THERMOMECHANICAL PROCESSING OF MICRO-ALLOYED STEEL

TERMOMEHANSKA PREDELAVA MIKROLEGIRANEGA JEKLA

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The paper deals with the thermomechanical processing of low-carbon micro-alloyed steel processed by means of a physical simulation on a unique device for simulating a real forging process. The main goal of the experiment was to assess the mechanical and microstructural properties of steel after additions of various amounts of micro-alloying elements. Heats with additions of V, Nb, Ti and Zr were prepared. Different austenitization temperatures and different deformations were applied. Their influence on the microstructure and mechanical properties was evaluated.

Keywords: micro-alloyed steel, thermomechanical processing

Članek obravnava termomehansko predelavo maloogljičnega mikrolegiranega jekla s fizikalno simulacijo na napravi za simulacijo realnega postopka kovanja. Glavni namen eksperimenta je bil ugotoviti mehanske lastnosti in mikrostrukturne značilnosti po dodatku različne količine mikrolegirnih elementov. Pripravljene so bile taline z dodatkom V, Nb, Ti in Zr. Uporabljene so bile različne temperature avstenitizacije in različne deformacije. Ocenjen je bil njihov vpliv na mikrostrukturo in mehanske lastnosti.

Ključne besede: mikrolegirano jeklo, termomehanska predelava

1 INTRODUCTION

High-strength low-alloy (HSLA) steels, or microalloyed steels, are designed to provide better mechanical properties and/or a greater resistance to atmospheric corrosion than conventional carbon steels. They are not considered to be alloy steels in the usual sense because they are designed to have specific mechanical properties rather than a specific chemical composition. In order to realize the full strengthening potential of micro-alloying additions, it is necessary to use a soaking temperature prior to forging that is high enough to dissolve all the vanadium-bearing precipitates¹.

A soaking temperature above 1100 °C is preferred. For the Nb-Ti micro-alloved steel a single-step austenite reheating temperature of 1150 °C provided better austenite conditioning than a higher reheating temperature of 1240 °C.² According to^{3,4} a complete dissolution of carbonitride precipitates based on Nb occurs at 1140 °C in the interval of 1100-1200 °C.5 A further processing after the austenitization should consist of two-phase forging. A higher amount of deformation at an elevated temperature (about 1100 °C) facilitates a dynamic recrystallization. At this stage a critical amount of strain is required for the austenite grain refinement through repeated recrystallization. The second stage should be carried out below 900 °C, i.e., below the recrystallization temperature (TR) in order to make the austenite grain pancaked and to introduce interfacial defects into hot austenite. These interfacial defects in turn lead to an increase in the effective grain-boundary areas, enhancing the nucleation potency of ferrite. Subsequently, the micro-alloying elements come out of the solution with a decrease in the temperature, inhibiting the austenite grain growth by forming micro-alloying carbides or carbonitrides at these interfacial defects. Controlled cooling after the forging leads to the desired microstructure. A subsequent tempering could bring another strengthening due to the precipitation of micro-alloying elements. A tempering temperature of 600 °C and a dwell time of 4 hours are used in this experiment to meet the customer requirements.

2 EXPERIMENT

The first part of the experiment was focused on a numerical simulation in the DEFORM 3D software. It consists of a simulation of the controlled last step of the forging from the 500 mm bar diameter to the 460 (440) mm bar diameter (**Figure 1**). The forging takes place in the interval range of 940–800 °C. Numerical modelling allows accurate computing of a precise deformation and temperature distribution in every location of the real forged product according to choice. One location 40 mm below the bar surface was chosen for a further experiment – a physical simulation. It is the location used for making the samples for mechanical testing.

The second part of the experiment – the physical simulation – was focused on the samples with various micro-alloying additions. A physical simulator is a unique device with the possibilities of temperature and deformation control. The testing cell is set up on a modified MTS servo-hydraulic tensile-test machine.

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Figure 1: Distribution of the temperature for one-step open-die forging – numerical simulation in DEFORM 3D. Diagram in the upper left-hand corner shows the temperature distribution from the surface to the core.

Slika 1: Razporeditev temperature pri enostopenjskem prostem kovanju – numerična simulacija z DEFORM 3D. Diagram levo zgoraj prikazuje razporeditev temperature od površine do sredine.

It allows simulating the conditions of a location of the real piece with respect to the temperature/deformation of the small specimen (**Figure 2**). The specimen shape is similar to the tensile-test sample (**Figure 3**) and it is resistive heated and loaded with tension and compression. A numerical simulation of the temperature distribution in the specimen was also done to make the simulation model more precise (**Figure 4**).

The device parameters are: the maximum force of 250 kN, the heating rate of up to 150 °C/s, the cooling



Figure 2: Specimen in the physical simulator during thermomechanical processing

Slika 2: Vzorec v fizikalnem simulatorju med termomehansko predelavo



Figure 3: Specimen for the physical simulator Slika 3: Vzorec za fizikalni simulator

rate of up to 150 $^{\circ}$ C/s, the maximum frequency of the cyclic loading of 30 Hz and the maximum forming velocity of 600 mm/s.

The cooling rate of the physical simulator allows a simulation of cooling in various environments. It is possible to simulate cooling in the air, quenching in oil and water. Simulation of cooling in the water was chosen to be the best for this task. For a producer, cooling the whole bar in the water is also the simplest way of cooling. Various deformation processes were applied to the specimens in the simulator (see the example on Figure 5). Two samples (base heat B and V1) underwent a physical simulation of forging in the range of 980-840 °C. Other samples were processed in the range of 940-800 °C. Also, the applied deformation was doubled on some specimens and the austenitization temperature prior to the deformation varied from 1000 °C to 1150 °C. All the samples were annealed at 600 °C after the forging. The chemical compositions of the experimental heats with different micro-alloying additions and parameters of the physical simulation are summarized in Table 1. We prepared the samples for tensile and mini-Charpy (notch toughness) tests after the physical simula-



Figure 4: Numerical simulation of temperature distribution in the specimen during physical simulation

Slika 4: Numerična simulacija razporeditve temperature v vzorcu med fizikalno simulacijo

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Figure 5: Example of temperature and deformation parameters of the physical-simulation process

Slika 5: Primer temperature in deformacijskih parametrov pri fizikalni simulaciji

tion and the subsequent heat treatment. The microstructure was observed by means of a light microscope Nikon Epiphot 200 with the quantitative-image-analysis software NIS Elements 3.2. A detailed observation with the EDX measurement was done using a scanning electron microscope JEOL 6380. The last experiment was

 Table 1: Chemical compositions of the analysed heats in mass fractions, w/%

 Tabela 1: Kemijska sestava analiziranih talin v masnih deležih, w/%

focused on the real experimental forging with a hydraulic press. One heat with no micro-alloys and one heat with Ti, Nb, V were subjected to identical forging processes. The aim was to compare the microstructures and mechanical properties of the non-micro-alloyed and microalloyed heats.

3 RESULTS AND DISCUSSION

3.1 Physical simulation

Tensile tests were executed at room temperature. Prior to the testing, the specimen dimensions were measured and the original gauge length needed for determining elongation A was marked on each specimen. Some specimens were tested in the "mini-tensile" shape. This allowed preparing three specimens from one specimen after the physical simulation (**Figure 6**). Impact tests were performed on the instrumented Charpy pendulum with the impact energy of 15 J. Dimensions of the tested samples were 3 mm × 4 mm × 27 mm. The tests were executed at minus 46 °C.

The minimum required values for the mechanical properties were as follows: $R_{p0.2} = 415$ MPa; $R_m = 530$ MPa; A = 20 %; KV_{mini} (-46 °C) = 2 J.

Heat	С	Mn	Si	S	Cr	Ni	Р	Cu	Мо	Ti	V	Nb	Zr	Applied defor- mation	<i>T</i> /°C of defor-mation	<i>T</i> /°C of austenitization
В	0.10	1.47	0.14	0.001	0.15	0.09	0.004	0.11	0.04	_	_	_	_	1	980-840	1000
V1	0.10	1.43	0.09	0.001	0.15	0.09	0.004	0.12	0.04	-	0.060	0.045	_	1	980-840	1000
V2	0.11	1.55	0.15	0.001	0.14	0.09	0.004	0.12	0.04	-	0.090	0.047	_	2	940-800	1000
V3	0.10	1.36	0.1	0.001	0.14	0.09	0.004	0.11	0.04	_	0.170	0.047	_	2	940-800	1150
V4	0.10	1.29	0.1	0.001	0.14	0.09	0.004	0.11	0.04	-	0.130	0.042	_	2	940-800	1000
14	0.11	1.57	0.16	0.001	0.14	0.09	0.004	0.11	0.04	-	0.130	0.052	0.140	2	940-800	1150
15	0.10	1.49	0.13	0.001	0.14	0.09	0.004	0.11	0.04	_	0.130	0.050	0.195	2	940-800	1000
19	0.10	1.47	0.14	0.001	0.15	0.09	0.004	0.11	0.04	0.17	0.110	0.045	-	1	940-800	1000
23	0.10	1.40	0.12	0.001	0.15	0.09	0.004	0.11	0.04	0.20	0.200	0.050	-	2	940-800	1100
26	0.11	1.51	0.18	0.001	0.14	0.09	0.044	0.11	0.04	_	-	-	-	2	940-800	1000
29	0.11	1.43	0.12	0.001	0.14	0.91	0.004	0.11	0.04	_	-	_	_	2	940-800	1000
33	0.07	0.50	0.03	0.001	0.11	0.09	0.004	0.11	0.48	_	-	_	_	2	940-800	1000

 Table 2: Results of mechanical testing of thermomechanically processed specimens (with the physical simulator)

 Tabela 2: Rezultati mehanskih preizkusov na termomehansko predelanih vzorcih (na fizikalnem simulatorju)

	Specimen		$R_{p0.2}/MPa$	<i>R</i> _m /MPa	KV _{mini (-46 °C)} /J	$T_{\text{aust}}/^{\circ}\text{C}$	Def	$T_{\rm def}/^{\circ}{\rm C}$
B (no microalloys)			365	606	6.9	1000	1	980-840
V. Nb	V1	415	543	6.2	1000	1	980-840	
	V Nh	V2	480	624	9.3	1000	2	940-800
	V3	600	732	2.2	1150	2	940-800	
		V4	481	593	8.0	1000	2	940-800
V. Nb. Zi	V Nh Zr	Zr1	474	710	0.6	1150	2	940-800
	v. NO. Zr	Zr2	482	625	3.3	1000	2	940-800
	T: V NI-	Ti1	422	547	10.0	1000	1	940-800
	11. V. IND	Ti2	622	699	1.0	1100	2	940-800
	Р	Р	412	486	9.5	1000	2	940-800
	Ni Ni		459	533	2.7	1000	2	940-800
	Мо	Mo	349	443	8.4	1000	2	940-800

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Figure 6: Mini-tensile test samples Slika 6: Natezni minipreizkušanci

The highest values of the mechanical properties (*YS* and *TS*) were reached for samples V3, Zr1 and Ti2 (**Table 2**). They were austenitized before the thermomechanical processing on the simulator at 1100 °C and 1150 °C, respectively. The microstructures of all the heats consist of fine-grained ferrite, tempered bainite/martensite and pearlite. The reason for higher yield and tensile strengths after a decrease in the deformation temperature is a decrease in the grain size due to a higher deformation ratio (the change in the grain size from 10.5 to 11.5) (**Figure 7**).

A higher austenitization temperature (1100-1150 °C) allowed a complete dissolution of carbonitrides before the thermomechanical processing and thus led to an increase in the tensile strength up to 730 MPa. But the notch toughness of these specimens was low. The lowest value of notch toughness in the case of heat Zr1 was caused by coarse undissolved ZrCN particles, which were also visible on the fracture surfaces and in the microstructures of the mini Charpy samples (**Figure 8**).



Figure 7: Decreased grain size due to a higher deformation: a) specimen Ti1, b) specimen V2

Slika 7: Zmanjšanje velikosti zrn z večanjem deformacije: a) vzorec Ti1, b) vzorec V2



Figure 8: ZrCN particles on the fracture surface of heat 15 (SEM) **Slika 8:** Delci ZrCN na površini preloma taline 15 (SEM)

Vanadium-based carbides/carbonitrides were observed in the microstructure of all the samples marked as Vx, Zrx and Tix. The strengthening mechanism did not occur on specimens P, Ni and Mo, which were not micro-alloyed at all. The lowest values of mechanical properties were reached for the heat marked as Mo due to the reduction of manganese and carbon amounts. The main mechanism for increasing the yield and tensile strengths of the micro-alloyed specimens consisted of grain refinement (due to a decrease in the forming temperature) and precipitation strengthening. The grain size



Figure 9: Three-dimensional model of an ingot and a numerical simulation of forging in DEFORM 3D

Slika 9: Tridimenzijski model ingota in numerična simulacija kovanja z DEFORM 3D

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Figure 10: Microstructure of the specimens after conventional opendie forging: a) no micro-alloys, b) micro-alloyed with Ti, V and Nb **Slika 10:** Mikrostruktura vzorcev po navadnem prostem kovanju: a) brez mikrolegiranja, b) mikrolegirano s Ti, V in Nb

 Table 3: Mechanical properties of forged ingots (without micro-alloys and micro-alloyed with Ti, V and Nb)

Tabela 3: Mehanske lastnosti kovanih ingotov (brez mikrolegiranja in mikrolegirano s Ti, V in Nb)

Specimen	$R_{p0.2}/MPa$	<i>R</i> _m / MPa	A5/ %	<i>KV</i> _(−46 °C) / J	Grain size/G
Base heat	326	480	34	179	9.5
Heat with Ti, Nb and V	433	534	32	155	11.0

of the specimen before thermomechanical processing was 6.5 and it decreased to the average value of 11.0 (ASTM E 112) after thermomechanical processing.

3.2 Forging experiment

The forging on the hydraulic press was performed according to the numerical-simulation calculation of deformation in the location of the real forged bar. The forging model and the specimen after forging are shown in **Figure 9**.

The forging of experimental ingots was done by means of a hydraulic press, Zeulenroda (PYE 40). The ingots were put in a Heraus atmospheric furnace and heated up to 1150 °C. Then they were being forged to the final dimensions for about 1 min and 10 s. The final temperature on the ingot surfaces was measured with a thermocouple, and it reached about 750 °C. The ingots were then cooled on air with no subsequent annealing. The microstructure of the ingots after forging shows substantial differences, especially in the grain size (**Figure 10**).

The yield and tensile strengths of an ingot with micro-alloys are considerably higher and the grain size is also different (**Table 3**). The yield strength increased by more than 100 MPa thanks to micro-alloying strengthening.

4 CONCLUSION

A new technological process of forging steel for the oil industry was designed by means of a numerical simulation in the DEFORM 3D software. This process was applied to the samples during the experiment with a physical simulation. The samples were made from various heats micro-alloyed with a combination of Ti, Nb, V and Zr.

The main mechanism for increasing the values of *YS* and *TS* consists of grain refinement and precipitation strengthening. A grain refinement was achieved thanks to a decrease in the final forging temperature below the temperature of austenite recrystallization. Also, good notch toughness at lowered temperatures was maintained. The best values of the yield strength and tensile strength were reached by micro-alloying with vanadium and niobium and, especially, with the samples preheated before the thermomechanical processing at higher austenitization temperatures. This process led to a complete dissolution of all the micro-alloying additions during austenitization; therefore, good yield and tensile strengths were reached after thermomechanical processing.

The forging experiment with real forging on the hydraulic press confirms the efficiency of the designed technological process. In the case of micro-alloying, the yield strength was by about 25 % higher than for the heat with no micro-alloys. The grain refinement of the micro-structure is also clearly visible.

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