DUAL PHASE FERRITE-MARTENSITIC STEEL MICRO-ALLOYED WITH V-Nb

For low-carbon V-Nb micro-alloyed steel, dual phase ferrite-martensitic microstructures were prepared with a variable martensite fraction, which ranged from V_m 20 to 88%. By measuring the temperature in the sample centre, the parameters of intercritical annealing and the relationship between V_m and the intercritical annealing temperature 720 °C $\leq t \leq$ 850 °C were determined. By analysing the microstructure, the strength and plastic properties of this dual phase steel, the preparation method of dual phase ferrite-martensitic steels was tested. Dual phase ferrite-martensitic microstructures at V_m above 53% formed continuous martensite areas. A change of the microstructure type as compared with the state with V_m < 53% resulted in a change of the increase of R_{p0_2} and R_m. With growing V_m the strength properties increased and the plastic properties decreased. Fractures after uniaxial loading had a ductile pit morphology. On extraction carbon replicas, changes of precipitation of fine disperse precipitates were studied on the normalized state and the intercritically annealed state.

Key words: *dual phase steel, intercritical annealing, mechanical properties, microstructure, heating, cooling*

Dvofazni feritno-martenzitni čelik mikrolegiran s V-Nb. Za nisko-uglični V-Nb čelik pripremljene su dvofazne mikrostrukture s promjenljivom martenzitnom frakcijom u rasponu od V_m 20 do 80 %. Mjerenjem temperature u centru uzorka određeni su parametri međukritičnog žarenja i odnosa između V_m i međukritične temperature žarenja 720 °C $\leq t \leq$ 850 °C. Analizom mikrostrukture ispitivane su čvrstoća i plastična svojstva ovog dvofaznog čelika. Dvofazne feritno-martenzitne mikrostrukture pri V_m iznad 53 % obrazovale su trajna martenzitna područja. Možebitna promjena u vrsti mikrostrukture u usporedbi sa stanjem V_m < 53 % dovodi do promjene u povećanju R_{p0_2} i R_m. S povećanjem V_m povećavaju se i svojstva čvrstoće, a plastična svojstva se smanjuju. Lomovi nakon jednoosnog opterećivanja imali su žilavi morfološki prelom. Nakon izdvajanja ugljičnih replika, proučavale su se promjene sitno disperziranih percipitata u normaliziranom stanju i međukritičnom žarenom stanju.

Ključne riječi: *dvofazni čelik, izmeđukritično žarenje, mehanička svojstva, mikrostruktura, grijanje, hlađenje*

INTRODUCTION

Low-carbon multi phase (MP) steels developed in recent decades show excellent mechanical properties, a high strain hardening exponent, good formability, exclusive surface treatment and they are comparable with high-strength low-alloyed steels (HSLA). Pearlitic HSLA steels developed as micro-alloyed steels with a variable alloying degree show improved strength properties when compared with carbon steels, but worse plasticity and formability. Dual phase (DP) steels have a ferrite-martensitic structure, a continuous yield point, a higher tensile strength, a higher strain hardening exponent and better plasticity and formability [1 - 4]. The research was aimed at studying the influence of the chemical composition on the microstructure, the strength and plastic properties and the austenitisation kinetics [5 - 10].

The influence of the volume fraction of martensite, as a hard phase, was investigated by a number of authors [1, 3, 4, 11]. The growth of the volume fraction of martensite results in increased yield point, tensile strength and impact strength of DP steels. According to [12 - 14], this effect was only observed for the volume fraction of martensite $V_m \sim 55\%$. At higher V_m values, the authors observed a decrease of strength properties, which they explain by a decreased carbon concentration in martensite.

In the work, the process of formation of dual phase structures using intercritical annealing of low-carbon micro-alloyed V-Nb steel was studied by measuring the temperature in the sample centre. On experimentally prepared samples their strength and plastic properties were studied.

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EXPERIMENT

For experimental work, commercial steel sheet of Slovak provenience was used, which was made of low-carbon V-Nb micro-alloyed steel with the thickness of 2,15 mm. The chemical element analysis of material is shown in Table 1.

Table 1. **Chemical element analysis (% by weight ×10²)**
Tablica 1. **Analiza kemijskog elementa (% tež. ×10²)**

C	Mn	Si	P	S	Al	Cu	Zr
8,8	131	1	1,4	0,8	4,4	4,4	0,1
Ni	Cr	As	Ti	V	Nb	Mo	
1,8	2,3	0,3	0,1	3,3	5,1	0,3	

Samples taken in the rolling direction with the dimensions 300 × 30 mm were normalized at 950 °C, with dwelling at the normalizing temperature for 30 minutes, and freely cooled by the air. Using this process, the initial microstructure was prepared, which was further experimentally processed. For each of the heat treatment processes, a through-flow protective N_2 atmosphere was used. Ferrite-martensitic microstructures with V_m 20 % (M1), 41 % (M2), 53 % (M3), 76 % (M4) and 88 % (M5) were prepared by intercritical annealing at 720 °C, 760 °C, 790 °C, 820 °C and 850 °C. The samples were heated in a furnace with a constant temperature. The Nabertherm furnace was used as a heat source. The qualitative analysis of the thermal field in the samples with the used experimental arrangement was made according to [15 - 18]. The heating regime was designed in such a way that the heating period of material in the steel sheet centre could exactly be known. The temperature was sensed by a thermocouple Pt-PtRh10. In each of the regimes of intercritical annealing at 720 - 850 °C and 950 °C, the temperature in the sheet centre reached the required intercritical annealing temperature, or the normalizing temperature, 15 minutes after putting the samples into the heated up furnace. The process of heating to the intercritical annealing temperature was designed in such a way that the total period of heating to the intercritical annealing temperature was 25 minutes; then the samples were cooled in various cooling media (air, oil, water, 10 % NaOH water solution); the temperature of the medium was 22 - 24 °C. The cooling rate necessary for obtaining the ferrite-martensitic structure was determined experimentally. The microstructure was observed using light microscopy. By cooling in the air, oil and water, mixed microstructures of ferrite, pearlite, bainite and martensite were formed. Dual phase ferrite-martensitic microstructures were formed by cooling in 10 % NaOH water solution. Samples with the ferrite-martensitic microstructure prepared by etching in 2 % Nital were analysed using light microscopy. Samples for scanning electron microscopy were prepared by deep etch-
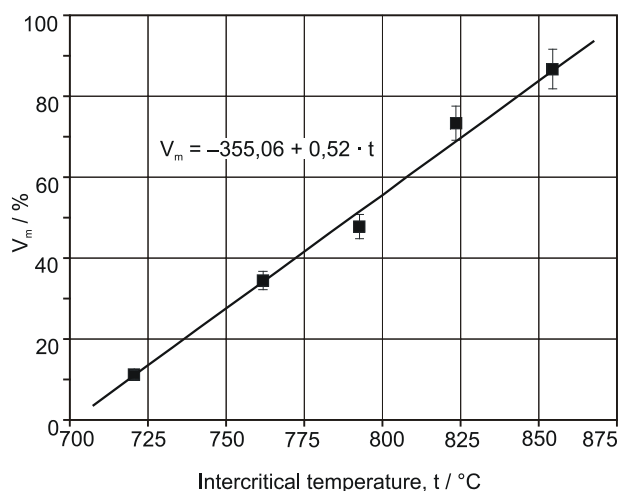

Figure 1. **Volume fraction of martensite as a function of intercritical temperature for DP Steels**
Slika 1. **Frakciski volumen martenzita kao funkcije izmeđukritične temperature za dvofazne čelike**

ing in Nital. Using stereometric metallography [19, 20], the fraction of structural components - V_m was determined, as well as the mean ferrite grain size. For each of the groups M1 - M5 and (NZ), static tensile tests were made under the following conditions: test temperature 23 °C, initial specimen length $l_0 = 80$ mm, loading rate 0,5 mm/min. The tests were made using the testing machine TiraTest 2300.

RESULTS AND DISCUSSION

Using intercritical annealing, dual phase ferrite-martensitic microstructures were prepared with a variable area fraction of martensite. The NZ state represented the initial ferrite-martensitic microstructure for the subsequent inter-
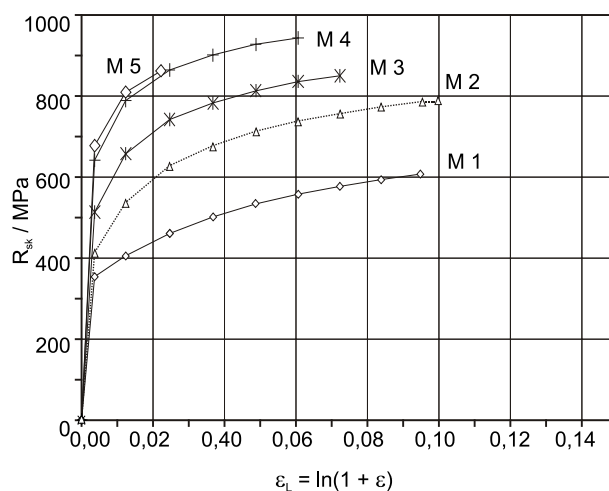

Figure 2. **Effect of the volume fraction of martensite on the true tensile stress-strain diagram**
Slika 2. **Učin frakcijskog volumena martenzita na dijagram naprezanje-istezanje**

critical annealing process. The relationship between V_m and the intercritical annealing temperature 720 °C $\leq t \leq$ 850 °C, within 5% error of experimental determination of V_m, was approximated using the following linear function:

$$V_m = -355,06 + 0,52 \cdot t \tag{1}$$

The type of the function (1) is in accordance with [13]. Using a static tensile test, the following values were determined: conventional yield point R_{p0_2} or R_{eH} and R_{eL} (for the normalized state), tensile strength R_m, elongation A_g, homogeneous plastic deformation ε_r and strain hardening exponent n. From the conventional stress-strain diagram, a correlation between the actual stress R_{sk} and the actual strain ε_L was determined (Figure 2.):

$$R_{sk} = k \cdot \varepsilon_L^n \tag{2}$$

Dual phase ferrite-martensitic steels are mainly intended for cold forming. The combination of strength and elongation is a crucial parameter of these steels. The mechanical properties are mainly influenced by the presence of martensite. The volume change connected with the martensitic transformation influences R_{p02}, R_{p02}/R_m, the continuous yield point and the strain hardening exponent n (Figure 3.). A
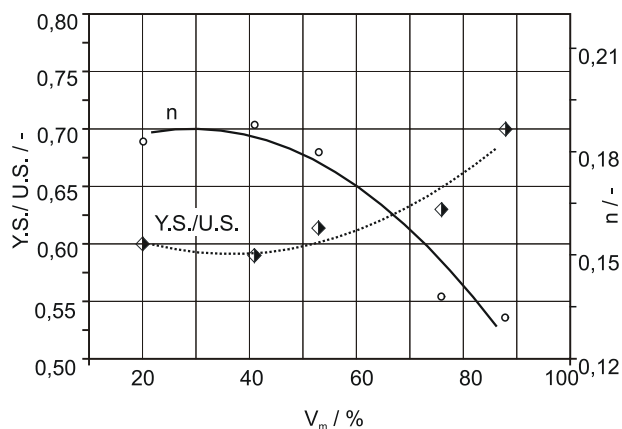
Figure 3. **Variation of strain hardening exponent (n) and ratio R_{p02}/R_m as a function of volume fraction of martensite for DP Steels**
Slika 3. **Varijacije eksponenta (n) očvršćivanja istezanjem i odnosa R_{p02}/R_m kao funkcije frakcijskog volumena martenzita za dvofazne čelike**

crucial factor that influences mechanical properties is V_m. The other factors, such as the C content in martensite, the martensite grain size, etc. are of a less importance [8]. With an increasing V_m, the strength increases and the uniform elongation decreases (Figure 4.).

Dual phase ferrite-martensitic microstructures create continuous areas at V_m above 53% (Figure 5.) and,

according to the classification in [21], a change in the microstructure type takes place when compared with the state with $V_m <$ 53 %. This effect is also shown in a change of growth of R_{p0_2} and R_m with growing V_m (Figure 4.) and is in accordance with [11, 12].
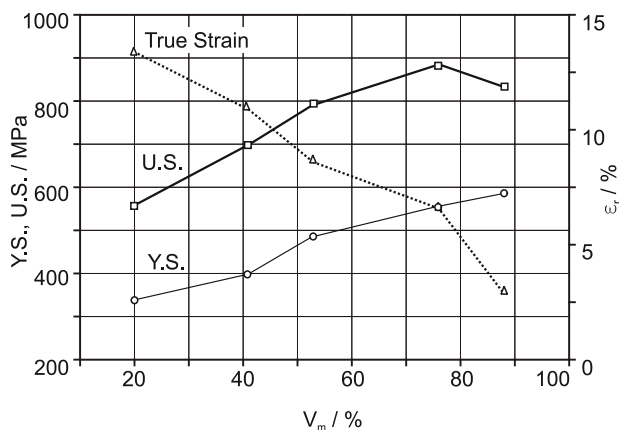
Figure 4. **Strength and percentage elongation as a function of volume fraction of martensite for DP steels**
Slika 4. **Čvrstoća i postotak izduživanja kao funkcije frakcijskog volumena martenzita za dvofazne čelike**

In connection with the volume change due to martensitic transformation, the surrounding ferritic matrix is deformed, dislocation sources are activated and the density of free dislocations increases. All these phenomena in mutual correlation cause a decrease of R_{p0_2} and the suppression of Lüders deformation at the yield point. This results in a non-linear relationship between R_{p0_2} and V_m. At a constant
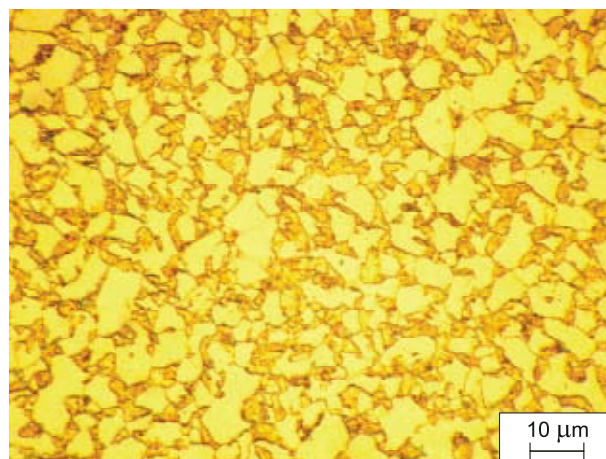
Figure 5. **Group M3, V_m = 53%**
Slika 5. **Skupina M3, V_m = 53%**

value of V_m, an increase of the size of martensite grains and simultaneously a decrease of their number has a significantly negative effect on the plastic properties [12, 13]. This phenomenon was observed in M5 (Figures 4., 6.). The plastic
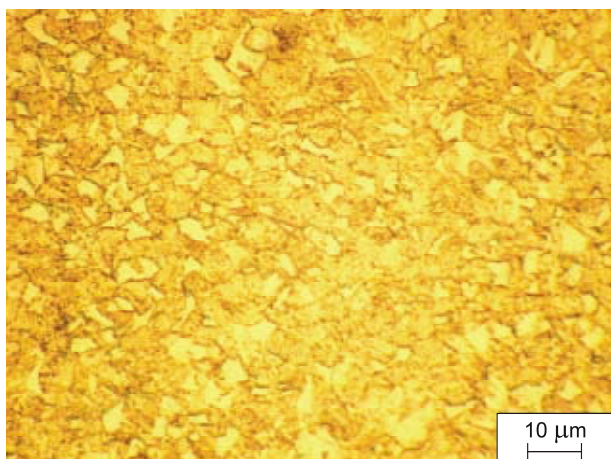
Figure 6. **Group M3, $V_m = 88\%$**
Slika 6. **Skupina M3, $V_m = 88\%$**

properties depend on the condition of the ferritic matrix. If the applied cooling rate is higher than 250 °C/s, then the intercritical annealing temperature directly determines the ferrite and martensite fractions. In these cases the strength properties significantly grow with the intercritical annealing temperature, while the plastic properties decrease (Figures 3., 4.). An accompanying effect is the oversaturation of ferrite with interstitial C. An increase of the cooling rate results in an increase of strength and a decrease of plasticity. The presence of martensite in dual phase steel influences strength properties, particularly the yield point.

During loading, the ferritic matrix is gradually filled with dislocations; their local accumulation ("pille up")
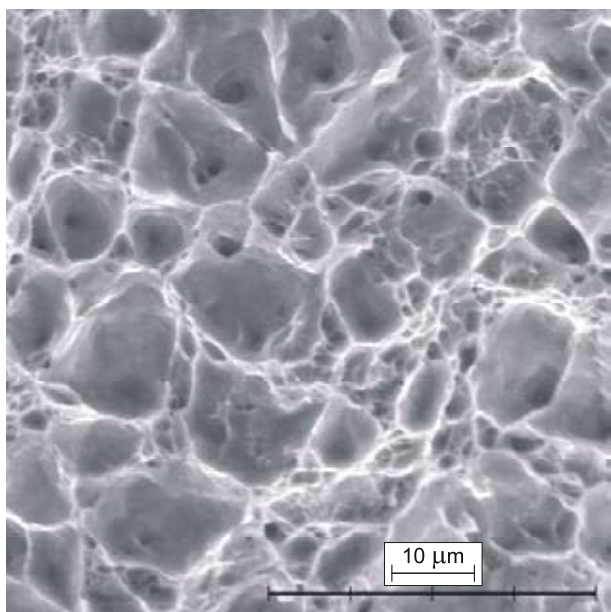

Figure 7. **Group M5, $V_m = 53\%$**
Slika 7. **Skupina M5, $V_m = 53\%$**

causes local failure or decohesion of martensite and ferrite even before the plastic stability point. At low plastic deformation degrees, residual austenite transformed into martensite [22]. The fracture surfaces of all the states from M1 to M5 after uniaxial tension loading had ductile pit morphology, with statistically distributed pits of greater sizes and depths. With growing V_m, the occurrence of deep and greater pits was more frequent (Figures 7., 8.).

The contributions of hardening mechanisms in the martensitic structure according to [1 - 9] include the solid solution substitution element hardening, the precipitation hardening, the primary austenitic grain size hardening and the martensite morphology hardening. The dominant hardening effect of martensite in dual phase steels is the carbon concentration in martensite. It is relatively difficult to formulate regression equations for the contributions of individual hardening mechanisms in martensite, as it is possible for polygonal ferrite, since it is impossible to separate individual hardening mechanisms in martensite [23].
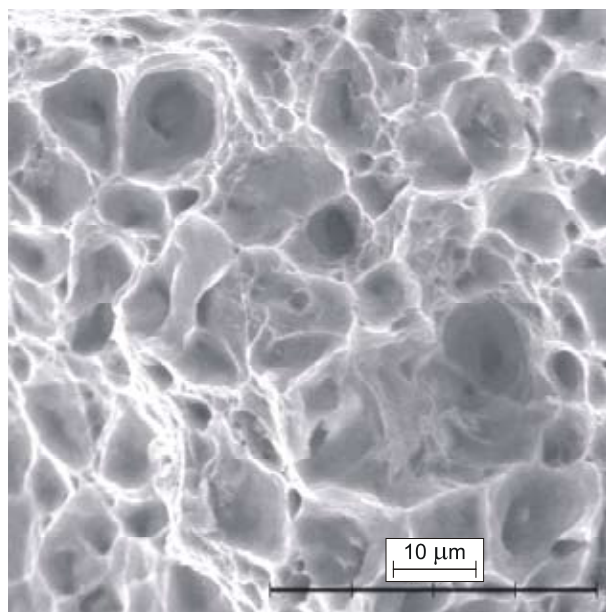

Figure 8. **Group M5, $V_m = 88\%$**
Slika 8. **Skupina M5, $V_m = 53\%$**

Ferrite is the main structural component of dual phase ferrite - martensitic steels; its yield point value R_e^F can be expressed as the sum of individual hardening contributions [24] using the following equation:

$$R_e^F = R_{PN} + R_{IN} + k_{Si} \cdot x_{Si} + k_{Mn} \cdot x_{Mn} + $$
$$+ k_y \cdot \frac{1}{\sqrt{d}} + k_{SZ} \cdot \frac{1}{d_L} + k_p^R \cdot \frac{1}{\lambda^2}$$

$$(3)$$

where:

d - the ferritic grain size,

x_{Mn}, x_{Si} - are the concentrations of dissolved substitution elements Mn and Si,

k_y - the characteristic of a barrier effect of grain boundaries against dislocation movement,

k_{SZ} - the shear stress rate necessary in order to unblock dislocations accumulated at the subgrain boundary,

d_L - the subgrain boundary or the cross linear slat size,

λ - the medium inter-particle distance of precipitates.

According to [22], the constant $k_p^R = 76{,}8 \cdot 10^{-8}$ N and it is approximately equal to the interaction force of an edge
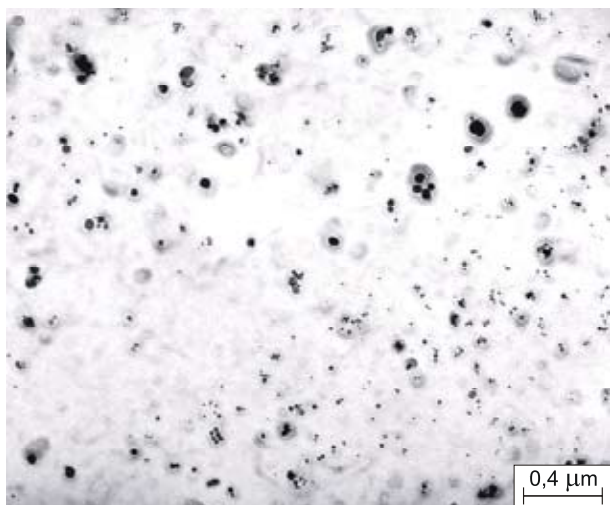

Figure 9.   **Group M1**
Slika 9.   **Skupina M1**

dislocation and the elastic field of a particle. Figures 9. and 10. show extraction carbon replicas of selected states. The
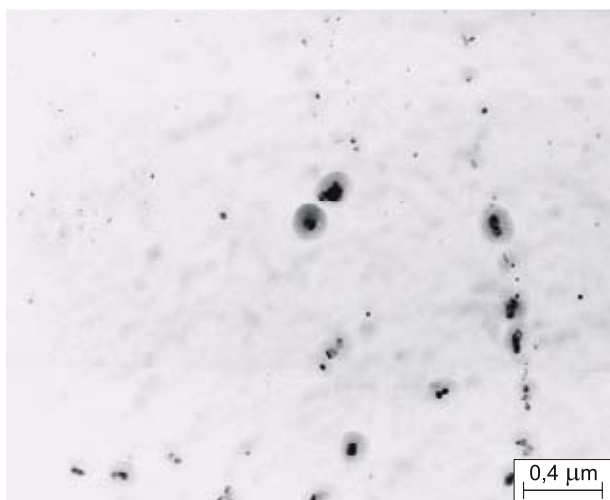

Figure 10. **Group M2**
Slika 10.   **Skupina M2**

photographs show fine dispersion particles of precipitates after normalizing and intercritical annealing at 720 °C $\leq t$ $\leq$ 850 °C (Figures 9., 11.). At higher intercritical annealing temperatures, fine precipitates partially dissolved and
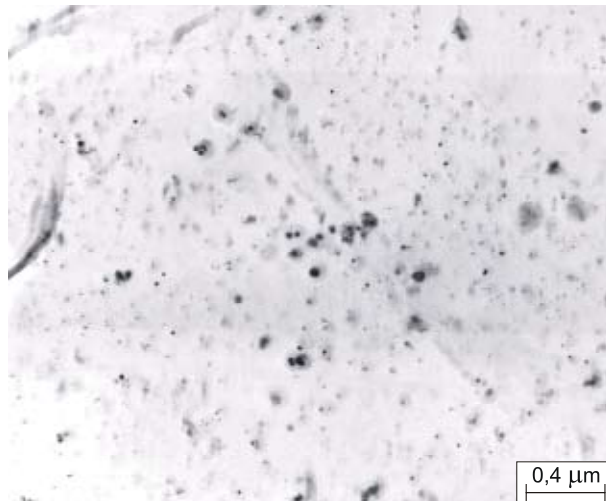

Figure 11. **Group NZ**
Slika 11.   **Skupina NZ**

their re-precipitation was suppressed by quick cooling from the intercritical annealing temperature (Figure 10.). Changes in precipitation of fine dispersion precipitates when compared with the normalized state were tested by annealing at 820 °C for 30 minutes and by cooling from this temperature at the cooling rate of 1 °C/min to 100 °C (Figure 12.). This process resulted in an increased density
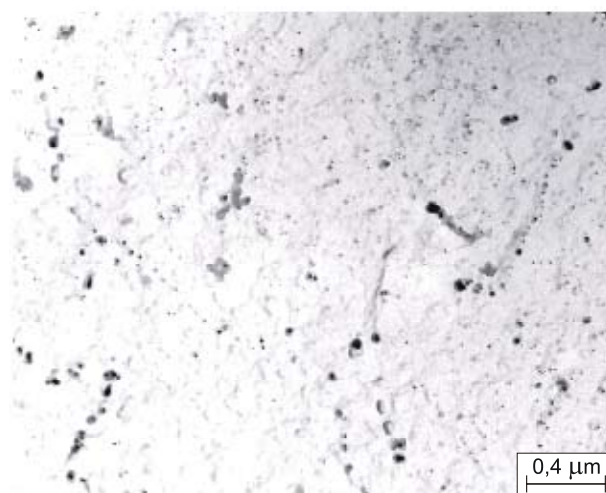

Figure 12. **Annealing: 820 °C/30min/ cooling 1 °C/min**
Slika 12.   **Žarenje: 820 °C/30min/ hlađenje 1 °C/min**

of fine dispersion precipitates when compared with the normalized state. Coarse particles of precipitates (Figures 9., 10.) are related to the annealing temperature.

CONCLUSION

In the work, the ferrite-martensitic microstructure of low-carbon micro-alloyed V-Nb steel was studied. Heating conditions and cooling rates at which the ferrite-martensitic microstructure was experimentally verified. Using a method of measuring the temperature in the sample centre, dual phase ferrite-martensitic microstructures were prepared with the martensite volume fractions of 20, 41, 53, 76 and 88 %, whose basic mechanical properties were determined. Their microstructure was analysed using the light, scanning and transmission electron microscopy. Within a 5% measuring error in the experimental determination of V_m, the change of V_m as a function of the intercritical annealing temperature was approximated using the following linear function (this only applies to the given chemical composition, size and shape of the experimental samples):

$$V_m = -355,06 + 0,52 \cdot t$$

Experimentally prepared states M1 to M5 had typical characteristics of dual phase ferrite-martensitic steels.

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