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Influence of higher partitioning temperatures on mechanical properties of heat treated high-strength steel alloyed with 1.3 % chromium

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Abstract. This paper deals with the innovative Quenching and partitioning (QP) heat treatment of low-alloyed chromium steel, and is especially focused on the higher temperatures of partitioning as well as longer holding times and its influence on mechanical properties of the material. As part of the experiment various heat treatment, metallographic analysis, hardness measurement, X-ray diffraction phase analysis (to determine retained austenite content), tensile test and Charpy impact test were performed. In QP treated specimens the best combination of tensile strength and elongation was observed after quenching in 200 °C salt bath and partitioning at 250 °C for 30 minutes. This specimen showed the tensile strength above 1900 MPa, elongation of 14 % and also good impact toughness (34 J). Equally good values of impact toughness (36 J) were also observed in the specimens partitioned at 300 °C or 320 °C for holding times up to 30 minutes. On the other side longer partitioning times at the temperature of 320 °C affected the elongation and impact toughness negatively.

1 Introduction

Current automotive structures and their safety requirements place great demands on the material and its mechanical properties. In the high strength steels considerable ductility and good fatigue properties together with high ultimate tensile strength (UTS) are often required. In order to achieve such a combination of mechanical properties the application of the innovative methods of processing is necessary. A convenient technology of heat treatment (HT) may be represented by quenching and partitioning (QP), nanobainitisation or some other derived processes utilizing the beneficial properties of film morphology retained austenite (RA) in the microstructure [1, 2]. These methods depend on precise parameters of processing. In the QP processing it is a cooling rate, temperature of austenitization and quenching media during the quenching step as well as temperature and holding time of the immediately following partitioning. After the quenching a martensitic microstructure with increased RA content is obtained. During the partitioning step diffusion processes (carbon migrating from supersaturated martensite to RA) results in stabilization of RA in the microstructure [3-5]. Due to film morphology RA in martensitic or bainitic microstructure composite-like microstructure with increased ductility and toughness is obtained [5-7].

Some experiments with the 42SiCr steel were carried out using QP process on thermomechanical simulator [8, 9] and also in the real process. Different quenching temperatures and quenching media were tested in order to change cooling rate and also different temperatures and holding times



of partitioning were applied [9-11]. The martensitic/bainitic microstructures with thin film RA were obtained. A goal of the study was to establish relations between the mechanical properties and microstructure development with respect to the method of HT. As described in this paper, previously tested [9, 11] relatively low temperatures and short holding times was completed with higher temperatures and longer holding times of partitioning in order to clarify the development of microstructure and ductility during such processing.

2 Experiment

As the experimental material high-strength steel alloyed with 1.3 % chromium and 2.03 % silicon was chosen (table 1). Increased silicon content should suppress carbide precipitation and helps to stabilize austenite during partitioning [10]. Chromium improves hardenability and together with carbon ensures high strength of the steel. The steel was further alloyed by some amount of manganese which should delay the carbide precipitation and pearlite transformation [9]. The suitability of the chemical composition for QP process was verified elsewhere [8, 12]. The phase transformation temperatures were calculated in JMatPro software.

Table 1. Chemical composition and transformation temperatures of the experimental steel.

C	Si	Mn	P	S	Cr	Mo	Ni	Al	Nb	Ms	Mf
[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[wt%]	[°C]	[°C]
0.43	2.03	0.59	0.009	0.004	1.33	0.03	0.07	0.008	0.03	298	178

The experimental material was supplied as the 10 mm thick rolled strip from which the specimens of approximately 110×80×10 mm were cut using waterjet. The initial microstructure was martensitic/bainitic with small amounts of pearlite and free ferrite (figure 1). Its hardness was 391 HV10. Prior to the experimental HT, the specimens were homogenized at 1100 °C for 4 hours in a furnace with an argon atmosphere. The resulting microstructure was pearlitic with a small amount of granular ferrite at prior austenite grain boundaries (figure 1) and the hardness was 280 HV10.

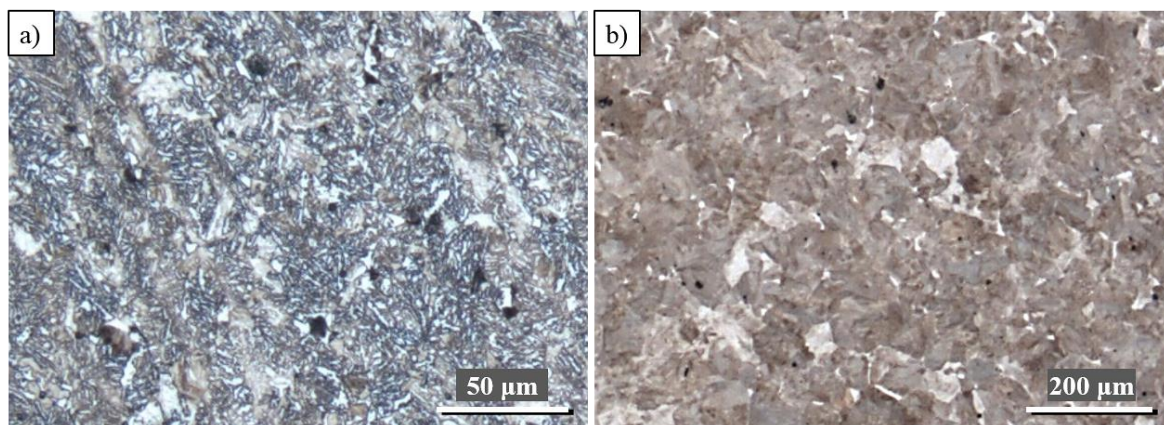


Figure 1. Microstructure of a) initial stock, b) homogenized steel.

Experimental program was divided into two stages. At the first stage, six QP process sequences were designed in order to describe the influence of partitioning temperature on evolution of the microstructure and their mechanical properties. The specimens were processed in a furnace without protective atmosphere. Austenitization at 950 °C for 20 minutes and quenching to the salt bath of 200 °C were applied. Partitioning at various temperatures (PT - 250, 300 and 320 °C) and holding times (15, 30 minutes) followed (figure 2). These parameters were chosen on the basis of results of previous experiments [8, 11]. The second stage of the experiment was focused on the long holding

times (up to 72 hours) applied at the partitioning temperature of 320 °C (figure 2) in order to study diffusion processes and their influence on the mechanical properties of the steel.

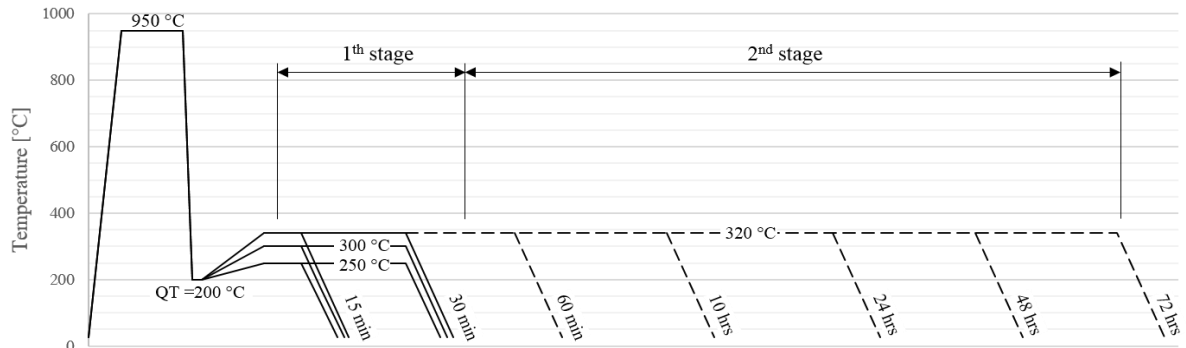


Figure 2. HT sequences of first and second stage of the experiment.

After HT microstructure was examined using light optical and scanning electron microscopy and its hardness was measured. X-ray diffraction phase analysis was carried out using AXS Bruker D8 Discover diffractometer equipped with HI-STAR detector and a cobalt cathode ($\lambda_{K\alpha} = 0.1790307$ nm). Measurements were performed in the center of metallographic sample in a range of diffraction angles 2θ from 25 to 105°. To determine mechanical properties static tensile test on the round bar specimens 5.0 mm in diameter and a gauge length of 25 mm and Charpy V-notch test on specimens 55x10x10 mm was carried out. The tests were performed on three samples. The mean values as well as standard deviations were calculated from the results.

3 Results and discussion

Metallographic analysis revealed martensitic microstructures with small amounts of bainite same in all the specimens (figure 3). No specimen showed signs of extensive precipitation of carbides.

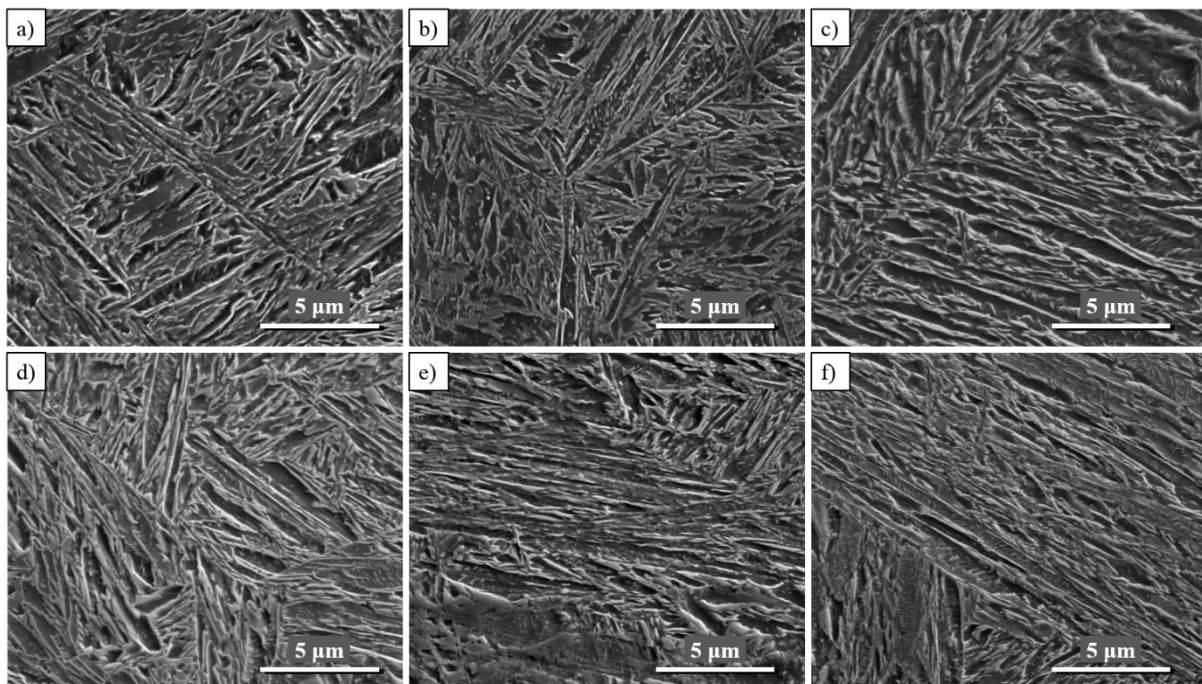


Figure 3. Martensitic/bainitic microstructures after partitioning at: a) 250 °C for 15 min, b) 300 °C for 15 min, c) 320 °C for 15 min, d) 320 °C for 60 min, e) 320 °C for 24 h, f) 320 °C for 72 h.

Any coarse RA austenite particles were not observed on SEM micrographs. Therefore, with respect to high content of RA (determined by X-ray diffraction analyzes), the film RA on grain boundaries can be supposed. The thickness of these films may be around tens of nanometers.

3.1 Effect of partitioning temperature (short holding times)

Using the X-ray diffraction analyzes the RA content of selected specimens were measured. The specimens partitioned at the temperatures of 250 °C and 300 °C showed lower RA content - 11% and 10.5 %, respectively (figure 4). An increase of the RA content was observed at a temperature of 320 °C (12.5% for 15 minute hold; 13% for 30 minute dwell). With respect to not very large measurement differences it can be assumed that the portion of RA only slightly increased with increasing PT.

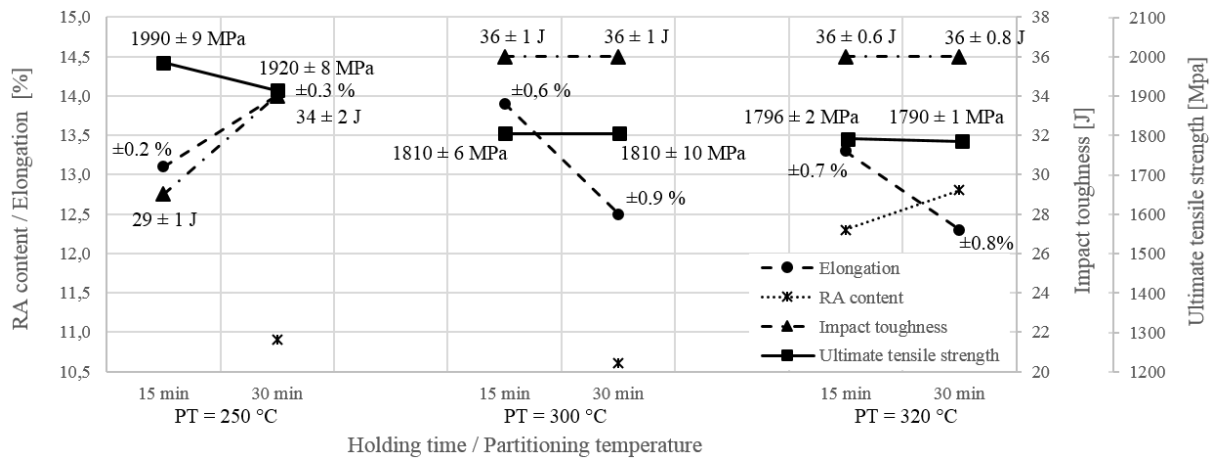


Figure 4. The UTS, elongation, impact toughness and RA content vs. PT and holding time.

A similar tendency was also observed in the results of Charpy impact tests. The specimens partitioned at 250 °C showed lowest impact toughness and at the same time an increasing tendency along with the increasing in holding times - 29 J and 34 J for 15 min and 30 minute dwell, respectively. Partitioning at the temperatures of 300 °C and 320 °C provided higher impact toughness: 36 J equally for both temperatures and holding times (figure 4). This increase can be attributed both, to a higher portion of RA and to a higher stage of martensite decomposition (higher intensity of diffusion processes). This assuming can be further supported by decrease of the UTS from 1990 MPa at the PT 250°C below 1800 MPa at the PT of 300 °C and 320 °C (figure 4). A rather inconsistent trend of elongation is due to small differences in measured values and relatively large standard deviations not reliable.

3.2 Effect of long holding times (partitioning temperature 320 °C)

A specific course of RA content (and also impact toughness and elongation) values was observed in these specimens. At first the values decreased with increasing time of partitioning but at very long dwells they slightly raised again (figure 5). The course of impact toughness corresponded quite well with the RA content evolution. The elongation measurement results were somewhat worse (for its relatively small range of measured values and quite a large standard deviations). The maximum values were observed at the holding times of 15 and 30 minutes: RA content above 12%, impact toughness 36 J and elongation above 12 %. During longer dwells on PT all these characteristics decreased quite rapidly. The RA content was reduced down to 11% beyond the holding time of 10 h. Such a long dwell at 320 °C probably caused exceeding of carbide nucleation energy limit which had been artificially increased by special alloying. It would enable diffusion of carbon from saturated stabilized RA to nearby carbides and hence the decomposition of some portion of RA.

The values of impact toughness and elongation also decreased down to 28 J and 12 % after 24 and 10 hours, respectively. However, even longer partitioning times caused again an increase up to 31 J and above 13%, respectively. The primary decline of ductility correlate quite well with the course of RA content and shows clearly the relation of film morphology RA content and the ductility of the steel. The loose of ductility can be also caused by rod-like morphology of carbides that could precipitated on martensite lathes boundaries. With even longer hold on the PT coarsening of the carbides and transformation into their globular form as well as further recovering and starting recrystallization could occur. This would explain the slow increase of elongation and impact toughness at the holds longer than 24 hours. The UTS was only slightly affected by the change of holding time. Gradual decrease from the 1800 MPa (for 15 minutes hold on PT) to 1740 MPa (for 72 hours hold) was observed.

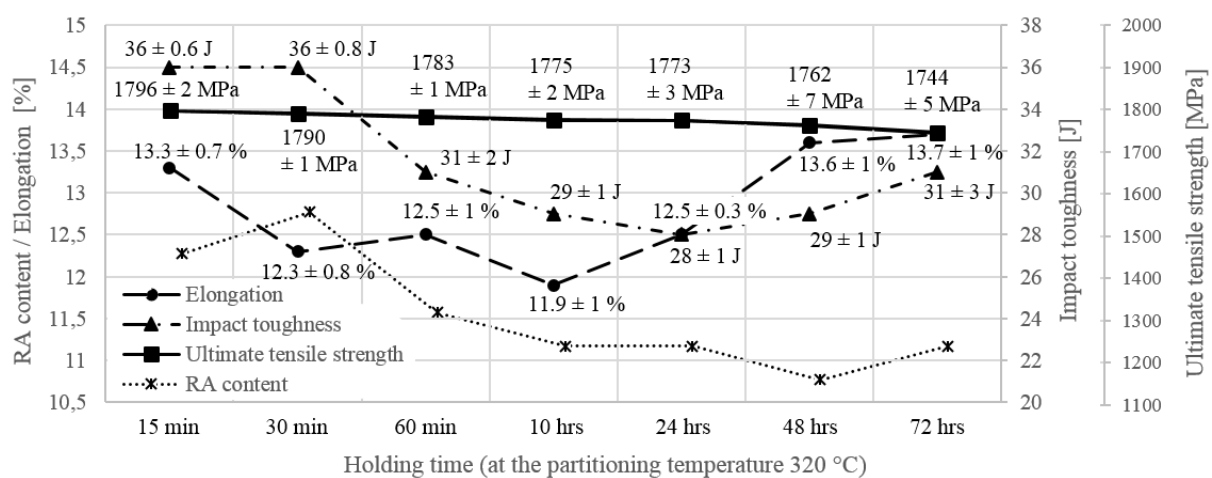


Figure 5. The UTS, elongation, impact toughness and RA content vs. holding time at PT 320 °C.

4 Conclusion

Described experiment dealt with the QP process of high-strength steel alloyed with 1.3 % chromium and 2.03 % silicon. It especially focused on the effect of higher partitioning temperatures (PT) and long holds and their impact to the microstructure and mechanical properties. The main task was to investigate whether the microstructures with even higher retained austenite (RA) content and so higher ductility and toughness could be obtained. Higher PT (300 and 320 °C) examined in the first stage of experiment resulted in only little contribution to the impact toughness - from 34 J to 36 J (30 min hold) but at the same time it caused a decrease in ultimate tensile strength (UTS) from 1990 to values around 1800 MPa. At this stage prolonged hold (especially at lower PT) appeared to be more beneficial. The long holds (at PT of 320 °C) in the second stage of experiment influenced the elongation and impact toughness rather negatively. A decrease in elongation, impact toughness and also the RA content at holding times longer than 30 min together with only gradually declining UTS indicated slow start of precipitation and following partial decomposition of RA. Holds longer than 48 hours resulted in slight increase in elongation and impact toughness again, and further slow decrease of UTS. The recovery and recrystallization processes obviously played the main role here. To confirm these theory further analyzes employing e.g. transition electron microscopy and detail study of the microstructure will follow in the next step.

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