THE EFFECT OF V-CARBIDES PRECIPITATION ON TENSILE AND FATIGUE PROPERTIES OF HEAT TREATED SPRING STEELS

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Abstract

This paper deals with a comparison between the standard spring steel grade 0.54C-1.45Si-0.7Mn-0.7Cr and a grade micro-alloyed spring steel with 0.12 % V in terms of microstructure, grain refinement, strength and ductile properties. The micro-alloyed steel is supposed to provide a better performance as it had been proven for hot-rolled and heat treated wire. The vanadium micro-alloyed steel grades are quenched in oil typically, so high strengths and high ductility can be expected. It has been confirmed especially for induction heat treatment and quenching temperatures 810 - 910 °C, and the tempering temperatures 430 °C and 450 °C. That phenomenon can be explained with the strengthening effect and precipitation of vanadium carbides that suppress the grain coarsening effectively. The mechanism has been confirmed with electron scanning and transmission microscopy.

Keywords: Spring steel, micro-alloying, vanadium carbides, precipitates, plasticity

1. INTRODUCTION

Nowadays, a reduction of vehicle weight leads to a reduced amount of exhausted gases (mainly CO_2) and belongs to the biggest saving trends in the automotive industry. Based on that, modern lightweight products are very welcomed. In other words, the requirements for car producers using steels are more demanding. Additionally, the costs make a huge impact on any development as the automotive production is a mass product field. All of these factors influence the implementation of micro-alloyed steel grades seriously.Vanadium shows the best combination of properties and costs for its implementation in mass production. As a micro-alloying element it is frequently used by steel producers in order to improve the characteristics of steel in terms of mechanical properties, grain refinement and delayed fracture propagation with significantly low effect on the costs [1, 2]. Another typical alloying element for micro-alloying steels - niobium - was deliberately excluded from this research although niobium micro-alloyed steel was reported to exhibit excellent results in the suppression of grain size grow [1]. Steel grades in this study are mass produced and used for a production of coil springs. For such application it is important to provide not only significant ultimate tensile strength (UTS) over 2100 MPa, but also excellent fatigue properties as coil springs are working under cyclic load up to 10^6 of cycles. In general, increasing in strength means decreasing of ductility, however the aim of modern technology is to identify the optimal parameters of the heat treatment that can provide satisfactory values on required level.

2. MEASURING PROCEDURE

In this study, two grades of spring steel were produced in a convertor steel plant. As a novelty, the chemical composition of one steel grade was additionally micro-alloyed with vanadium, then silicon was used for a deoxygenation and desulfurization was done in the standard way. Similarly for both grades, the billets were produced by continuous casting with slow down cooling. After a re-heating the hot rolling followed. Wire rod 14.00 mm in diameter was produced with a cooling down with an air cooling conveyor. After the cooling a cold drawing followed with a reduction of the diameter to 12.50 mm. Finally, the process was finished with a rapid induction heat treatment.

The microstructures of the cross cut wire rods and wires after the heat treatment were investigated by means of scanning electron microscope (SEM) Zeiss Sigma 300, and transmission electron microscope (TEM) Philips CM12. As the etching agent the Nital (nitric acid in alcohol, volumetric concentration 2%) was used. The grain size was measured at the longitudinally cut samples by linear method according to the ASTM E112, in 6 pictures taken for each sample as for the required accuracy. Samples for grain size measuring were additionally annealed for 1 h under the temperature 550 °C.Mechanical properties of the material were identified by means of tensile test machine WPM UPC 1200 and standard cylindrical samples.

3. HOT-ROLLED WIRE

The microstructures and content of exact elements of steel grades (in wt. %) can be seen in **Figure 1** and **Table 1**. The complete chemical composition is not presented due to protective reasons. Steel grade **A** is a standard spring steel grade and steel grade **B** is referred as a V-micro-alloyed steel. Both steel grades show a perlite-ferrite microstructure (**Figure 1**).

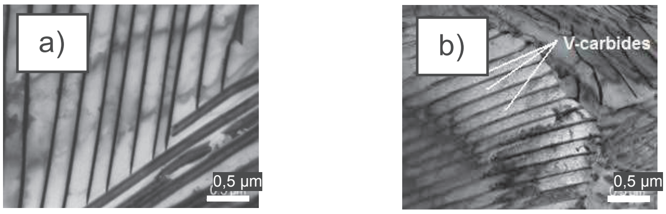


Figure 1 Microstructure of wire rod, TEM. *(a)* - steel A, *(b)* - steel B

As the temperature of re-heating of billets before the hot rolling is higher than the defined solution temperature of V-carbides (1019 °C), vanadium is expected to be in a solid solution state [3]. Primary V-carbides that can be referred as carbides that are produced during continuous casting process and dissolve during hot rolling process. It was identified for the steel grade **B** in this work by TEM examination (**Figure1b)**). On the opposite, the steel grade **A** does not have any ingredients contributing to the carbide formation (**Figure 1a)**).

Table 1 Chemical composition (wt. %).

	C	Si	Mn	Cr	V	Fe
Steel A	0.55	1.39	0.73	0.66	-	ballanced
Steel B	0.60	1.55	0.56	0.55	0.16	ballanced

Tensile mechanical properties of the wire rod for steel grades **A** and **B** were defined by standard tensile tests, see **Table 2**. Steel grade **B**, micro-alloyed with vanadium, showed higher tensile strength (30 %). It can be explained by a higher content of carbon first, but also with the vanadium strengthening effect and grain refinement. An interphase precipitation into lamellae structure can be observed in **Figure 1**. The SEM pictures of the fractured surfaces after the tensile test (**Figure 2**) show a ductile breakage mechanism, with a formation of micro-voids.

Table 2 Mechanical properties of investigated steel grades in hot-rolled wire condition

	Tensile strength, MPa mm^2	Reduction of area, %
Steel A	1040	54
Steel B	1352	46

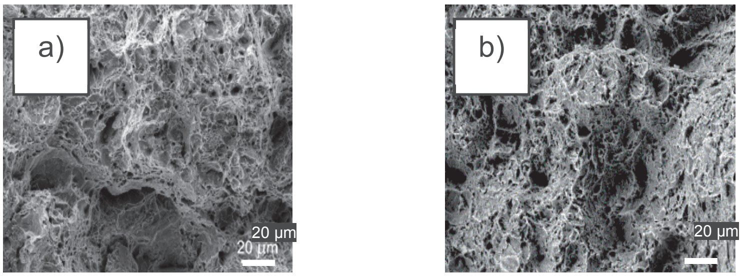


Figure 2 Fracture area. (*a)* - steel A, (*b)* - Steel B

4. HEAT TREATMENT

Quenching and tempering (QT) were performed in water spraying box after rapid induction heating. Range of austenitization temperature (T_a) was 810 - 910 °C with sampling and steps of 20 °C in the range. It should be pointed out that due to technological specification of the equipment for the rapid induction heating, the initial heating-up temperature of the samples was 80 °C higher than T_a. It was measured by a pyrometer at material surface. It is important to take into account when the grain size is discussed because T_a is the temperature that defines the structure and mechanical properties after the QT. Anyway, the range of temperatures is lower than the solution temperature (1019 °C), so no V-carbides can dissolve in steel **B** during austenitization.

Tempering temperature (T_t) 450 °C was chosen in order to investigate the structure and mechanical properties of steel grade **B**. Additionally, T_t = 430 °C was chosen to test in order to obtain a higher strength after QT process.

4.1. Grain size

It is known that the growing of grain size is observed with the increasing of austenitization temperature for any steel grade, but at the same time alloying elements such as molybdenum, niobium, vanadium, etc. can suppress the growth of grains. Over the years of research on vanadium micro-alloyed steel grades it was proved that it suppresses the growth of grains within the wide range of quenching temperatures in comparison with construction steel grades due to formation of V_4C_3. However, the temperature of 1000 - 1010 °C was defined as the critical temperature where the abnormal growth of grains for V-micro-alloyed steels occurs and then the growth of coarsened grains is stabilized with the grain diameter about 70 µm [1 - 4].

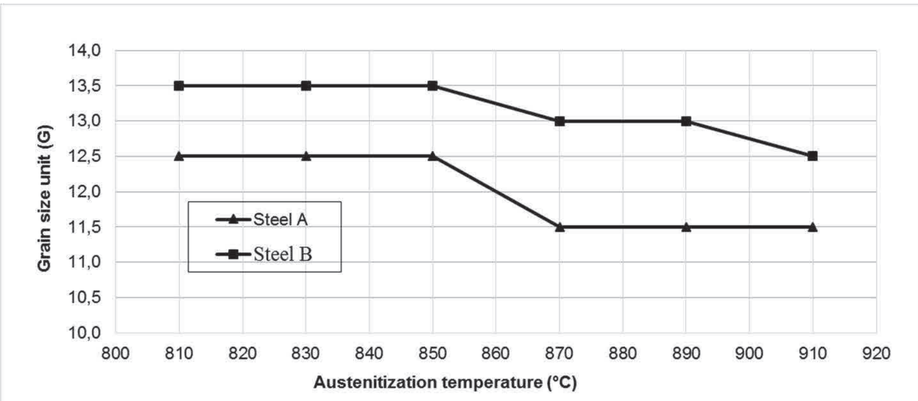


Figure 3 Grain size depending on the austenitization temperature

In this study, the austenitization temperature 850 °C for both steel grades was found as a critical temperature for the beginning of the grain growth (**Figure 3**). However, grain size of the steel grade **B** was 1.0 - 1.5 G-value finer. In this way the effect of V-carbides in micro-alloyed steel of grade **B** can´t prevent the growth of grains. The decrease of grain size value G was observed a bit similar to the V-free steel (**Figure 4**).

It was also experimentally proven for steel grade **A** that the speed of heating does not affect the grain growth below the austenitization temperature of 850 °C. So, the low austenitization temperature is more significant than the speed of heating. Observed results also correspond well with the curve obtained by Maropoulos and others [3] apart of the different test conditions (a long-time conventional heating in furnace). On the other hand if the austenitization temperature is above 850 °C, the time of heating becomes more significant even for rapid induction heating. The longer heating time is used for bigger diameters, so it can be expected that the wire of smaller diameter would have finer grains under the same temperature.

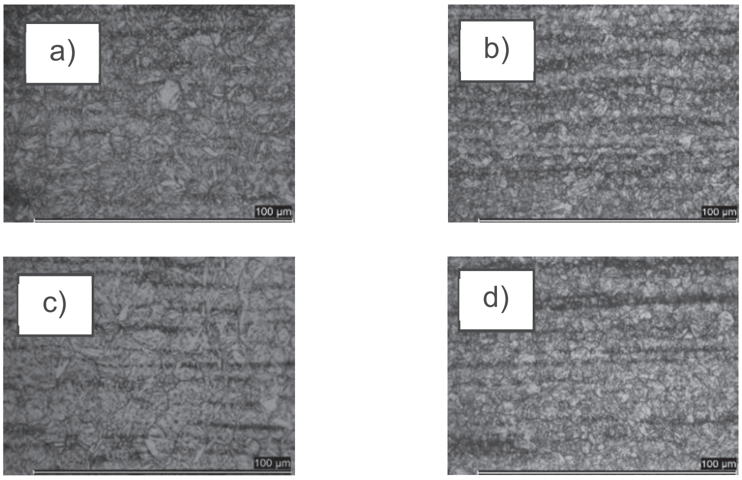


Figure 4 Grain size. T_a = 850 °C: *(a)* - steel A, *(b)* - steel B. T_a = 890 °C: *(c)* - steel A, *(d)* - steel B

4.2. Microstructure

The austenitization temperature of 850 °C was previously defined as a borderline for the beginning of grain growth. The samples made under the conditions of quenching / tempering 850 °C / 450 °C were chosen as a starting point for the investigation in order to compare the structures of steel grades **A** and **B**.

The observed martensitic microstructure of steel grade **A** corresponds with all previous works. According to diagram presented in [5] steel up to 0.6 wt. % of carbon shows after QT treatment lath martensite in the structure (**Figure 5, a)**).

As the carbon content of steel grade B is 0.60 wt. %, nano-twins become the significant part of the microstructure [5] that was proved by TEM observation (**Figure 5, b)**; **Figure 6**). The next factor of the modification of the microstructure of steel **B** is vanadium addition. Vanadium spreads the bainite area in TTT diagram so that the mixed structure of bainite and martensite could be easily obtained under the same cooling conditions. For steel grade **B** retained austenite at the bainite-martensite matrix was observed.

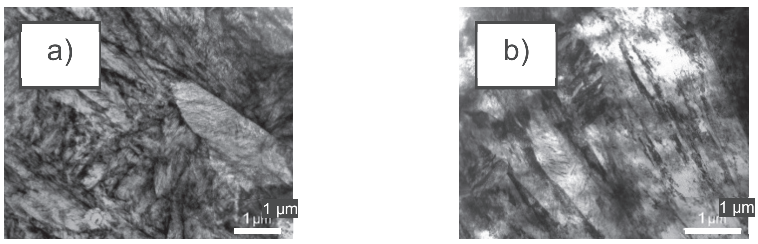


Figure 5 Microstructure of QT wire, TEM. (*a*) - steel A and *(b)* - steel B

Both steel grades consist of more than 0.5 wt. % of carbon that could lead to intergranular embrittlement, especially under low temperature tempering conditions. During the first stage of tempering (100 - 200 °C) the fine ε-carbides are formed within the martensite laths. Crystal and morphological changes in ε-carbides (Fe_2C) were described in [6]. During the second phase of tempering when the temperature is higher than 200 °C, ε-carbides (Fe_2C) convert into cementite (Fe_3C) [2]. At the same time addition of silicon retards formation of ε-carbides (Fe_2C) and move the tempering embrittlement to higher temperatures [7].

The SEM analyses of the structures for steel grades **A** and **B** perfectly illustrate the precipitation of carbides (**Figure 6**). Firstly, carbides are formed on the boundaries of prior austenite grains (PAG). Then also carbides are visible at the martensite block boundaries and within the lath. The reason for the intercrystalline failure along the boundaries of PAG and the decrease of fracture properties can be found in a formation of layer of precipitates at the boundaries of PAG. The precipitates have been proved to be a chain of globular particles that coalescence and look like a film under higher magnifications [8, 9]. Intergranular failures were not observed in this study.

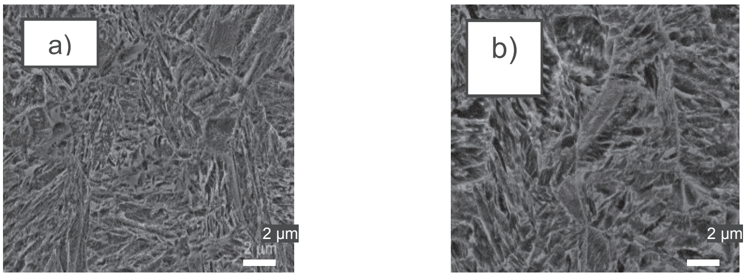


Figure 6 Precipitation of cementite (Fe_3C): (*a*) - steel A, (*b*) - steel B

4.2.1. Mechanical properties

Mechanical properties of steel grade **A** were taken as a reference for the investigation of micro-alloyed steel. The results of tensile tests are given in **Table 3** for both steel grades anyway. Based on the results of SEM investigation steel grade **A** shows a ductile fracture morphology and acceptable properties in terms of yield and ultimate tensile strength. Due to higher content of carbon and the vanadium strengthening effect samples of steel **B** prevailed in strength with a difference of 200 MPa, but exhibited a lower ductility under the same temperature conditions. The best combination of ductility and strength values for steel grade **B** were observed for the samples 2B and 6B, produced under T_a / T_t parameters 850 °C / 430 °C and 850 °C / 450 °C correspondently. Sample 3B also has acceptable ductility / strength combination, but grain coarsening was observed for Ta = 870 °C (**Figure 3**). Three QT state of steel grade **B** could be implemented in the production. SEM investigation of tested samples 2B, 3B and 6B show ductile mechanism of breakage (**Figure 7**).

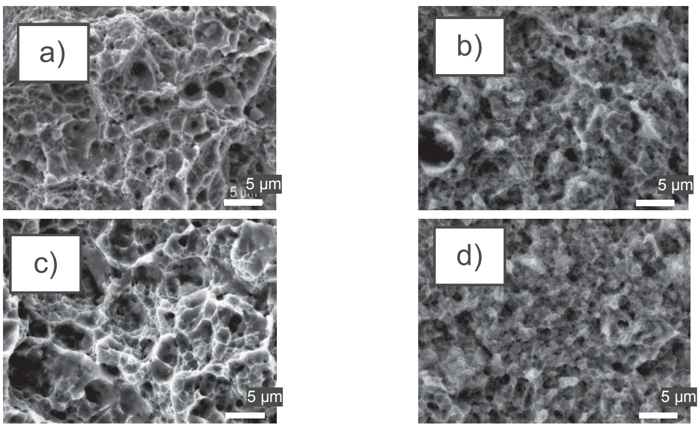
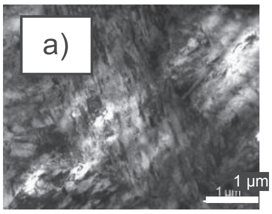


Figure 7 Fracture area, SEM. (*a*) - sample 1A, (*b*) - sample 2B, (*c*) - sample 3B, (*d*) - sample 6B

Table 3 Mechanical properties of steel grades A and B after QT process

Steel grade	Sample number	T_a / T_t, °C	Yield strength, MPa	Tensile strength, MPa	Reduction of area, %
Steel A	1A	850 / 450	1805	2002	54
Steel B	1B	830 / 430	2068	2282	24
	2B	850 / 430	2046	2285	31
	3B	870 / 430	2026	2283	36
	4B	810 / 450	2026	2200	33
	5B	830 / 450	2055	2217	32
	6B	850 / 450	2061	2221	40
	7B	870 / 450	2045	2218	42

For the samples 2B and 6B that show the optimal mechanical properties the investigation of structure was performed by TEM (**Figure 8** and **Figure 6** correspondently).



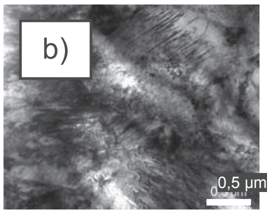


Figure 8 Microstructure of QT wire, TEM. *(a ,b)* - steel B, sample 2B

5. CONCLUSION

Steel grade **B** micro-alloyed with vanadium after the processing and rapid inductive heat treatment (QT) showed an acceptable combination of the mechanical properties (up to 2280 MPa UTS and 30 - 35 % of reduction of area) and morphology of fractured surfaces. The result can be used in the spring production. The parameters of the heat treatment allow to reduce the diameter of the wire significantly and hence to reduce the coil spring weight.

Further optimalization of QT is planned with respect to the improvement of ductile properties of the heat treated material and preservation of the high UTS. As the next step to continue this investigation a study of carbon partioning process to obtain optimal microstructure of the material can be done, as it has been suggested e.g. in [10].

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