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Effect of thermal regime on the microstructure and microhardness of steels microalloyed with Nb and V

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Microalloying of high strength steels with elements, such as Nb, Ti and V, having high affinity to carbon and nitrogen, contribute to a significant improvement of its mechanical properties. Microalloying of steel using these elements allows the obtainment of mechanical strength and good toughness at low cost as compared with conventional alloyed steels. If the metallurgical processes for obtaining (steelmaking and refining) and thermo-mechanical processing (rolling, forging etc.) are appropriate, precipitates may act within the material by blocking the movement of grain boundaries, which allows the increasing of toughness and thermal stability characteristics at high temperatures. To study the effect of thermal regime on the microstructure and microhardness of steels microalloyed with niobium and vanadium were made several charges, with different chemical composition, using two types of metallurgical units: a Balzers type vacuum induction furnace – VIM and vacuum arc remelting equipment – VAR. In order to estimate the effect that the applied thermal regime have on the steel microstructure and hardness characteristics, samples of steel microalloyed with Nb and V were normalized at 950…970°C for 20 minutes, after which they were cooled in air. Microstructure analysis and microhardness measurements were performed. It was found that after applying the heat treatment, for most analyzed steel samples, the microhardness has decreased with different values, depending on the chemical composition.

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1. Introduction

Precipitation phenomena into the microalloyed high strength steels must be well controlled to avoid the occurrence of the phenomenon of over-aging or the embrittlement of the metal matrix, due to the increase in the size precipitates, accumulation on grain boundaries or the reduction of the dispersion [1, 10]. Such phenomena can cause secondary effects that lead to the decreasing of some strength characteristics (yield strength or tensile strength, hardness), which requires the strict control of the following parameters:

- *TRA* - austenite recrystallization temperature (temperature below which do not occurs the recrystallization of austenite grains plastically deformed, as a result of the presence of some fine precipitates);
- *TCA* - the temperature of the grain growth of austenite (the temperature above which the fine precipitates undissolved in austenite cannot block of grain growth).

Formation of precipitates is controlled by the chemical composition of the steel and its thermo-mechanical history. For development of the model that can predict the behaviour precipitates, must be studied the types of equilibrium phases, the general compositional domains and specific quantities of items that forms precipitate to a certain temperature. The maximum amount of precipitates is given by the solubility limit of the element in the matrix, the calculation base being the level of saturation and the driving force for their growth.

Contribution of different mechanisms to enhance the yield stress of the steels can be expressed by the following relationship [2]:

$$\sigma_y = \sigma_o + \sigma_{ss} + k_d d^{-1/2} + \sigma_d + \sigma_{pp}, \tag{1}$$

were: σ_o is the lattice friction stress;
- σ_{ss} is the contribution from the elements that are present into solid solution;
- k_d is a constant related to the dislocation density;
- d is the average grain size of ferrite;
- σ_d is the contribution of the dislocations;
- σ_{pp} is the precipitation strengthening term.

The contribution of the Nb to the yield stress, $\Delta\sigma_y^{Nb}$, can be estimated using different experimental values that express the solute and grain size contribution. As a rule, it can be stated that by decreasing the rolling temperature in the range of 750…600°C can be obtained increases of the Nb contribution to the yield stress.

Precipitation strengthening depends on the particles size and the inter-particle spacing of the precipitates and can be expressed through Ashby-Orowan's equation [2, 3]:

$$\sigma_{pp}(MPa) = k\frac{\sqrt{f_v}}{r} ln\frac{r}{6,125x10^{-4}} \tag{2}$$

were fv is the volume fraction of the particles, r is the average precipitate diameter in μm and k is a constant. In the Ashby-Orowan's equation, k adopts a value of 10,8MPa μm, but Buessler et all. [2] reported the value of 17MPa μm for the case of Nb precipitation in ferrite.

Niobium is used as an alloying element for grain refinement but also can promote the precipitation strengthening effect. This determines the delay in austenite recrystallization during hot plastic deformation, due to the diffusion from solid solution and producing internal tension into lattice by formation of carbo-nitrides precipitates. In the structural steels with low carbon content, niobium increases the yield strength with about 150 N/mm2, and the tensile strength with more than 100 N/mm^2. It is used also as a stabilizer of the structure (formation of stable compounds) to ensure a good behaviour at welding, in particular for the structure of heat affected zone [4 - 8].

Vanadium is an element used mainly for strengthening by precipitation of micro-alloyed steels. Strengthening obtained by forming the vanadium precipitates depends on their size. For each 0.1 wt% V added, can be obtained an increase of 50-60 MPa of tensile strength. It forms stable nitrides and carbides up to 1150 ^{o}C. Vanadium carbo-nitride precipitates promotes both finishing and stabilizing of ferrite grains. Vanadium has a high affinity for nitrogen and carbon that is a cheap alloying element in steel. The large number of precipitates of type V(C, N) allows the steel obtaining with lower economic efforts.

2. Experimental procedure

2.1. Obtainment of microalloyed steels containing Nb and V

To study the effect of thermal regime on the microstructure and microhardness of some experimental steels, microalloyed with niobium and vanadium, were made several charges with different chemical compositions, using two types of metallurgical units: a Balzers type vacuum induction furnace - VIM (Fig. 1) and vacuum arc remelting equipment - RAV (Fig. 2).

Fig. 1. Obtainment of microalloyed steels in the VIM furnace and metallic samples.

During the obtainment process was established a special regime for electrical heating and pressure, to ensure proper assimilation of alloying elements without exceeding the desired compositional gap. The program of experiments was based on a predetermined composition of microalloyed steel, Saarstahl - 38MnVS6 (38MnSiVS5) grade, used for automotive components such as crankshafts, bearings, shafts, hubs, piston heads, etc., having the chemical composition shown in Table 1.

Fig. 2. Preparation of metallic charge for VAR equipment and metallic samples.

Table 1. Chemical composition of 38MnVS6 steel, %.

C	Si	Mn	P	S	Cr	Mo	V
0.34 - 0.41	0.15 – 0.80	1.20 – 1.60	< 0.025	0.02 – 0.06	< 0.30	< 0.08	0.08 – 0.20

For the obtainment of the microalloyed steels have been used raw materials with low content of phosphorus and sulphur, due to the fact that neither of the two obtaining units (VIM and VAR) may not perform the processes of dephosphorization and desulphurization. In the furnace charge were used high purity materials: ARMCO (MK3) type iron, metallic Cr, metallic Mn, silicon, metallic V, sponge Ti, FeNb, graphite, etc.

The establishment of the metal load were taken into account theoretical levels of assimilation of elements in the melt, and possible evaporation losses during metallurgical process development in vacuum or controlled atmosphere of argon.

Thus, for charge composition was taken into account the following metallic elements losses: $a_C = 5\%$, $a_{Si} = 1\%$, $a_{Cr} = 1\%$, $a_{Mn} = 1\%$, $a_V = 1\%$, $a_{Nb} = 2\ \%$. These values were estimated based on data from the literature, considering: the oxidation degree of elements, specific characteristics, position in the series of electrochemical potential, unit characteristics, experience of researchers team relating to the obtaining process [9].

In the experimental program, charges were prepared having chemical compositions shown in tables 2 and 3. As result from chemical composition values of experimental microalloyed steels shown in Tables 2 and 3, the research aim was to analyze the separate and combined effects of V and Nb on the microstructure and microhardness. The samples marked with „0” do not contain niobium. For the other charges, which contain both Nb and V, it was attempted to maintain a constant value of one element, to evaluate the effect of increasing for the other.

2.2. Heat treatments

Application of usual heat treatment ensures only close compliance of requirements in yield strength. Toughness at -46°C is usually 20 to 120J (for KV test, considering the transition area) therefore it is very complicated to meet these requirements and ensure reproducibility of the results by applying conventional heat treatment [8]. In order to obtain better toughness, the samples has been heat treated at 950…970 ° C for austenitising by maintaining at high temperature for 20 minutes, using an electric furnace, followed by cooling in air. Subsequently, the samples were analyzed by optical microscopy, followed by microhardness measurements.

Table 2. Chemical compositions of experimental charges of microalloyed steel produced in VAR equipment.

Sample	Chemical composition, %									
	C	Si	Mn	P	S	Cr	Mo	V	Nb	Fe
38MnVS6 **VAR 0**	0.35	0.70	1.65	0.02	0.024	0.66	0.05	0.14	0	Ball.
VAR 1	0.34	0.69	1.65	0.022	0.019	0.41	0.05	0.14	0.12	Ball.
VAR 2	0.32	0.61	1.37	0.025	0.024	0.36	0.05	0.14	0.17	Ball.
VAR 3	0.38	0.74	1.67	0.025	0.022	0.44	0.05	0.16	0.21	Ball.

Table 3. Chemical compositions of experimental charges of microalloyed steel produced in VIM unit.

Sample	Chemical composition, %									
	C	Si	Mn	P	S	Cr	Mo	V	Nb	Fe
VIM 0	0.1	0.61	1.55	0.032	0.021	0.19	0.03	0.16	0	Ball.
VIM 1	0.3	0.44	1.17	0.024	0.017	0.19	0.03	0.16	0.13	Ball.
VIM 2	0.30	0.46	1.38	0.025	0.019	0.23	0.05	0.15	0.17	Ball.
VIM 3	0.26	0.43	1.51	0.027	0.021	0.24	0.05	0.16	0.22	Ball.

3. Results and discussion

Microstructural analysis of experimental microalloyed steels was performed by optical microscopy (Olympus GX51) using the etching reactive NITAL 5% [11]. Microhardness measurements were performed with Shimadzu HMV 2T microhardness tester.

The sample microalloyed only with V and 0.35% C, contains in microstructure fine martensite and austenite (VAR - Fig. 3), while the sample obtained in VIM, with a lower carbon content (0.1% C), contain grains of coarse needle-like ferrite, pearlite and bainite (Fig. 4). Using Nb as microalloying element allowed to reducing the proportion of martensite (VAR 1, Fig. 5).

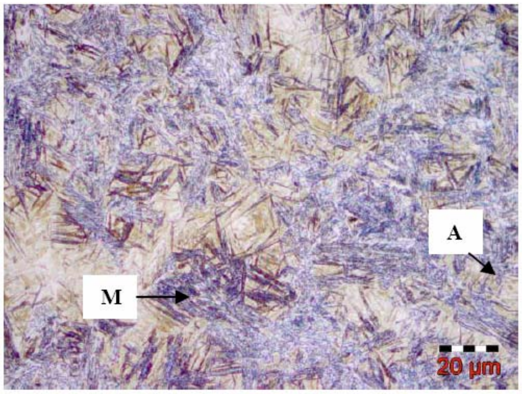

Fig. 3. Sample VAR 0 - Microstructure containing fine martensite (M) and austenite (A) untransformed, 1000x.

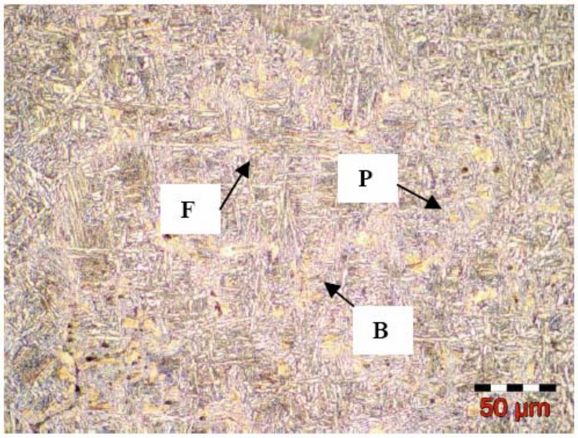

Fig. 4. Sample VIM 0 - Microstructure with coarse needle-like ferrite (F), pearlite (P) and bainite(B), 500x.

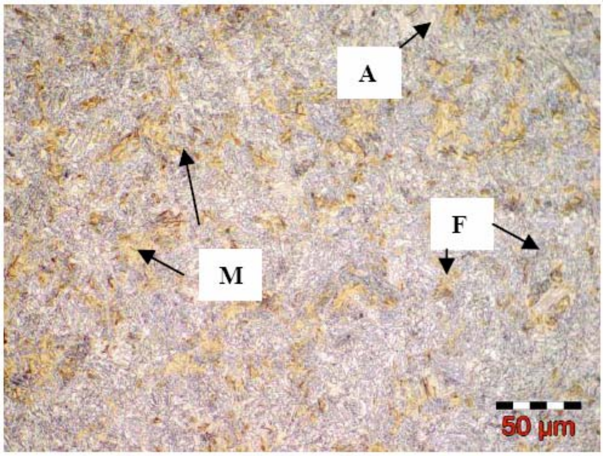

Fig. 5. Sample VAR 1 - Microstructure with fine martensite (M), needle-like ferrite (F) and untransformed austenite (A), 500x.

For sample VIM 1 was obtained a needle-like microstructure containing ferrite, pearlite, bainite and carbides (Fig. 6).

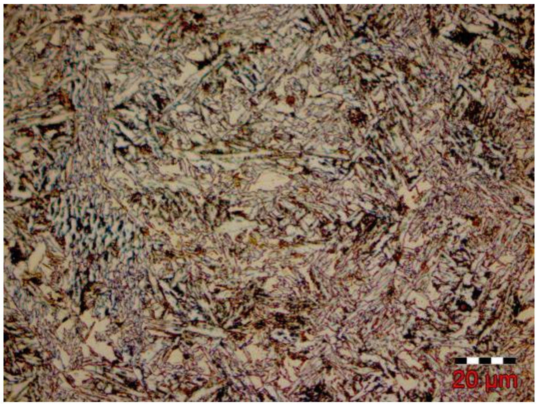

Fig. 6. Sample VIM 1 - Microstructure containing acicular ferrite, pearlite, bainite) and carbides, 1000x.

By increasing Nb content (0.17%) and keeping constant the V content (0.14%), it was obtained a microstructure almost similarly with those presented in Fig. 3, with the difference that is visible the precipitation of carbides (VAR 2, Fig. 7).

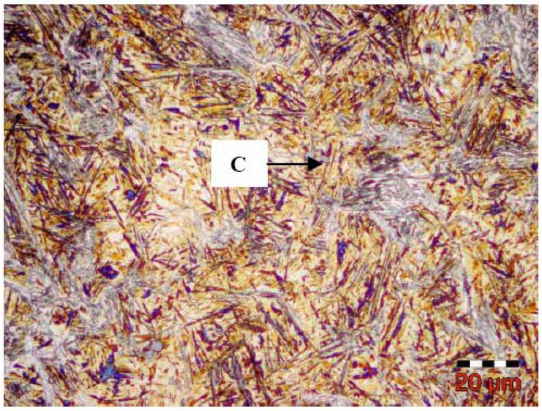

Fig. 7. Sample VAR 2 - Microstructure containing fine martensite, austenite untransformed and carbides(C), 1000x.

In the case of the sample obtained in VIM unit (Fig. 8), for 0.3% C, 0.15% V and 0.17% Nb, the microstructure is almost the same as in Fig. 6, with the difference that is visible the precipitation of carbides.

Further increase of Nb content to 0.21%, for a C content of 0.38% and 0.16% V (sample VAR 3), it was obtained a microstructure similar to Fig. 5 but with precipitation of carbides in needle-like ferrite areas (Fig. 9).

For the sample VIM 3, with a lower carbon content (0.26%), V (0.16%) and Nb (0.22%), the microstructure shows needle-like of ferrite, pearlite, ferrite pro-eutectoide, bainite and carbides precipitates inter and intra-granular (Fig. 10).

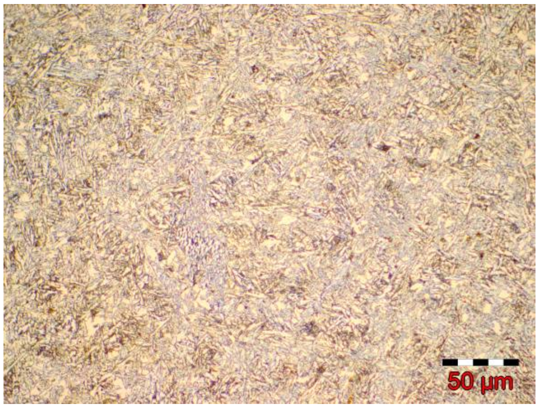

Fig. 8. Sample VIM 2 - Microstructure containing coarse acicular ferrite, pearlite, bainite and carbides, 500x.

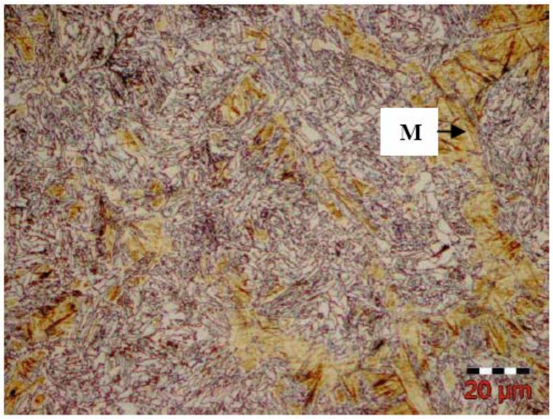

Fig. 9. Sample VAR 3 - Microstructure containing fine martensite(M), acicular ferrite, austenite untransformed and carbides, 1000x.

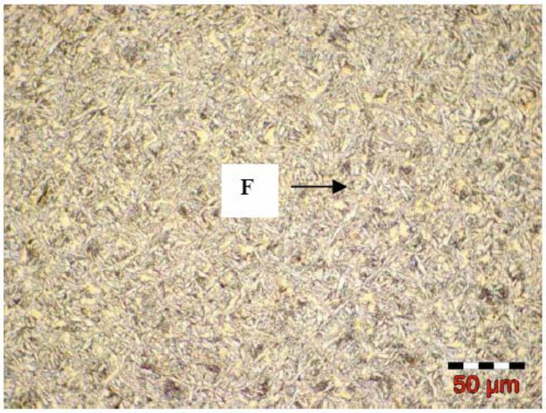

Fig. 10. Sample VIM 3 - Microstructure containing acicular ferrite(F), pearlite, pro-eutectoide ferrite, bainite and fines carbides precipitates, 500x.

Microhardness measurements reveal the degree of hardening of the metal matrix by the appearance of stable carbides that precipitated after alloying with V and Nb. The measured values of microhardness are shown in Table 4.

Table 4. Microhardness values $HV_1/10$.

Sample	Individual values					Average value
VAR						
VAR 0	478	482	483	491	488	**484**
VAR 1	328	342	333	374	327	**340**
VAR 2	609	644	647	669	649	**644**
VAR 3	380	373	372	370	367	**372**
VIM						
VIM 0	266	274	273	282	263	**272**
VIM 1	310	317	333	318	320	**320**
VIM 2	339	346	417	309	347	**352**
VIM 3	359	339	385	392	362	**367**

4. Conclusions

Microstructural analysis revealed the changes of phase morphology for certain combinations of elements (V + Nb), such as grain refinement and quenching, which lead to a combination of stable microstructural constituents (such as ferrite and pearlite) or metastable (martensite and bainite or carbides) which can provide good mechanical properties.

The samples obtained in VIM unit, maintained for longer periods at high temperatures, allowed to a greater progress in the phase transformation to yield constituents, like ferrite and pearlite type, mixed with different proportions of bainite and carbides. Due to rapid cooling in VAR equipment, mainly microstructure is composed of martensite with different proportions of needle-like ferrite, carbides and untransformed austenite.

The hardening effect of the metal matrix depends on the percentage of alloying with V or Nb, in correlation with carbon content, which also provides hardening by martensite formation and by the formation of carbides micro-precipitates.

The maximum hardening rate (644 $HV_1/10$) is obtained by simultaneous use of micro-alloying elements V (0.14%) and Nb (0.17%) for a carbon content of 0.32%. This is in correlation with the type of microstructure obtained, that contain fine martensite, untransformed austenite and carbides.

The increase of Nb content to 0.21% resulted in no further hardness increasing, while by alloying with V was obtained a hardness of 484 $HV_1/10$.

Very important is also the cooling rate of the samples, which greatly influences the microstructural changes that occur. Thus, by rapid cooling after melting in VAR equipment, before the heat treatment was obtained an average microhardness of 557 $HV_1/10$ (sample VAR 3).

Unfortunately, after the heat treatment, the microhardness decreased to 372 $HV_1/10$. This can be explained by the decreasing of the martensite percentage, by the increasing of untransformed austenite percentage and by formation of acicular ferrite due to the cooling in air.

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