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# High strength hot rolled and aged microalloyed 5%Ni steel

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## Properties

### **ABSTRACT**

**Purpose:** Purpose of this paper was to give information about low temperature strength and impact CharpyV toughness of low carbon microalloyed 5%Ni bainitic steel after thermomechanical rolling (TMR) or thermomechanical controlled processing (TMCP) and ageing at different temperatures: 580°C/2 h, 640°C/1h and 680°C/1h.

**Design/methodology/approach:** The tensile strength tests were performed at -196, -60 and 20°C and Charpy V samples were broken at -100, -80, -60, -40, -20 and 20°C temperatures. The tensile strength TS, yield strength YS, elongation A5 and reduction of area RA were established from tensile experiments. After TMCP 16 mm steel plate had YS = 730MPa, TS = 950 MPa, A5 = 22,5% and RA = 61% and impact energy > 50 J at -196°C.

**Findings:** The best combination of mechanical properties; yield strength and Charpy V toughness was achieved for steel after TMR and ageing  $580^{\circ}$ C/ 2h; YS = 800MPa, TS = 900 MPa, A5 = 22.5%, at -1000C KVmin.= 110 J.

**Research limitations/implications:** The precise methodology for retained austenite identification and its amount content determination in the investigated microstructures is still metallographic problem which needs to be resolved.

**Practical implications:** The best combination of yield strength and Charpy V toughness was achieved for steel after TMR and ageing  $580^{\circ}C/2h$ . At liquid nitrogen temperature ultrahigh strength properties were: YS = 1140 MPa, TS = 1280 MPa, A5 = 26%, RA = 55% and KV 122 J at -100°C.

**Originality/value:** The detailed microstructure examination of the steel with optical and mainly scanning transmission electron microscopy was needed to explain its good properties at very low temperature. TRIP effect was observed due to the presence of highly alloyed retained austenite in the microstructure. That type of steel may be used for contemporary military and structural applications working at low temperatures. **Keywords:** Properties of materials and products; Low carbon microalloyed bainitic steel

### **1. Introduction**

Ultrahigh strength is usually achieved in HSLA steels by increasing the carbon level (AISI 4340 type) or introduction of high copper addition to chemical composition (HSLA 130 steel) [1-3]. These steels are produced by a repeated quenching and reduced carbon content to 0.10-0.16%. However, the great amount of alloying elements (> 10%) such as Ni, Cr, Mo and Co are added to these steels to achieve an increased strength level [5,6].

In the present paper efforts have been made to develop low carbon steel with an improved toughness and weldability and satisfying strength level. Thus carbon content was reduced below tempering processes. The interstitial strengthening of carbon in martensite impairs toughness and weldability of steel. Recently this was observed for new developed steel with high amount of alloying elements: 0.18%C, 1.67%Mn, 3.36%Ni, 1.95%Cu, 0.78%Mo, 0.03%Al, 0.07%Ti, 0.05%Nb which after thermomechanical control processing (TMCP) at finishing rolling temperature in the range 850 – 700°C had YS 1456 MPa, UTS 1756 MPa but

low CVN impact Charpy V energy at -40 °C ~ 20J [4]. Better mechanical properties were achieved for another category of ultrahigh strength steels HY 180 and AF- 1410 with 0.05%C, sulphur <0.005%, phosphor < 0.01% and amount of nickel was increased to 5%, chromium 0.6% and molybdenum 0.45%. Microalloying of 0.03%Al, 0.07%V and 0.05%Nb was applied. A continuous cooling transformation diagram (CCT) of the investigated HN5MVNb steel was constructed from dilatometric measurements [7]. The influence of hot deformations and cooling rates after commencement of rolling on course of phase transformations was shown in previous paper [8]. The information gathered there allowed for selection of proper thermomechanical control processing of the steel. However, research in this area is still in progress [9-11].

#### 2. Work methodology, materials for research and CCT diagram

The HN5MVNb steel was prepared in an induction furnace under protective argon atmosphere, poured into ingots 100 x 100 mm and then after reheating at 1200°C/2 hours hot rolled to 20, 16 and 12mm thick slabs. Selected ingots were thermomechanical control processed (TMCP) to 12 mm thick plates according to deformation schedule described in paper [8]. These plates were cutted to the pieces which were aged after rolling at temperature 580°C/2 hours, 640 and 680°C during 1 hour. Charpy V longitudinal specimens and tensile samples 8 mm in diameter were machined from each plate. Charpy V energy was measured using 300 J pendulum hammer at temperatures -100, -80, -60, -40, - 20°C and room temperature. Ductile fracture surface was estimated from visual observation. The tensile tests were done on MIT machine at +20°C and low temperature -60°C and at liquid nitrogen temperature. Dilatometric specimens \$\$ 5 mm x 10 mm length were also prepared. Dilatation curves were obtained by heating the samples in an argon atmosphere at the rate  $10^{\circ}$ C/s to 860°C/300s and then cooling at various controlled rates from 0.05 - 80°C/s in Bahr 805 dilatometer. The Ac<sub>3</sub> and Ac<sub>1</sub> temperatures were 812°C and 669°C respectively. Figure 1 shows the CCT diagram obtained for the present steel.

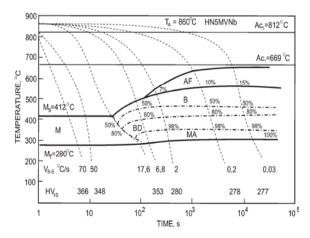


Fig.1. Continuous Cooling Transformation diagram of HN5MVNb steel

The martensite start temperature (M<sub>s</sub>) was established as 412°C and martensite finish (M<sub>F</sub>) as 280 °C being horizontal up to time which corresponds to cooling rate 10°C/s. Between 45 to 200 seconds only bainite- martensite transformation were detected. For lower cooling rates than 2ºC/s acicular ferrite bay was observed above bainitic one. It was established experimentally, that deformation of austenite in temperatures below 900°C accelerates ferrite transformation which proceeds bainitic formation at cooling rates in the range  $0.5 - 60^{\circ}$ C/s [8]. Thus thermomechanical working of austenite of HN5MVNb steel was possible during plate rolling. The remaining areas of austenite after ferrite transformation occurrence were transformed to bainite -martensite islands during cooling to room temperature. Typical microstructure of HN5MVNb steel after TMCP and air cooling to room temperature observed with optical microscope at magnification 500X is shown in Fig.2. The effect of deformation on elongation of former austenite grains may be seen because recrystallization stop temperature  $T_{R\gamma}$  has not been overcame when austenitizing at 860°C/300s. Influence of micro alloying with V, Nb and Al was estimated according to equation (1)

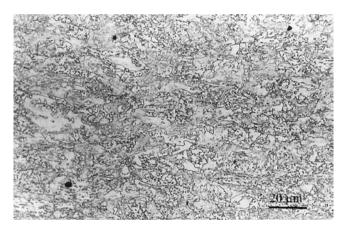


Fig.2. Microstructure of acicular ferrite and bainite –martensite islands of TMCP HN5MVNb steel plate cooled in air. Mag. 500X

$$T_{R\gamma} = 887 + 464C + 6445Nb - 644Nb^{1/2} + 732V - 230V^{1/2} + 890Ti + 363A1 - 357Si$$
 (1)

 $T_{R\gamma}$  value 957.65°C is far greater than 860°C thus recrystallization of austenite during reheating was uncompleted at austenitizing temperature for dilatometric investigation.

### 3. Description of achieved results

#### **3.1.** Mechanical properties

After TMCP rolling HN5MVNb steel when tested at room temperature had YS = 730 MPa, TS = 950 MPa,  $A_5 = 22.5\%$ , RA = 61% while at liquid nitrogen temperature YS = 970 MPa, TS = 1180 MPa,  $A_5 = 15.5\%$  and RA = 51%.

It is obvious that lowering tensile testing temperature will increase the yield and tensile strength of steel under investigation and lowers its plasticity. That is not true in the case of HN5MVNb steel. Only reduction of area of round samples was decreasing with lowering the tensile test temperature while plasticity  $A_5$  measured in percentage was increasing to 26% or was on stable level 22% i.e. higher than that for samples in as rolled state ( $A_5$  in the range 15.5 – 22.5%) The yield strength and tensile strength of the specimens aged at 680°C/1 hour were lower at – 60°C and -196°C from those reported for steel in as rolled state. Contrarily ageing at 580 – 640°C was increasing both YS and TS when comparing to as rolled state samples. So mechanical properties depends on microstructure conditioning during ageing (tempering) of as rolled structure of the investigated steel.

The detailed mechanical properties of HN5MVNb steel at room and low temperature -60°C and -196°C are given in table 1 and shown in Figures 3-6.

Table 1.

Low temperature mechanical properties of HN5MVNb steel

Heat	Temp.	Re	Rm	$A_5$	RA
treatment	${}^{0}C^{-}$	MPa	MPa	%	%
As rolled	+20	730	950	22.5	61
	-60	850	1050	18.5	57
	-196	970	1180	15.5	51
580°C/2h	+20	800	900	22.5	66
	-60	880	1060	24	60
	-196	1140	1280	26	55
640°C/1h	+20	700	860	22.5	65
	-60	819	1024	24	54.6
	-196	1140	1230	26	55
680 <sup>0</sup> C/1h	+20	640	840	22	73
	-60	840	970	22	60
	-196	960	1060	22	56

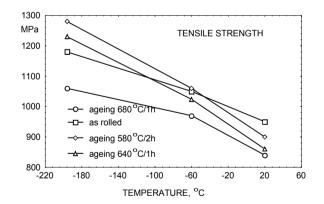


Fig.3. Tensile strength properties of HN5MVNb steel at roomand low temperatures in as rolled condition and after different ageing heat treatments: 580°C/2h, 640°C/1h, 680°C/1h

From presented results it may be stated that ageing at temperature  $620 \pm 20^{\circ}$ C during one hour would be the best condition for highest tensile and plastic properties of HN5MVNb steel. The lowest YS and TS values were achieved after ageing at

 $680^{\circ}$ C/lh and elongation was at the stable level A<sub>5</sub> = 22% in the range +20 to -196°C.

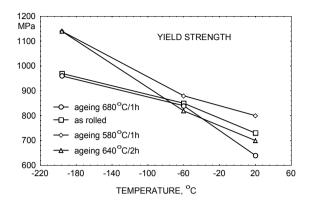


Fig.4. Yield strength of HN5MVNb steel at room- and low temperatures in as rolled condition and after different ageing heat treatments: 580°C/2h, 640°C/1h, 680°C/1h

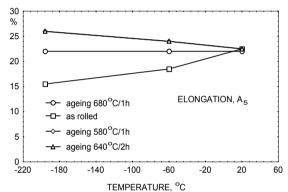


Fig.5. Elongations  $A_5$  % of HN5MVNb steel tensile specimens at room- and low temperatures in as rolled condition and after different ageing heat treatments:  $580^{\circ}C/2h$ ,  $640^{\circ}C/1h$ ,  $680^{\circ}C/1h$ 

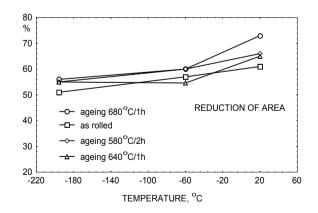


Fig.6. Reduction of area of round 8 mm samples of HN5MVNb steel at room- and low temperatures in as rolled condition and after different ageing heat treatments: 580°C/2h, 640°C/1h, 680°C/1h

The Ac<sub>1</sub> temperature equals 669°C, thus ageing at 680°C was done just above critical temperature in the ferrite + austenite range. The newly formed austenite was highly supersaturated with carbon and alloying elements as manganese [12] and nickel at 680°C. So on fast cooling to room temperature austenite was partly maintained in sample as remaining austenite in the microstructure. Austenite is usually softer phase than ferrite thus mechanical properties of the microstructure consisted of recrystallised ferrite and islands of austenite and low carbon bainite-martensite components has lower tensile properties and highest reduction of area from other tensile specimens.

After ageing at  $580^{\circ}$ C/2h TS is the highest when testing at temperatures – 60 and -196°C (Fig.3) and YS has highest values in the testing range -196 to +20°C (Fig. 4). This situation may be explained by morphological changes in the structure of the tempered bainite. We observe precipitation hardening of the only partially recrystallised ferrite matrix. In some grains polygonization process of bainitic and martensitic lathes was observed with the help of transmission electron microscope Philips 301G. The islands of slightly tempered martensite was also visible in the microstructure what is shown in figure 7.

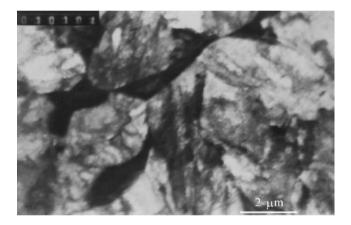


Fig.7.Mirostructure of HN5MVNb steel after TMCP and ageing at  $580^{0}\mathrm{C/2hours}$ 

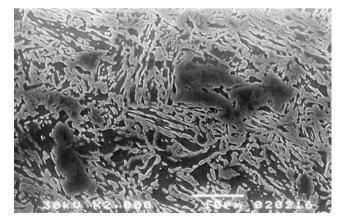


Fig.8. Microstructure of HN5MVNb steel aged 640°C/1h after TMCP observed with SEM

The characteristic microstructure at higher tempering temperature 640°C/1h was observed with the scanning electron microscope (SEM) Joel 5400 at magnification 2000 X due to very fine lathes of tempered bainite and some carbides on their grain boundaries. The areas of recrystallised ferrite was also visible. That type of microstructure had highest YS at  $-196^{0}$ C and toughness and impact energy in the whole range of testing temperatures  $-100^{\circ}$ C to  $20^{\circ}$ C.

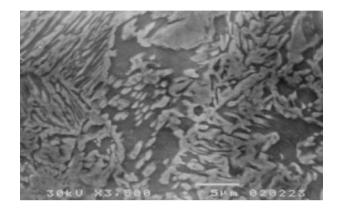


Fig.9. Microstructure of HN5MVNb steel after ageing at 680°C/1h and fast cooling to room temperature

On cooling from 680°C/1h temperature new low carbon martensite was formed. Precipitation of carbides around former austenite grains were seen what is shown in figure 9. This structure was observed with SEM Joel 5400 on slightly etched specimens. Broad areas of the recrystallised ferrite and fresh martensite as well as retained austenite are also visible.

# **3.2.Charpy V impact toughness** energy tests

The ductile properties after different ageing temperatures of as rolled plates were established from Charpy V impact tests performed at 20,-20, -40, -60, -80 and -100°C temperature. The hammer 300 J energy was applied when striking the Charpy V specimens.

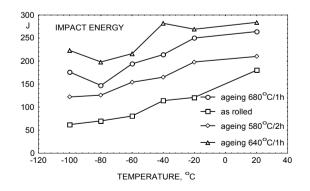


Fig.10. Impact energy of breaking of longitudinal Charpy V specimens at given test temperature of HN5MVNb steel after different state conditions

Fracture areas were always ductile for each of the three specimens broken at given testing temperature. Thus only the average impact energy values were shown in figure 10 at testing temperature.

In order to fulfill the regulations for brittle fracture steel grade a Charpy V toughness is tested at -50 to -80°C. For very thick plates and higher yield strength grades a tempering process can be used after the accelerated cooling of thermomechanically rolled steels. TM- rolled plates with minimum yield strength values of 500 MPa are supplied in thickness up to 100 mm for hydropower, offshore platforms and special ships [13].

In order to have the highest degree of material redundancy against brittle fracture, the toughness requirement was defined by a Charpy V test at  $-80^{\circ}$ C [14,15]. For civil engineering required energy KV at  $-80^{\circ}$ C is 50 J while for military ship application 100 J. So in as rolled TMCP condition HN5MVNb steel plates may be used in civil engineering. For the military applications ageing (tempering) after rolling is needed. The best toughness was achieved after 640°C/1h, almost 200 J was determined at  $-80^{\circ}$ C. The small lowering of Charpy V energy values after 680 °C/1h beneath those established for ageing 640 °C/1h are a consequence of transformation of retained austenite highly saturated with carbon into twinned martensite under high strain rate and stresses developed during breaking of specimens at low temperature.

Effect of acicular ferrite (AF) amount and martensiteaustenite islands (M-A) on Charpy V impact strength was investigated by De Vito for X80 grade steels [16]. The slightly detrimental influence of M-A islands on toughness was established in equation (2) for maximum impact strength in [J/cm<sup>2</sup>].

$$KCV_{max} = 0.52*AF + 112 \exp(-0.042*M-A)$$
 (2)

Assuming 15% of M-A islands in acicular ferrite matrix of HN5MVNb steel tested at -80°C the calculated value from above relationship gives value 103.85 J/cm<sup>2</sup> which corresponds to energy 83J. That value is lower than 120 J determined at -80°C for steel tempered at 580°C/2h but higher than energy 70 J for as rolled (TMCP) plate. With first approximation mentioned above equation may be used to evaluate KCV toughness of the steel.

#### **3.3.STEM** investigations

Confirmation of the existence of martensite – austenite islands was done by chemical analysis of small grainy areas with energy dispersive spectrometer (EDS) attachment combined to STEM Philips C20M. The characteristic X- ray intensity was measured and quantitative calculations of the elements observed within the island are presented in figure 11. Assuming 10 to 15% of M-A islands present in the microstructure of steel tempered at  $680^{\circ}$ C/1h, they contain 0.3333 to 0.50% C and 0.6%Si, 0.3%V, 0.8%Cr, 1.5%Mn and 6.9%Ni. It is possible to estimate local M<sub>s</sub> temperature of such island using relationship (3) given by Mikuła and Wojnar [17].

$$\begin{split} M_S &= 635,02-549,82C-85,441 Mn, - 68,967 Si - 18,07 Cr - \\ &30,965 Ni - 69,301 Mo - 6,603 V + 420,26 Nb + 553,8 Ti \\ &- 1746,5 B \end{split}$$

The calculated values of  $M_s$  temperature depending on carbon concentrations equals  $52^{0}$ C for 0.333%C or  $-39.5^{0}$ C at 0.50%C in the island. Martensite start temperature below room temperature allows for retention some amount of austenite which did not undergo phase transformation on cooling from ageing temperature  $680^{\circ}$ C. Thus the existence of M-A islands was confirmed by mathematical calculation. The additional confirmation was done by electron diffraction in STEM and X-ray measurements of the content of different elements in retained austenite in samples after ageing at  $680^{\circ}$ C/1h (Fig.11) and higher temperatures in alfa + gamma temperature range [18-22].

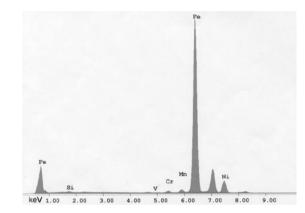


Fig.11. Characteristic X-ray intensities of the elements present in area of the martensite- austenite island in microstructure of HN5MVNb steel tempered at 680<sup>o</sup>C/1h after TMCP

Mainly higher concentrations of Ni in the range 6.3-7.0% and Mn 1.5-2.5% were observed in retained austenite islands. The retained austenite undergoes martensite transformation under stresses and strain during tensile testing increasing the tensile strength and also uniform elongation as well as total elongation of the specimens (Fig.5). Transformation induced plasticity (TRIP) effect was beneficial for ductility of tensile specimens and to the some extent for impact energy measurements with Charpy V specimens which were broken below -80°C (Fig.10). On the other hand ageing at 680°C/1h caused decrease of the values of impact energy when compared to values measured after  $640^{\circ}$ C/1h. One possible explanation is that local micro segregation of chemical composition of steel leads to formation of stable austenite lathes during tempering at 640°C. They improve fracture toughness of steel plates (Fig.10) and give the best elongation properties at tensile testing even at low temperatures down to liquid nitrogen temperature (Fig.5).

### 4.Conclusions

- 1. The heavy plates of HN5MVNb steel may be used for structural applications in as TMCP condition with mechanical properties as follows YS >730 MPa, TS > 950MPa,  $A_5 > 22\%$  and at  $-60^{\circ}$ C KV > 60 J.
- 2. For military and civil engineering constructions for severe applications additional ageing (tempering) after TMCP is needed

to fulfill requirements of YS above 640MPa and good plasticity and very good toughness of 100 J at temperature - 80°C.

- 3. The best combination of mechanical properties; yield strength and Charpy V toughness was achieved for steel after TMR and ageing 580°C/ 2h; YS = 800MPa, TS = 900 MPa,  $A_5 = 22.5\%$ , at -100°C KV<sub>min</sub> = 110 J.
- 4. From presented results it may be stated that ageing after TMCP at temperature  $620 \pm 20^{\circ}$ C during one hour would be the best heat treatment for the highest tensile and plastic properties of HN5MVNb steel.
- Existence of retained austenite islands in the microstructure of HN5MVNb steel heavy plates aged at 680°C was confirmed.
- 6. Transformation induced plasticity (TRIP) effect was observed in HN5MVNb steel after ageing at 680°C/1h and was beneficial to the ductility of tensile specimens and to some extent increased the impact energy of Charpy V specimens which were broken below temperature -80°C.

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