

## Effect of vanadium microaddition on the microstructure and mechanical properties of casing pipes after normalizing process

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### ABSTRACT

**Purpose:** The aim of the paper is to determine the impact of vanadium microaddition on mechanical properties and microstructure of two steel grades with a different content of V, applied for production of casing pipes used in the extractive industry.

**Design/methodology/approach:** Pilger process technology with subsequent normalizing was used. The tests were carried out on an industrial batch produced in the Rurexpol Department, and the research was done with the use of industrial research laboratories of Alchemia S.A. Group. The microstructure and mechanical properties were determined in the initial state and after normalizing at two temperatures: 940°C and 880°C. Static tensile test and Charpy V impact test have been applied.

**Findings:** Mechanical properties, macrostructure and microstructure of steel pipes, obtained during production under industrial conditions, have been analyzed in the article. The effects of V content and hot-working and normalizing treatment affect substantially the mechanical properties and microstructure of the investigated steels.

**Research limitations/implications:** It was found that the application of vanadium microaddition to the normalizing process after the hot-rolling has a beneficial effect on a strength-toughness balance only at a narrow range of technological parameters of the normalizing.

**Practical implications:** The knowledge of the vanadium microaddition effect on the industrial production route is indicated in the paper.

**Originality/value:** : The effect of vanadium microaddition has been proved to be very sensitive to the temperature conditions of the heat treatment.

**Keywords:** Microalloying; Hot-rolling; Normalizing treatment; Casing pipe; Toughness

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### PROPERTIES

## 1. Introduction

Microalloyed steels are now more and more commonly used, successfully replacing common unalloyed steels or C-Mn steels, basically in all fields of industry [1-3]. The use of microalloyed steels is substantial because of weight reduction of constructions and improvement of utility indicators, as well as the possibility of meeting a number of strength and technological requirements [4-6]. They are also less expensive when comparing to toughened steels [7]. Taking into consideration the fact that microalloyed steels are primarily assigned for products of the machine-building industry, marine constructions-drilling and mining platforms, lifting devices, systems for transportation of liquid gases, pressure vessels and other highly loaded structural elements, high metallurgical purity, fine-grained microstructure and a high level of yield strength (YS min. 500 MPa) is required, while still maintaining proper ductility and guaranteed crack resistance at decreased operating temperature [8-12].

These quality requirements also apply to seamless steel pipes. The production of such pipes is one of the most difficult hot-working processes, where depending on applied technology, high levels of hot processing degrees ( $\lambda=1.2-6.0$ ) can be attained [13]. Depending on the application, the following assortment of seamless pipes can be distinguished: drill, boiler, constructional and stainless steel pipes. In the recent years, particular attention has been given to drill pipes used in the extractive industry, due to fluctuations of petroleum prices. The drill pipes are assigned for geological and exploration drilling and for deposit mining; these are obtained in accordance with API-5CT, API-5D standard. They enclose [13-15]:

- casing pipes-applied to protect walls of producing wells and to separate water, oil and gas courses.
- drill pipes-used for rotary and percussion drilling. Their task is to transfer the power from the engine to the drilling rig and to supply drilling mud to the bottom of the well.
- mining pipes - used for oil and gas output.

Pipes production technology is dated back to 1889, when Mannesmann brothers invented their method. Nowadays, seamless pipes are produced in pilger, pusher, automatic, continuous, three-high and two-high helical rolling mills. The most modern and most efficient are cone roll piercers, driven by Diescher's disc guides, continuous jobbing mills (4-5 stand) with three-high PQF stands, Assel mills with floating or controlled mandrel and multistand (24-30 stands) reduction mills with group drive, planetary, Accu-Roll mills - with controlled mandrel [13-16].

Currently, the market trends encourage the use of microadditions in steels assigned for production of pipes with continuous thermo-mechanical rolling due to simplification of manufacturing processing (avoiding normalization process) and cost reduction through careful selection of elements such as V, Nb, Ti and N, which influence refinement of microstructure and lead to precipitation strengthening [17,18].

Analyzing the current offer of many companies producing seamless steel pipes, it can be observed that the application of modern methods of rolling, in conjunction with the use of microadditions, has significantly increased the quality while reducing production costs and introduced new standards into this market. The key requirement was to decrease the C content and simultaneously the carbon equivalent  $C_e$ , even in case of pipes, for which good weldability is not required. For this reason, it has been attempted in the work to use a feedstock for production of pipes, in which microaddition of vanadium will be present, aiming to impact grain refinement and precipitation strengthening during the production using pilger technology, which historically launched the technological development of seamless pipes production.

## 2. Material and research methodology

Two grades of steel were tested, differing in concentration of Mn and V. These grades are to be applied for production of casing pipes, wherein an increased amount of V was used (Table 1). Particular attention in the work was given to the impact of V microaddition on the microstructure and mechanical properties, achieved after pilger rolling process and subsequently after normalization.

Table 1.  
Chemical composition of the investigated steels

	C	Mn	Si	P	S	V	N
Grade 1	0.29	1.27	0.31	0.014	0.002	0.084	0.0061
Grade 2	0.32	1.55	0.27	0.017	0.007	0.168	0.0143

Heats were obtained in the converter process in the ArcelorMittal Poland Dąbrowa Górnicza, in two formats: 280x300 mm and 280x400 mm; the 280x400 mm format was subjected to additional re-rolling into 300x300 mm format. Rolling of ingots was conducted in Alchemia SA Huta Bankowa in Dąbrowa Górnicza, and rolling of pipes took place in Alchemia SA, in Rurexpol Division.

Table 2.  
Heat treatment parameters for raw material and normalizing process

Dimensions			Soaking		Raw material	Normalizing		
Diameter, mm	Wall, mm	Grade	Temperature, °C	Time, h	Sample	Temperature, °C / Time, min.		Sample
244.5	8.94	1	1260	4	No. 1	940	22	No. 2
244.5	8.94	1	1260	4	No. 3	880	22	No. 4
244.5	10.03	2	1260	4	No. 5	940	25	No. 6
244.5	10.03	2	1260	4	No. 7	880	25	No. 8

Tests were performed by rolling pipes from the size and API 5CT series of types: 244.5 with two gauges: 8.94 mm and 10.03 mm, with different soaking temperature of the mill feedstock prior to rolling and normalizing temperature shown in Table 2.

Austenitizing temperature of the charge was equal to 1260°C and 1320°C. The difference in normalizing temperature was equal to 60°C, i.e.  $T = 880^{\circ}\text{C}$  and  $940^{\circ}\text{C}$ . Mechanical properties in the normalized state were determined for specimens from the mill feedstock austenitized at the temperature of 1260°C.

Before starting the tests, calculations of solubility of vanadium nitrides in a temperature range from 900 to 1200°C were performed. Thermodynamic equation, describing the product of MX-type phase solubility in austenite, was implemented. For conditions of equilibrium, the product of vanadium nitride solubility is expressed with the following dependence:

$$\log [V] [N] = 3.02 - 7840 / T \quad (1)$$

where  $[V]$  and  $[N]$  – are the concentrations of microalloy (V) and interstitial (N) dissolved in austenite (wt-%) at the  $T$  temperature [K].

The experimental part began with the studies of macrostructure of the charge, depending on the content of V. Calibration was performed according to BN-76/0601-10. In the second stage, metallographic microscopic examinations were carried out at magnifications of 100 and 500 times and nital was applied to reveal the microstructure of metallographic specimens. Tests were done on Neophot-2 optical microscope. When analyzing metallographic images, special attention was paid to the impact of V microaddition on the microstructure, depending on the austenitizing and normalizing temperatures.

Mechanical properties were determined on the basis of static tensile test, both in the initial state and after normalization process, using specimens cut according to the axis of the pipe. Tests were carried out using Zwick 1200E testing machine and Zwick hammer with breaking energy of 300 J, using three samples for which arithmetic mean was determined. Impact tests were performed on V-notched samples at the temperature of 0°C. Due to thickness of the pipe and its rounding, 7.5 mm undersize samples with a 2 mm notch were used. Tensile and bar test pieces were prepared in accordance with ASTM A370/ASTM E23 standards.

### 3. Results and discussion

#### 3.1. Macrostructure and thermodynamic calculations

The macrostructure of the feedstock for both steel grades (grade 1 and grade 2) are presented in Figs. 1 and 2. Both patterns meet the requirements of charge assigned for production of pipes, and were calibrated in accordance with BN-76/0601. In case of grade 2, only fine inner cracks can be observed, which are not critical during the production of pipes when located this way. It's associated with increased concentration of V, which in both cases was added in a form of nitro-vanadium with determined nitrogen in converting process. Taking into account the atomic weight of vanadium and nitrogen and the content of these elements in the steel (Table 1), it can be calculated that the concentration of vanadium required to bind total nitrogen is equal respectively to:  $3.64 \times 0.0061 \text{ wt.\% N} = 0.022 \text{ wt.\%}$  – for grade 1 and  $3.64 \times 0.0143 \text{ wt.\% N} = 0.052 \text{ wt.\%}$  – for grade 2 (less than in chemical composition of both steels). Hence it follows, that the whole nitrogen should be bound

into VN particles, and the relative portion of vanadium bound into VN is equal approximately to 25% and 33% of the total content of this element in steel. The remaining part should be bound into VC particles or can be present in dissolved state in a solid solution.

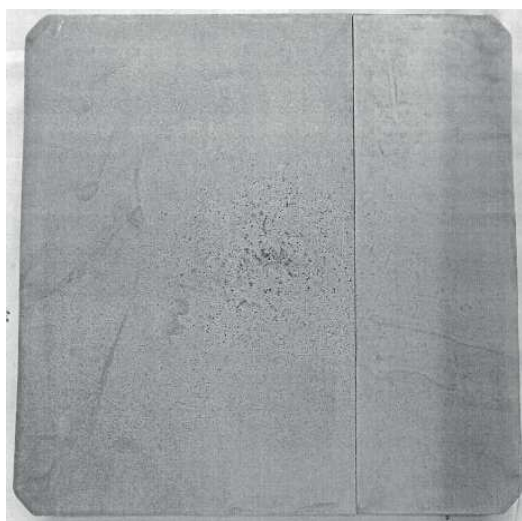


Fig. 1. Macrostructure of the low-V steel

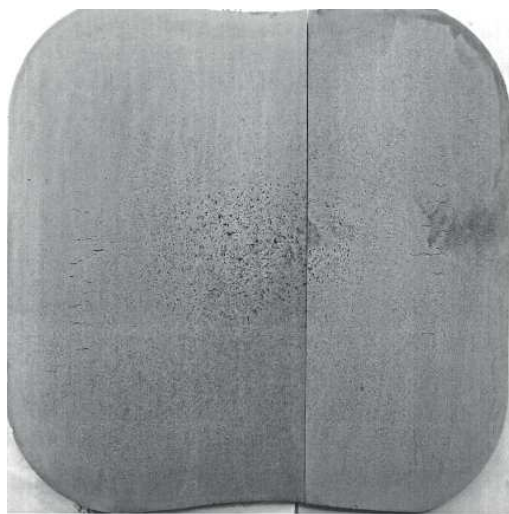


Fig. 2. Macrostructure of the high-V steel

Minor stratifications visible in Fig. 2 suggest that the portion of vanadium nitrides and carbides in the steel no. 2 is significant, and precipitation strengthening caused by the presence of considerable quantity of particles leads to local delaminations.

Thermodynamic calculations, carried out on the basis of the equation (1), allowed estimating a precipitation temperature of VN particles in austenite of both steels

under equilibrium conditions. In case of steel with the lower concentration of vanadium, this phase precipitates starting from the temperature of about 970°C (Fig. 3). Taking into consideration that finish temperature of pilger rolling is in a range from 1000 to 900°C (experimentally determined), it shall be assumed that practically whole portion of VN precipitates precipitate during slow air-cooling following hot-rolling.

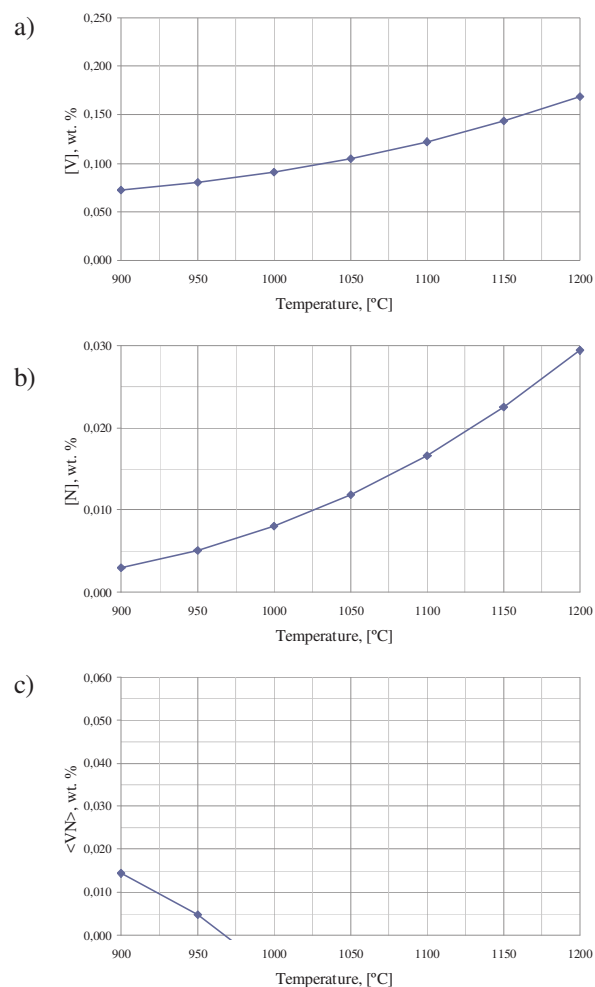


Fig. 3. Temperature range of VN precipitation in the austenite of the low-V steel: a) wt% V dissolved in the solid solution, b) wt% N dissolved in the solid solution, c) wt% fraction of <VN> compound precipitated in the austenite

In case of steel with twice as high concentration of vanadium, the temperature of the beginning of VN precipitation increases significantly to about 1120°C (Fig. 4). The portion of vanadium and nitrogen dissolved in a solid solution decrease along with the decrease of temperature.

As a result, the weight amount of precipitated VN is equal approximately to 0.06 % (Fig. 4c) at the temperature of 900°C and is about 4 times higher when compared to the grade 1 (Fig. 3c). This means that VN particles in the steel with higher concentration of vanadium precipitate already during pilger rolling, and subsequently after its finish.

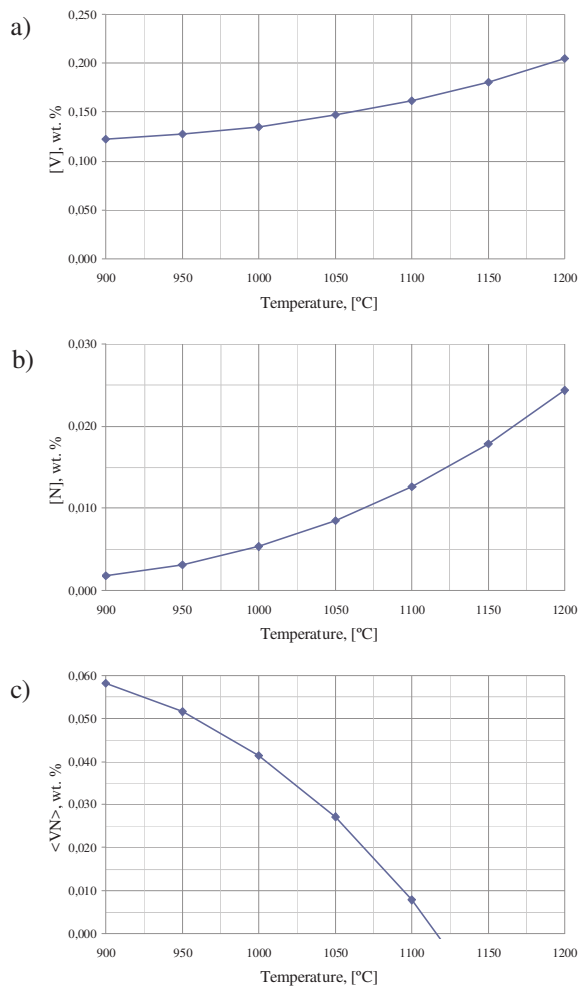


Fig. 4. Temperature range of VN precipitation in the austenite of the high-V steel: a) wt% V dissolved in the solid solution, b) wt% N dissolved in the solid solution, c) wt% fraction of <VN> compound precipitated in the austenite

### 3.2. Properties and microstructure in feedstock state

Mechanical properties were determined for two grades of steel, differentiated by austenitizing temperature. The yield strength (YS) and tensile strength (UTS) of the

steel in feedstock state are presented in Fig. 5. The figure shows that differentiation of austenitizing temperature in a range from 1260°C to 1320°C has no substantial effect on mechanical properties for both steel grades. The value of yield stress for grade 1 is at the level of 540 MPa, and tensile strength reaches from 760 to 790 MPa. The data presented in Table 3 reveals also that the austenitizing temperature has no significant influence on the elongation (A) and on the impact toughness.

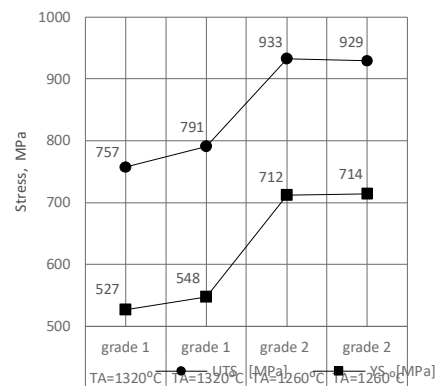


Fig. 5. Effects of soaking temperature and steel grade on yield stress (YS) and ultimate tensile strength (UTS) for raw materials

However, it can be observed that the steel with higher concentration of vanadium is characterized with much higher mechanical properties in comparison with the grade 1. The yield stress is higher by about 170 MPa and a tensile strength by about 140 MPa. Elongation is equal approximately to 25% and is by 6% lower than in case of steel with lower concentration of vanadium. Impact energy for both steel grades samples in initial (feedstock) state is relatively low and is in a range from 14 to 40 J, yet the values are lower for the steel containing 0.168 wt%. of vanadium.

Different mechanical properties of investigated steels in the initial state result from the microstructure, being a derivative of chemical composition. In case of the grade 1, it is ferritic-pearlitic microstructure with slight prevalence of pearlite (Fig. 6). The observed sample reveals 3/2A banding microstructure in accordance with PN-H-04504: 1963 standard, which is acceptable at this stage of production. In case of steel austenitized at the temperature of 1320°C, the portion of pearlite is similar, but slightly smaller defragmentation of pearlite colonies can be observed (Fig. 7). This may explain lower impact energy of Charpy V-notch specimens, when compared to steel austenitized at lower temperature (Table 3).



Table 3.

Results of mechanical properties for the feedstock materials

Dimentions			Feedstock state				
Diameter	Wall thickness	Grade	Soaking temp.	YS	UTS	A	KV <sub>ave</sub>
mm	mm		°C	MPa	MPa	%	J
244.5	8.94	1	1260	527	757	33	40
244.5	8.94	1	1320	548	791	30	22
244.5	10.03	2	1260	712	933	26	14
244.5	10.03	2	1320	714	929	24	21

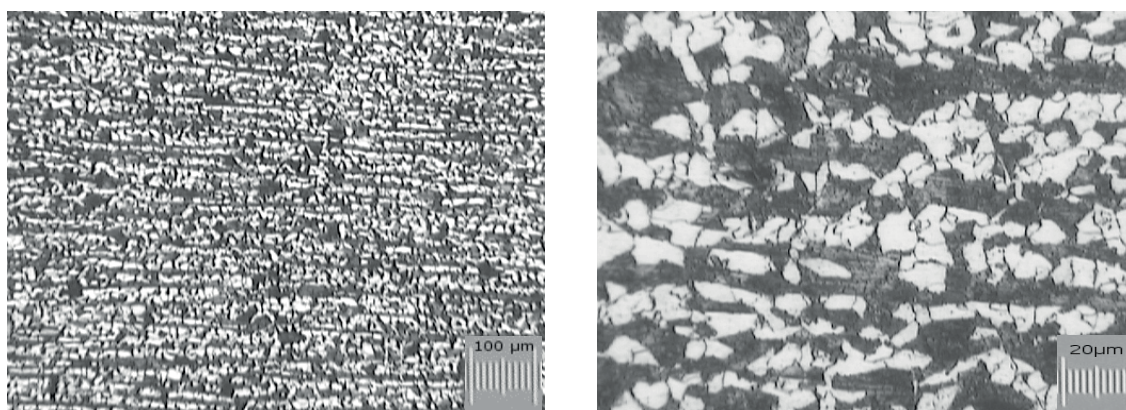


Fig. 6. The band-like ferritic-pearlitic microstructure of the low-V steel in the initial state after the soaking at 1260°C

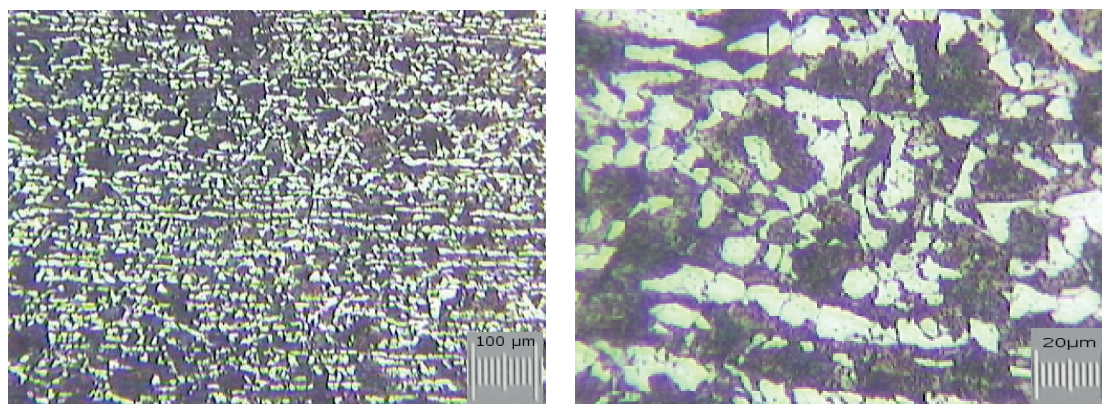


Fig. 7. The band-like ferritic-pearlitic microstructure of the low-V steel in the initial state after the soaking at 1320°C

Higher mechanical properties and lower impact toughness of steel no. 2 should be explained by increase in the portion of pearlite, which is associated with a somewhat higher concentration of C and Mn in the steel.

The course of recrystallization during elongating rolling and successive pilger rolling, leads effectively to grain

refinement of austenite, and after phase transition, also forming relatively fine-grained ferrite and pearlite (Fig. 8), although the size of some of pearlite colonies reaches 40 µm. Contribution of larger portion of pearlite is difficult to be quantitatively separated from strengthening interaction of particles of vanadium nitride. It will be the subject of further research.

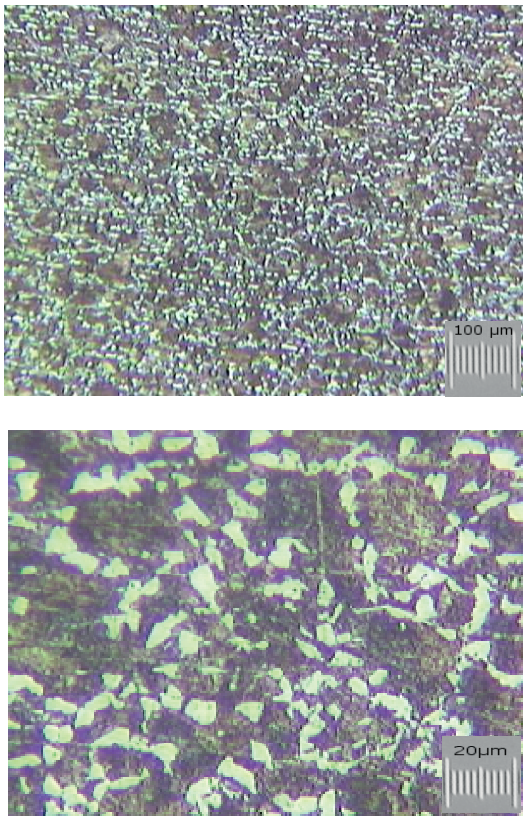


Fig. 8. The pearlitic-ferritic microstructure of the high-V steel in the initial state after the soaking at 1260°C

### 3.3. Mechanical properties and microstructure in normalized state

The properties in the initial state varied considerably, depending on austenitizing temperature. For this reason, samples were taken for further research in normalized state, after austenitizing at the temperature of 1260°C (Table 4).

Table 4  
Mechanical properties for the normalized state

Pipes after normalization process								
Grade	Austenitizing temp., °C	YS	UTS	A	KV			
					Ave.	I	II	III
		MPa		%				
1	940	497	736	32	49	48	54	44
1	880	514	720	33	52	46	53	57
2	940	563	718	29	71	66	62	84
2	880	665	888	29	22	24	23	20

Figure 9 presents mechanical properties after normalization at two temperatures, i.e. 880°C and 940°C. In case of grade 1, decrease of yield and tensile strength by about 30 MPa (Table 4) at the elongation at the same level, were obtained for both values of normalizing temperature.

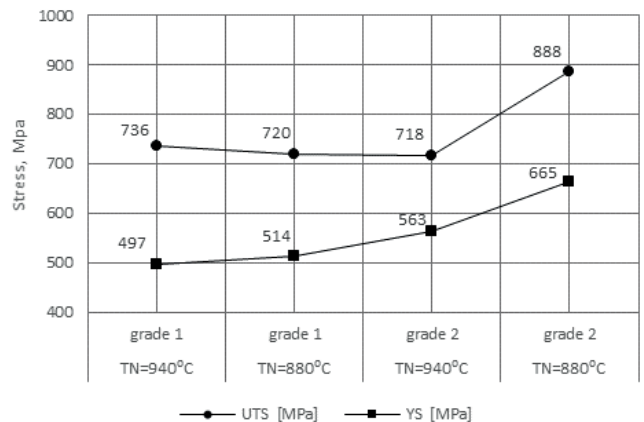


Fig. 9. Strength properties after the normalizing treatment under various conditions

In case of steel austenitized at the temperature of 940°C, slight refinement of structural constituents was obtained (Fig. 10), in comparison with the initial state (Fig. 6). This refinement is to be assigned to the pearlitic-ferritic transformation into austenite during heating, and then re-transformation of austenite into a mixture of ferrite and pearlite. In relation to the initial state, minor increase in the portion of ferrite can be noted, which leads to decrease of mechanical properties (Fig. 9). Increasing the amount of the  $\alpha$  phase along with refinement of microstructure results in increased impact energy of Charpy V specimens up to about 50 J (Fig. 11).

Reduction of normalizing temperature to 880°C does not significantly affect mechanical properties and fracture toughness of the steel 1 (Table 4), when compared with samples normalized at the temperature of 940°C. No meaningful changes in the microstructure were observed (Fig. 12). It shall be assumed that re-heating the steel to 880°C resulted in partial dissolution of VN particles formed after rolling and their complete dissolution at the temperature of 940°C (Fig. 3). Due to their relatively small portion (Fig. 3c), the differences in thermal stability and their repeated precipitation after normalization do not lead to significant differentiation of mechanical properties.



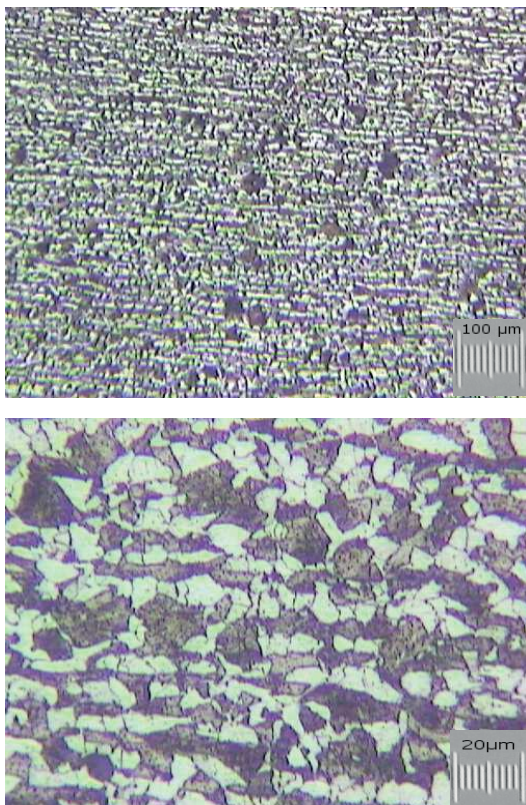


Fig. 10. The ferritic-pearlitic microstructure of the low-V steel after the normalizing treatment at 940°C

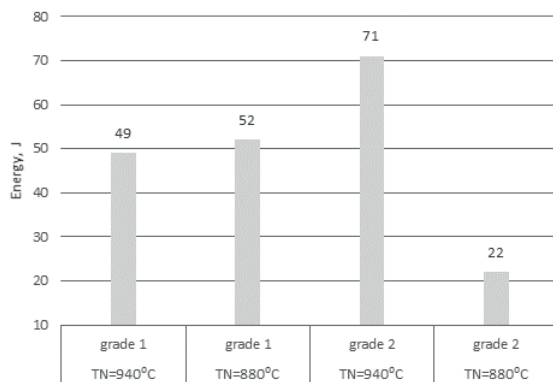


Fig. 11. Results of impact toughness for two steel normalized at different temperatures

Considerably greater differences in mechanical properties can be noted for the grade with higher content of vanadium. This applies both to comparison of initial and normalized states, and the differences between normalizing temperature (Fig. 9). In case of steel 2, normalized at the temperature of 940°C, decrease of

yield strength by about 150 MPa as well as a decrease of tensile strength by over 200 MPa were obtained, when compared with the state prior to normalization (Tables 3 and 4). The elongation increased by 3%, while impact energy of Charpy V specimens increased from 14 to 71 J (Fig. 11).

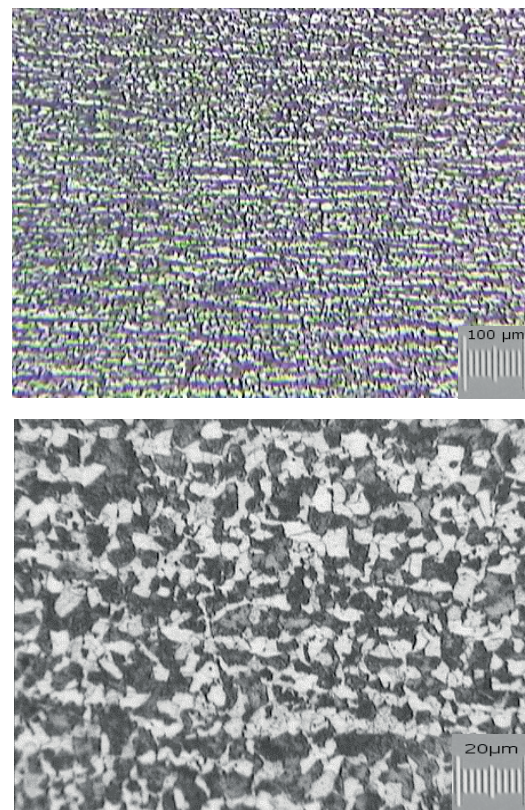


Fig. 12. The ferritic-pearlitic microstructure of the low-V steel after the normalizing treatment at 880°C

The change in mechanical properties is accompanied by substantial refinement of microstructure and significant increase of portion of ferrite (Fig. 13) in comparison with the initial state (Fig. 8). Higher content of ferrite explains the decrease of mechanical properties, which, however, is greater than it would be expected, taking into account high concentration of vanadium in the steel. Nevertheless, it follows that most of VN particles was dissolved during normalization and levelled the effect of precipitation strengthening. This explains also considerable increase of impact toughness of steel austenitized at the temperature of 940°C.

Unlike the steel 1, the decrease of normalizing temperature in case of steel 2 substantially affects the



change in both mechanical properties and ductility of steel. In comparison with the initial state, yield strength and tensile strength decreased only by about 50 MPa (Tables 3 and 4). In this case, increase of ferrite amount and significant grain refinement (Fig. 14), overcoming the decrease of mechanical properties, was also noted. However, characteristic banding arrangement of ferrite and pearlite grains results in low impact toughness of steel held at the temperature of 880°C (Fig. 11).

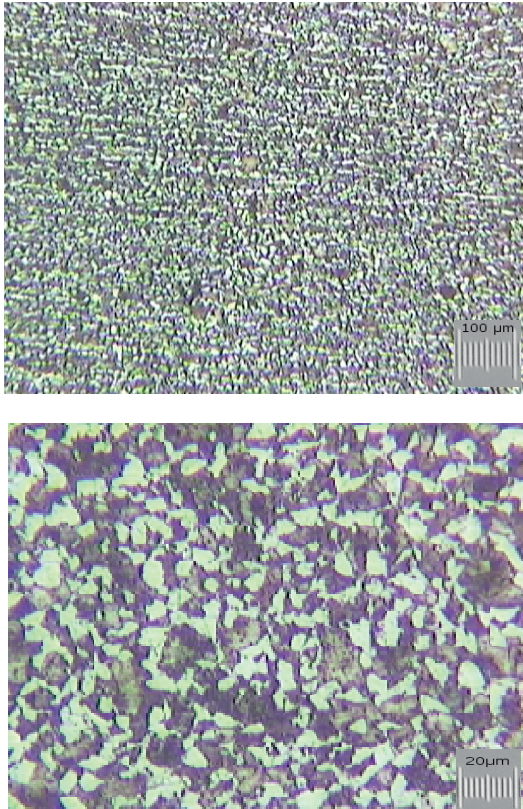


Fig. 13. The ferritic-pearlitic microstructure of the high-V steel after the normalizing treatment at 940°C

In principle, the impact toughness of samples is comparable with the initial state. Banding arrangement of grains can suggest normalization of steel at the transition between the single-phase austenite and the two-phase range. In this case, the fraction of vanadium particles is also considerably larger than in the steel 1. Taking into account lower normalizing temperature, it should be assumed that these particles remain undissolved, rendering normalization process of austenite difficult. For these austenitizing conditions, normalization has not proved to be effective in improving impact resistance of steel.

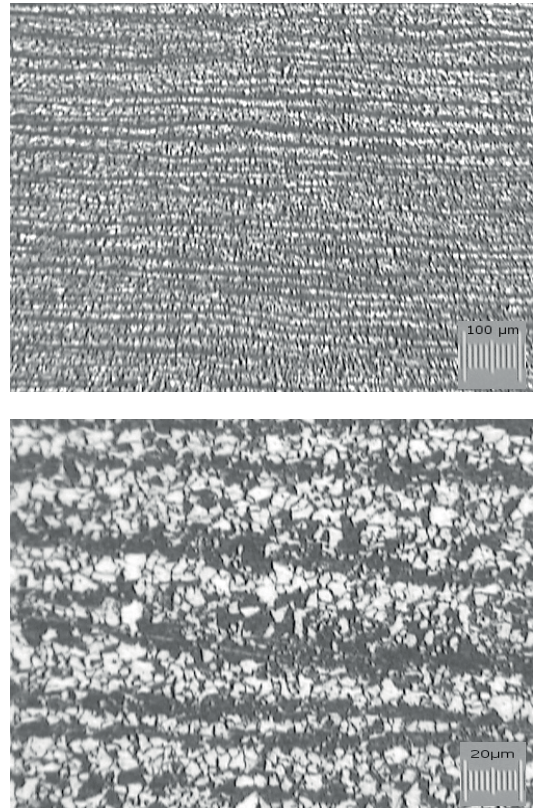


Fig. 14. The ferritic-pearlitic microstructure of the high-V steel after the normalizing treatment at 880°C

#### 4. Summary

The industrial research carried out on two steel grades with different concentrations of V and N has revealed that significant increase in the content of these microadditions did not result in distinct improvement of mechanical properties. Moreover, high content of V in combination with high concentration of C and Mn adversely affect macrostructure of the feedstock, contributing to formation of cracks inside of blooms. In case of steel with lower concentration of vanadium, improvement in impact strength with a slight decrease of mechanical properties was attained, irrespectively from normalizing temperature. The potential increase of strength, resulting from refinement of microstructure, is leveled by a partial softening of precipitation strengthening.

The steel with higher concentration of V is more sensitive to the conditions of normalization. Normalization at the temperature of 940°C results in the increase of impact toughness, but with significant (more than 200 MPa) decrease of mechanical properties. In turn, the temperature

of 880°C is too low for proper normalization of steel, what results in no improvement of impact toughness. It follows that normalization after the process of pilger rolling of steel with V microaddition requires strict control of heat treatment conditions, due to the opposing processes of grain refinement and decrease in precipitation strengthening from the particles containing vanadium.

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