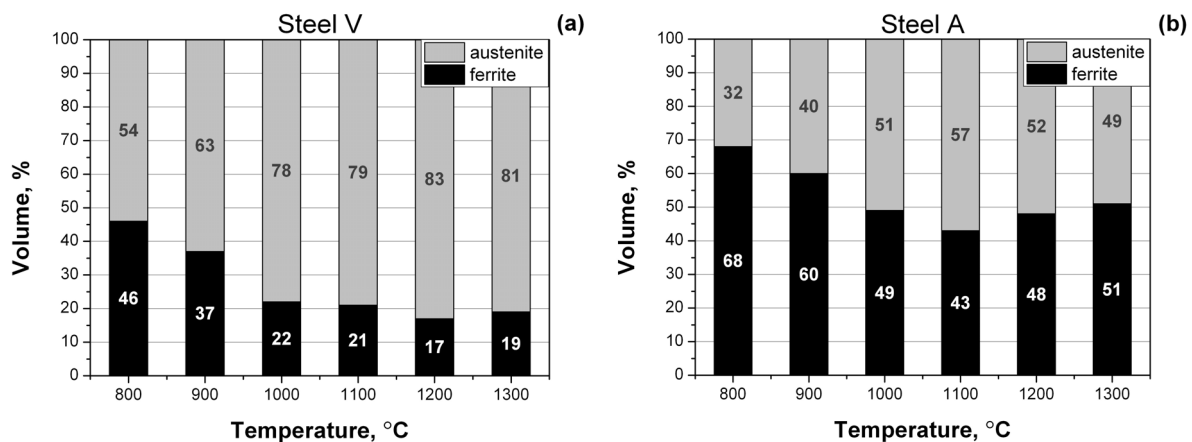


Fig. 4. Volume fractions of austenite and ferrite in steel V (a) and in steel A (b) determined for the specified temperatures after holding during 10 minutes

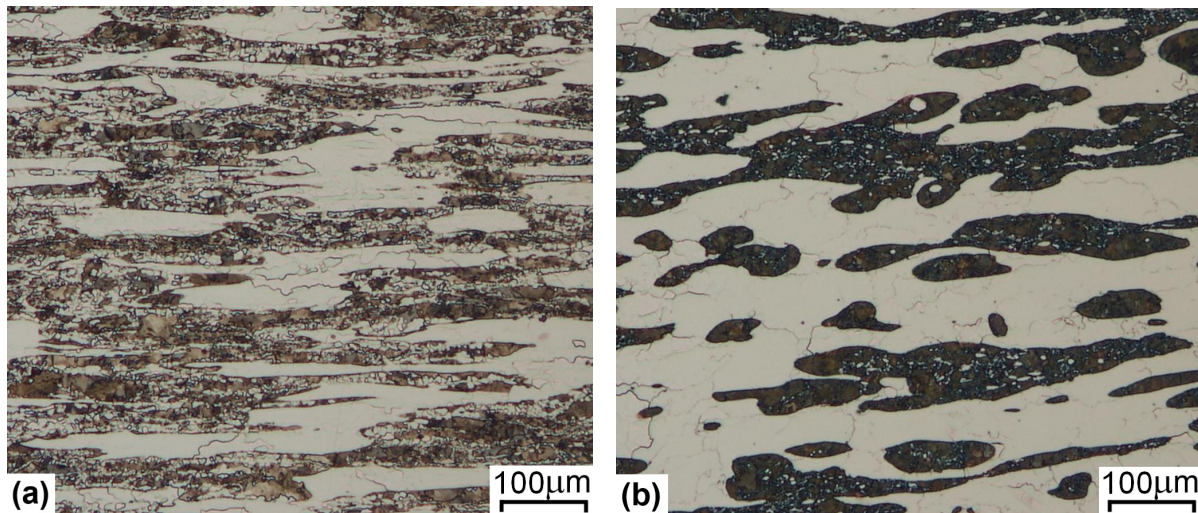


Fig. 5. Optical micrographs showing microstructure of the specimens of steel V (a) and A (b) prepared for controlled hot rolling experiments

Before rolling the specimens were reheated in an electric furnace. They were held at required reheating temperature during about ten minutes. At each pass strain rate was about  $6.0 \text{ s}^{-1}$  and after the final pass the specimens were cooled according to the schemes indicated in column 12: a – at a rate of  $1\text{-}3^\circ\text{C s}^{-1}$  (in the air, cooling rate depended on thickness of the specimen) to a temperature of  $700^\circ\text{C}$  and further cooling at a lower rate of  $0.2\text{-}0.3^\circ\text{C s}^{-1}$ ; b – at a rate of  $1\text{-}3^\circ\text{C s}^{-1}$  (in the air) to room temperature; c – at a rate of  $1\text{-}3^\circ\text{C s}^{-1}$  (in the air) to a temperature of  $450^\circ\text{C}$  and further cooling at a rate of  $0.2\text{-}0.3^\circ\text{C s}^{-1}$ . The reheating temperatures of  $800^\circ\text{C}$ ,  $900^\circ\text{C}$ ,  $1000^\circ\text{C}$  and  $1100^\circ\text{C}$  for steel V and of  $800^\circ\text{C}$ ,  $1000^\circ\text{C}$  and  $1100^\circ\text{C}$  for steel A were used, therefore microstructure of the both investigated steels after reheating consisted of a mixture of two phases  $\alpha$  and  $\gamma$  with proportion of the phases depending on reheating temperature (Fig. 4). In Table 2 deformation temperature and amount of strain in the each applied rolling pass, as well as the actual total strain obtained are given. The amount of strain was calculated using the following formula:

$$\varepsilon_i = \ln(h_{i-1}/h_i) \quad (4)$$

where  $\varepsilon_i$  is the true strain at pass „ $i$ ”,  $h_{i-1}$  is the thickness of the rolled specimen before pass „ $i$ ” and  $h_i$  is the thickness of the rolled specimen after pass „ $i$ ”.

The rolling tests were carried out in temperature range of  $680\text{-}1000^\circ\text{C}$  and the amount of deformation applied in a single pass was from 0.20 to 1.0.

### 3.2. Mechanical properties of thermo-mechanically processed specimens

Results of the uniaxial tensile test of the specimens thermo-mechanically processed according to variants characterised in Table 2, producing attractive mechanical properties, are shown in Table 3. The variants listed in Table 3 represents the simulation of rolling comprising the passes carried out above  $A_1$  temperature (V3 and V4 for steel V, A2 and A4 for steel A) and below  $A_1$  temperature (V1 and V2 for steel V, A1 and A3 for steel A). Generally, the lower deformation temperature and the higher amount of total deformation were applied, the higher strength and lower ductility (plasticity) were obtained. Some of the rolling and cooling schedules produced strength over 1.0 GPa and good ductility level, better than 15% as measured by the total elongation: variant V2 for steel V and variant A3 for steel A. The tensile curves for variants V2 and A3 are shown in Fig. 6. A parabolic shape of the tensile curves results from a high abil-

TABLE 2

Parameters of controlled hot rolling of specimens made of steels V (variants V1 – V4) and of steel A (variants A1 – A4)

Test No	Variant of rolling	Reheating temperature, $^\circ\text{C}$	Temperature ( $^\circ\text{C}$ ) and true strain in passes 1-4								Cooling scheme	Final thickness, mm	Total strain, $\varepsilon_{tot}$
			$T\varepsilon_1$	$\varepsilon_1$	$T\varepsilon_2$	$\varepsilon_2$	$T\varepsilon_3$	$\varepsilon_3$	$T\varepsilon_4$	$\varepsilon_4$			
1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	V1	900	800	0.85	700	0.35	680	0.25	—	—	a	4.2	1.45
2	V2	1000	1000	0.45	900	0.40	800	0.35	700	0.25	a	4.2	1.45
3	V3	1100	1000	0.35	950	0.25	900	0.20	800	0.20	a	4.7	1.00
4	V4	1100	1000	0.35	950	0.25	900	0.20	800	0.20	c	4.7	1.00
5	A1	800	800	0.85	700	0.35	—	—	—	—	a	5.5	1.20
6	A2	1000	1000	0.90	800	0.40	—	—	—	—	b	4.8	1.30
7	A3	1000	1000	0.90	800	0.35	700	0.25	—	—	b	3.9	1.50
8	A4	1100	1100	0.75	1000	0.40	900	0.30	800	0.20	b	3.5	1.65



ity to work hardening of the both steels, especially steel A. The ability to work hardening mainly depends on the morphology of microstructure, what is discussed in the next paragraph.

TABLE 3

Mechanical properties determined in the tensile test for specimens subjected to controlled hot rolling according to variants shown in Table 2

Variant of rolling	YS <sub>0.2</sub> , MPa	UTS, MPa	TEL, %
Steel V			
V1	818	911	14.6
V2	794	1051	16.3
V3	529	777	18.7
V4	513	762	20.2
Steel A			
A1	732	883	18.5
A2	740	941	16.5
A3	679	1058	16.6
A4	660	1025	16.0

### 3.3. Microstructure of thermo-mechanically processed specimens

As described in paragraph 2.2, one of the experimental steel was melted in a vacuum furnace and the other in an open furnace, in order to assess the effect of the melting atmosphere on metallurgical cleanliness of steel, i.e. on the amount and morphology of non – metallic inclusions and – in consequence – on mechanical properties. In a previous work it has been found that main types of inclusions in steel V were separate particles of AlN and MnS, while in steel A complex inclusions composed of AlN and MnS, and aggregates of inclusions containing aluminium oxides, AlN and complex oxy-sulphides containing Al and Mn were present [22]. The air melted steel A contains by about 30% more non – metallic inclusions than the vacuum melted steel V, but still steel A is characterised with good enough metallurgical cleanliness for industrial applications.

Microstructure of the specimens subjected to hot rolling and controlled cooling after the final pass, according to variants described in Table 2, is characterised with laminated (lamellar) morphology. Parameters of the microstructure obtained, particularly thickness of the lamellae, as expected, depended on values of temperature and amount of deformation in particular rolling pass and on number of the applied passes.

The thinnest lamellae in the microstructure of steel V was produced by rolling in the lowest temperatures used, between 680°C and 750°C. The obtained final microstructure contains microlamellae of ferrite deformed and recrystallised or recovered during rolling, with thickness of about 1 µm or less, and lamellae or grains of phases developed during transformation of austenite. Transformation of the deformed austenite during cooling produces various phases – from pearlite to martensite, depending on the rate of cooling and chemical composition of the austenite. For higher rolling temperatures between 800°C and 1000°C and lower amounts of deformation, in the specimens of steel V a layered microstructure developed, with thickness of ferrite lamellae in the range of 2-5 µm. In Fig. 7 microlaminated microstructure of the specimen of steel V subjected to thermo-mechanical processing according to variant V1 (Table 2) is shown and in Fig. 8 distribution of ferrite lamellae thickness ( $t$  in equation (2)) for the same rolling variant is presented, confirming plausibility of developing microlaminated microstructure in steel V by thermo-mechanical rolling.

In steel A the applied variants of thermo-mechanical rolling allowed to obtain a microlaminated microstructure with the minimum ferrite lamellae thickness of about 2 µm. After reheating or deformation in the temperature range of 700-800°C, particles of Fe<sub>3</sub>AlC<sub>0.5</sub> were identified. The final microstructure obtained after applied thermo-mechanical rolling in specimens of steel A – similarly as in steel V – contains lamellae of ferrite deformed and recrystallised or recovered during rolling, and lamellae or grains of phases developed during transformation of austenite during cooling, i.e. pearlite, new ferrite, bainite, martensite and retained austenite. After some of the processing variants amount of retained austenite in the final microstructure is substantial. For

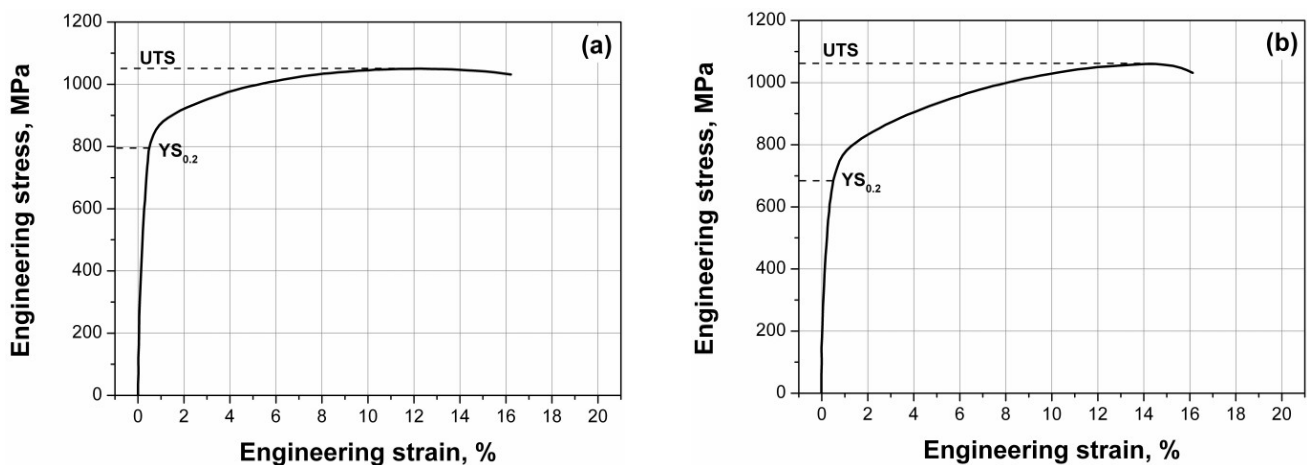


Fig. 6. Engineering stress – engineering strain tensile curves of specimens subjected to thermo-mechanical processing: a – steel V processed according to variant V2 shown in Table 2; b – steel A processed according to variant A3 shown in Table 2

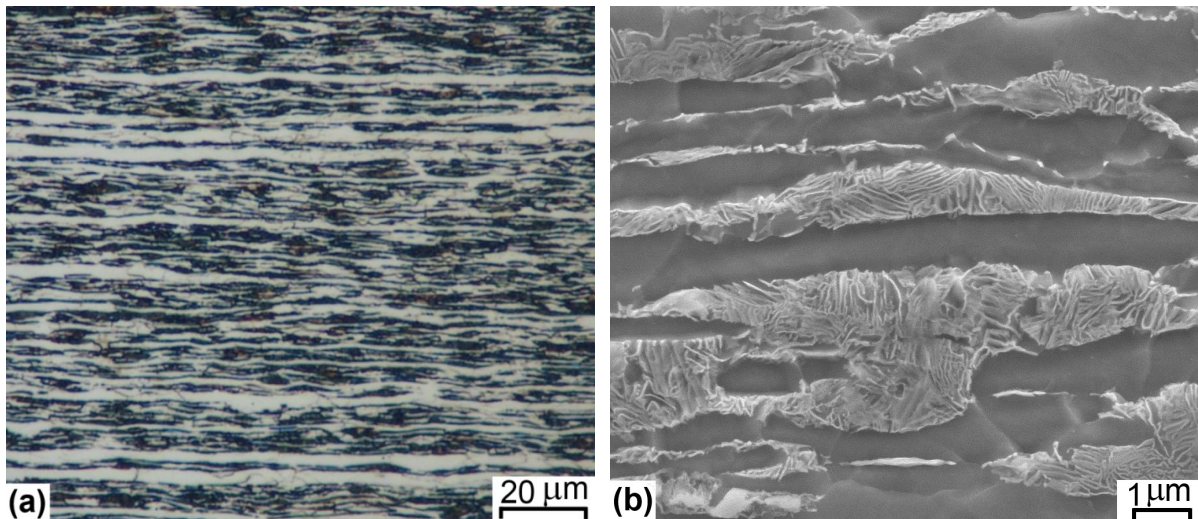


Fig. 7. Optical (a) and SEM (b) micrographs representing microlaminated microstructure of steel V on longitudinal section of the specimen subjected to thermo – mechanical processing according to variant V1 shown in Table 2

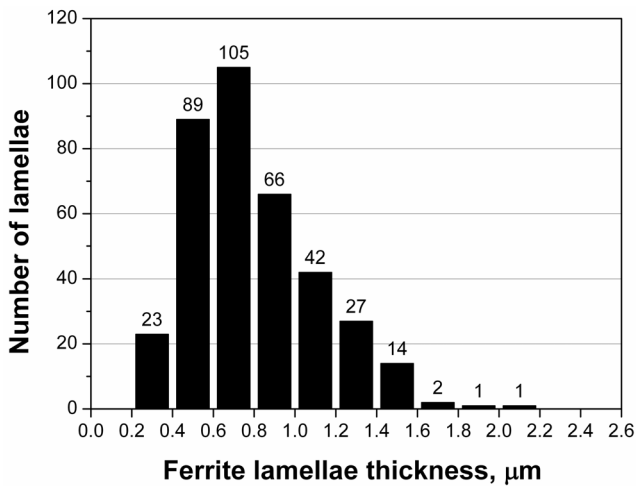


Fig. 8. Distribution of ferrite lamellae thickness (parameter  $t$  in equation (2)) measured for microstructure of steel V on longitudinal section of the specimen subjected to thermo – mechanical processing according to variant V1 shown in Table 2

example, microstructure of the specimen subjected to processing according to variant A3 (shown in Fig. 9) contains 18 vol. % of retained austenite, as it was determined by XRD method.

#### 4. Summary and conclusions

Two compositions of aluminium – alloyed experimental steels containing 0.30% C, 1.7% Mn and 3.0% Al (steel V) and 0.35% C, 2.7% Mn and 5.3% Al (steel A), with the  $\gamma + \alpha$  stability region at temperature range characteristic for hot rolling were designed, to facilitate development of the microlaminated microstructure by thermo-mechanical processing. To assess the effect of melting atmosphere on the metallurgical cleanliness of the steels alloyed with Al, one of the laboratory heat (V) was melted in a vacuum furnace and the other (A) in an open furnace. The air melted steel contained by about 30% more non-metallic inclusions than the vacuum melted steel, but the level of metallurgical cleanliness of the both steels was good enough to obtain attractive mechanical properties.

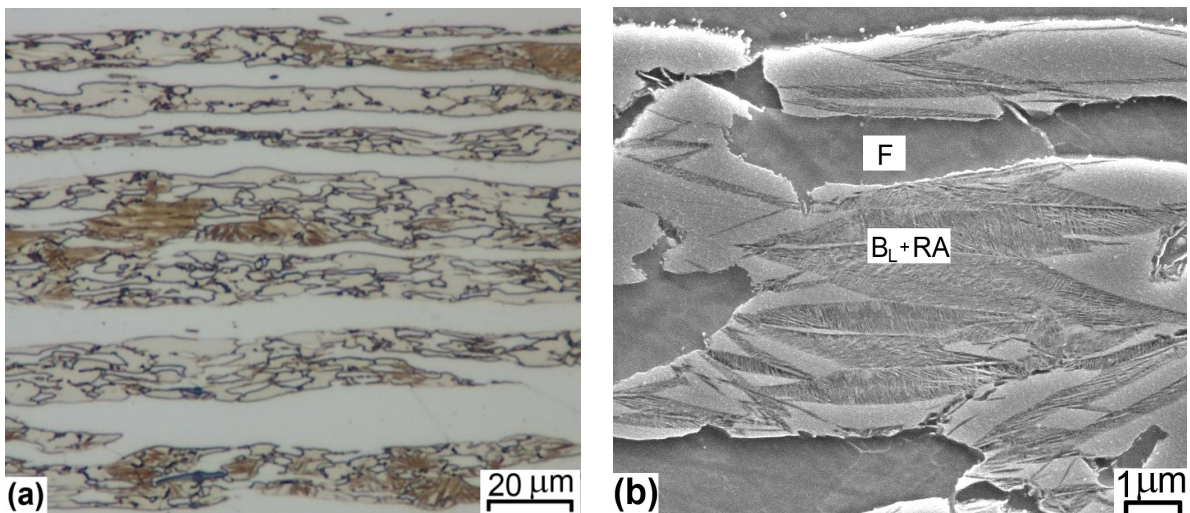


Fig. 9. Optical (a) and SEM (b) micrographs representing microlaminated microstructure of steel A on longitudinal section of the specimen subjected to thermo – mechanical processing according to variant A3 shown in Table 2

The final microstructure of steel V subjected to thermo-mechanical rolling in the  $\gamma + \alpha$  stability region finished between 680°C and 750°C, characterised with the microlaminated morphology composed of lamellae of ferrite with thickness down to 1  $\mu\text{m}$  or less and lamellae or grains of phases developed during transformation of the austenite. Transformation of the hot worked austenite during cooling produced various phases – from pearlite to martensite, depending on the rate of cooling and local chemical composition of the austenite. For higher rolling temperatures (in the range of 800°C-1000°C) and lower amounts of deformation, in the specimens of steel V a layered microstructure developed with thickness of ferrite lamellae in the range of 2-5  $\mu\text{m}$ . In steel A the applied variants of thermo-mechanical rolling allowed to obtain a microlaminated microstructure with the minimum ferrite lamellae thickness of about 2  $\mu\text{m}$ .

Thermo-mechanical processing of the aluminium – alloyed experimental steels with microlaminated microstructure produced by controlled hot rolling allowed to achieve very high tensile strength over 1.0 GPa and total elongation over 15%.

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