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Accelerated Carbide Spheroidisation and Refinement in Spring Steel 54SiCr6

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Abstract. Reduction of processing time is the goal of every business. In some cases, speeding up manufacturing processes is impossible or takes place at the expense of quality. The purpose of soft annealing is to obtain globular carbides in the microstructure. However, it is one of the most time-consuming heat treatment operations. Even its optimised versions require several hours of time. „Accelerated Carbide Spheroidisation and Refinement" (ASR) is an alternative process which can be performed using induction heating or thermomechanical treatment. It only takes several minutes to complete and has a beneficial effect on resultant mechanical properties. In this experiment, the impact of various microstructures in 54SiCr6 spring steel on hardening behaviour was explored. For this purpose, specimens with two different microstructures containing spheroidised carbides were produced by ASR accelerated annealing and by conventional soft annealing. Another initial microstructure, with lamellar pearlite, was obtained by hot rolling. It was found to have a certain effect as well. Quenched and tempered microstructures were observed using electron microscopy (SEM). Their mechanical properties were measured. The effects of various initial microstructures on resultant mechanical properties after cryogenic treatment were studied as well.

1 Introduction

Soft annealing inserted prior to hardening is an important stage in the treatment of springs. Parts from spring steels are mostly subjected to alternating cyclic stresses and relaxation, which is why manufacturers strive to improve their yield strength and ultimate strength while maintaining their ductility [1]. Accelerated Spheroidisation and Refinement (ASR) is one way to produce spheroidised microstructure [2]. The classical method is soft annealing, which is lengthy due to diffusion processes involved. The material is held near the A_{c1}. Where shorter times are required, slow cycling around A_{c1} is often used. Diffusion, a slow and long process, is required for carbides to change from the lamellar to the globular form. At long annealing times, the carbides which are already globular undergo Ostwald ripening. Consequently, their particles coarsen and their thermodynamic potential decreases, resulting in lower hardness and strength [3].

The ASR process is much less time-consuming. In its accelerated annealing, it relies on rapid cycling around the A_{c1} temperature. The partial austenitization and transformations accelerate the formation of globular particles [3]. Fully spheroidised microstructure can thus be attained within minutes without coarsening. The result is uniformly-dispersed fine globular carbides.

Morphological distinctions between the products of soft annealing and ASR, particularly the particle size, are reflected in mechanical properties as well [4].

They can mislead those who evaluate the extent of carbide spheroidisation. The reason is that successful spheroidisation is usually defined as 95% of carbides being in the spheroidal form.

However, morphological criteria are rarely set in engineering practice and the only criterion for spheroidisation annealing is the maximal permissible hardness of the treated material. ASR-processed material may meet the morphological criteria but fail to meet the hardness limits imposed on soft-annealed material [5]. In still other cases, the differences between spheroidised microstructures obtained by different methods may affect the kinetics of the subsequent processes, such as quenching. The experiments reported in this paper explored the impact of the initial microstructure of 54SiCr6 spring steel on its mechanical properties and microstructure after quenching and tempering. The initial microstructures either contained globular carbides (cementite) produced by either conventional long-time soft annealing (SA) or accelerated annealing (ASR) – or lamellar cementite obtained by hot rolling (HR). In some sequences, cryogenic treatment was performed between the quenching and tempering operations. The motivation was to find whether it reduces the effects that the initial microstructure may have or whether differences between mechanical properties will be retained [6].

2 Materials and methods

The experimental steel was the 54SiCr6 grade, which is used widely for the manufacture of springs. The steel was made at COMTES FHT a.s. The chemical composition of the steel is given in table 1. It was cast in a vacuum induction furnace, then forged, hot-rolled, and air-cooled after rolling.

Table 1. Chemical composition of the experimental steel 54SiCr6.

Element	C	Si	Mn	Cr	Mo	Cu	P	S	Fe
wt. %	0.57	1.51	0.68	0.75	0.03	0.01	0.008	0.003	bal.

Carbide spheroidisation was carried out using two different sequences. The accelerated spheroidisation process (ASR) was completed using medium-frequency (fmax=17 kHz) induction heating equipment with a maximum power of 24 kW. The specimens were 20 mm-diameter bars with 130 mm length. Temperature was measured with a thermocouple welded onto the specimen. The ASR sequence comprised of three thermal cycles with follows regime: heating at 19°C/s to 820°C, holding for 15 seconds, cooling in still air (at approx. 1.5°C/s) to 725°C and holding for 5 minutes. This was followed by cooling in still air to 600 °C. The second cycle was identical. In the third cycle, the hold at 725°C was extended to 10 minutes. The final step was cooling in still air to the ambient temperature. The sequence is plotted in figure 1. The total duration of the spheroidisation stage was approximately 30 minutes.

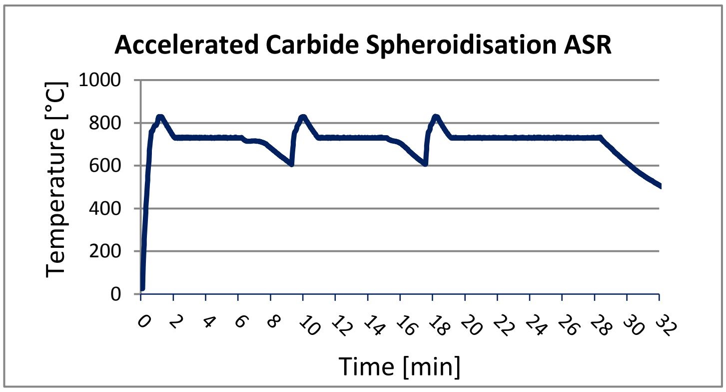

Figure 1. ASR induction heat treatment for the 54SiCr6 grade

Soft annealing (SA) was performed in an electrical air furnace. Specimens had the same size as those for the ASR sequence. In the SA sequence, the material was held just above the A_{c1}. The sequence was as follows: heating at 10°C/min to 720°C, then at 15°C/hour to 770°C, holding for 5 hours, furnace cooling at 5°C/hour to 720°C, then at 25°C/hour to 650°C, free cooling in the furnace to 400°C and then cooling in still air. The total time until removal from the furnace was approximately 27 hours. With this steel, the slow cooling at 5°C/hour in the soft annealing sequence and the isothermal hold at 725°C in the ASR sequence were necessary for preventing formation of new cementite lamellae.

Quenching and tempering was carried out in electrical air furnaces. Quenching temperatures were in the range from 810 to 890°C and the soaking time was 20 minutes. The quenching medium was oil. Tempering at 400°C/2 hours was followed by cooling in air.
Cryogenic treatment (CT) was performed in a cryogenic box. The sequence comprised cooling to -160°C over 1 hour, holding for 20 hours and reheating to 20°C in 1 hour.
The effects of the differences between initial microstructures and heat treating sequences were studied in the processed specimens using scanning electron microscopy (SEM) and mechanical testing, which involved measurement of hardness and tensile tests.

3 Results and discussion

3.1 Initial state of material

The initial microstructure after hot rolling (HR) was pearlite with hardness of 290±7 HV10. The microstructure was homogeneous, composed of a ferritic matrix and lamellar cementite (figure 2). Both spheroidised microstructures, post-SA and post-ASR, contained globular cementite particles in a ferritic matrix. In addition, there were some scarce cementite lamellae in them. Soft annealing (figure 3) produced large globular particles up to 2 µm in size. By contrast, the ASR sequence (figure 4) led to densely-dispersed small globular cementite particles (0.5 µm max.) in a ferritic matrix. The hardness after long-time soft annealing was 207 ± 1 HV10. After ASR, it was higher by 17 points, owing to a finer microstructure. Both values meet the delivery specifications for 54SiCr6 steel annealed to obtain globular cementite.

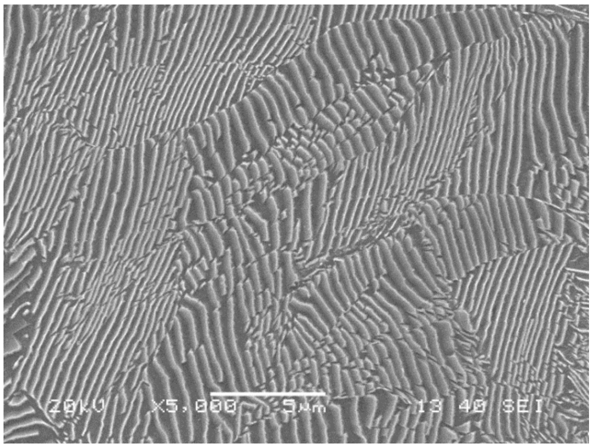

Figure 2. Microstructure after hot rolling (HR)

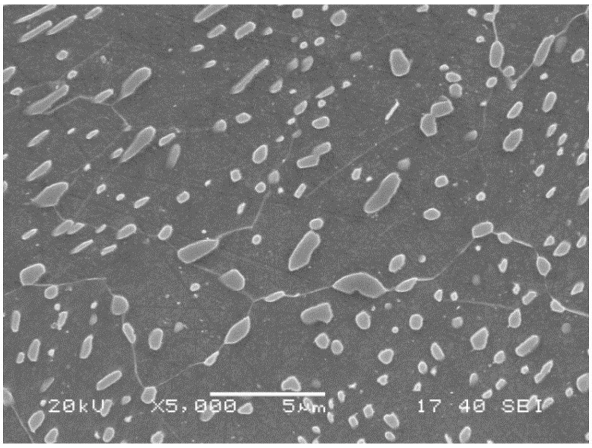

Figure 3. Microstructure after soft annealing (SA), 207 HV10

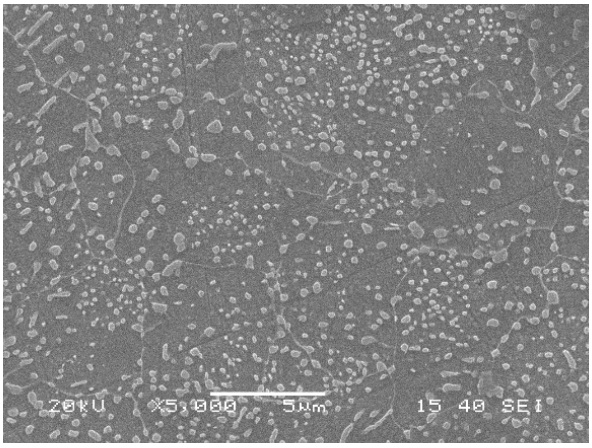

Figure 4. Microstructure after accelerated annealing (ASR), 224 HV10

3.2 Quenching and tempering

Quenching was performed from 810, 830, 850, 870 and 890°C. The austenitizing time was 20 minutes. Soft-annealed specimens, which were quenched from 810, 830 and 850°C, contained, in addition to martensite and undissolved cementite, some ferrite grains. With higher quenching temperatures, the matrix was fully martensitic. The amount of undissolved cementite decreased with increasing quenching temperatures. Yet, some undissolved globular cementite was found in the material upon quenching from 890°C. In the soft-annealed material, the largest undissolved cementite particles had a size of approx. 1000 nm. They remained in the material even after quenching from higher temperatures (figure 5) because the smaller ones had dissolved more quickly.

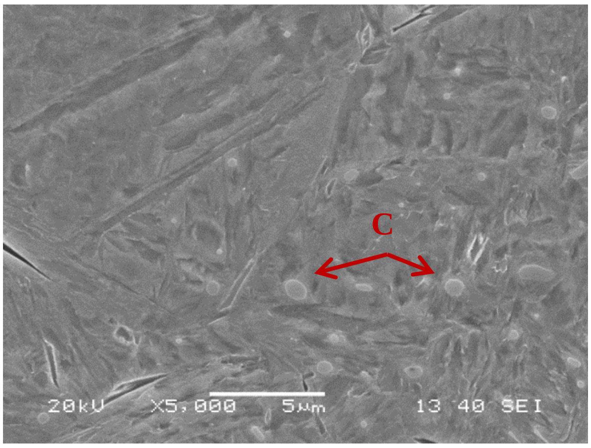

Figure 5. Quenched sample SA 870°C, 742 HV10, (C – undissolved cementite)

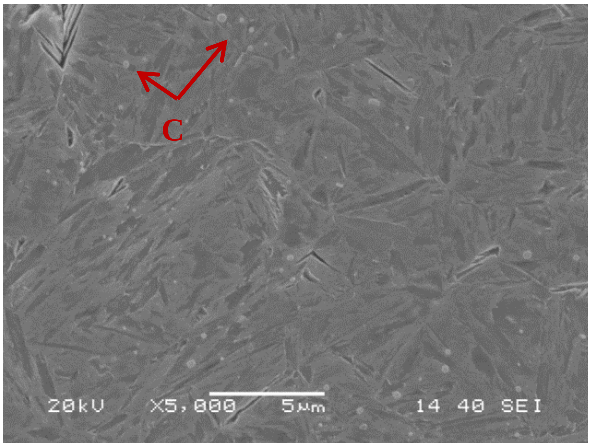

Figure 6. Quenched sample ASR 830°C, 756 HV10, (C – undissolved cementite)

Quenched ASR specimens only contained ferrite after quenching from 810°C. Fine globular cementite dissolved much more readily. After ASR, the maximum size of undissolved cementite particles was 300 nm (figure 6). Following the quench from 870°C, undissolved cementite was rare. After quenching 890°C, it was dissolved completely.

Quenching of pearlitic specimens obtained by hot rolling had similar results. Lamellar cementite austenitized even faster than the globular structure after ASR. Some remaining ferrite grains were also found after quenching from 810°C. Their amount was smaller than in the other cases. The largest undissolved cementite particles in the martensitic matrix had a size of 200 nm. A summary of the austenitizing experiment is given in table 2.

Table 2. Austenitizing (the highest T_{quench} upon which 54SiCr6 steel still contains a particular phase)

	Soft annealing (SA)	Accelerated annealing (ASR)	Hot rolling (HR)
Ferrite	850 °C	810 °C	810 °C
Undissolved cementite	890 °C	870 °C	870 °C

During tempering, cementite precipitated within and along martensite plates. Apart from the size of undissolved cementite particles, no differences were found between tempered microstructures obtained in specimens in different initial conditions.

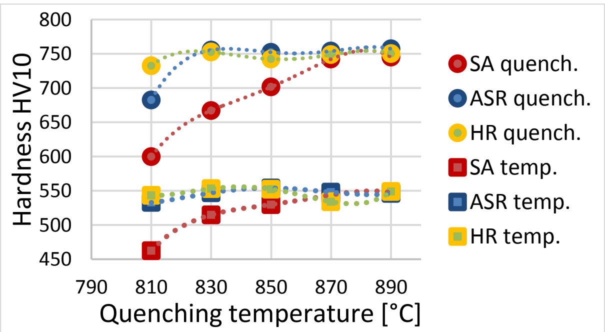

Figure 7. Hardness after quenching and tempering for 54SiCr6

Hardness is in agreement with the microstructure, as seen in figure 7. When the extent of austenitization proved insufficient (ferrite in the microstructure) in the specimens, lower hardness levels were found. Hot-rolled and ASR-treated specimens proved well-hardened even upon quenching from 830°C but the soft annealed ones required 870–890°C. The dissolution of remaining cementite particles has not led to a major increase in overall hardness. After transformation of ferrite to austenite, hardness appeared almost constant.

Maximum hardnesses were similar for all initial conditions. When quenching was appropriate, the hardness level was around 750 HV10 and the hardness upon tempering was approx. 550 HV10.

In the ASR-treated material, carbides dissolved almost as quickly as lamellar pearlite. In soft-annealed material, austenitizing was retarded, as cementite was present in large globules whose neighbourhood was depleted of carbon. Hence, slower dissolution of large particles and the need for long-distance diffusion of carbon poses greater demands on austenitizing prior to quenching. The desired properties were only attained once the quenching temperature had been raised to 870°C or higher.

The ASR process therefore combines the advantages of soft-annealed initial condition and lamellar pearlite. Thanks to fine carbides, austenitizing is rapid and high quenching temperatures are not needed. In 54SiCr6 steel, the difference in quenching temperatures is 40°C. Globular morphology of cementite guarantees the reduction in hardness which is the purpose of soft annealing.

In cryogenically-treated (CT) material, the microstructure was examined and hardness was measured as well. The sequences involving SA and quenching temperature of 870°C and ASR a HR with the quenching temperature of 830°C were selected for this purpose. However, no differences from non-CT tempered microstructures were found in these specimens using scanning electron microscopy (SEM). Despite that, their hardness levels were higher. Upon tempering, the increase was approx. 20 HV10. Hardness values after cryogenic treatment are shown in table 3.

3.3 Tensile tests

Room-temperature tensile tests were in accordance with ČSN EN ISO 6892-1. The gauge sections had a size of Ø10×60 mm. The thread in the head was M16 and the total length of the specimens was 110 mm. Specimens prepared by sequences with quenching temperatures of 830 and 870°C were chosen. They were tested in tempered condition. Results of the tensile tests are given in table 3.

The readings obtained after soft annealing and quenching from 830°C suggested that austenitization had been insufficient. With 870°C, the yield strength was 1635 MPa and the ultimate strength reached 1834 MPa. After ASR (830°C), yield strength and ultimate strength were higher by 66 MPa and 43 MPa, respectively. There was no appreciable difference between the conditions obtained by quenching from 830°C and 870°C after ASR. The ASR material quenched from 870°C had the highest elongation and reduction of area, despite its yield strength and ultimate strength being higher than in the SA material. The hot-rolled material quenched from 830°C showed an even higher yield strength and ultimate strength (by 18 MPa and 31 MPa, respectively) than the ASR material. However, its elongation and reduction of area was similar to the values upon SA. After quenching from 870°C, mechanical properties were slightly lower. This means that the microstructure with lamellar pearlite (HR) began to coarsen at higher quenching temperatures.

Table 3. Final results of tensile tests and hardness

Heat treatment	$R_{p0.2}$ [MPa]	R_m [MPa]	A_g [%]	A_5 [%]	Z [%]	HV10
SA 830 °C	1592 ± 4	1766 ± 5	3.1 ± 0.1	8.1 ± 1.0	15.3 ± 2.5	515 ± 3
SA 870 °C	1635 ± 7	1834 ± 12	3.4 ± 0.1	8.9 ± 0.2	16.5 ± 0.5	544 ± 2
SA 870 °C + CT	**1764 ± 23**	**1943 ± 19**	**2.6 ± 0.1**	**8.0 ± 0.1**	**30.5 ± 0.6**	**566 ± 2**
ASR 830 °C	1701 ± 17	1877 ± 16	3.1 ± 0.1	8.1 ± 0.9	16.4 ± 2.7	547 ± 2
ASR 830 °C + CT	**1786 ± 10**	**1957 ± 9**	**2.5 ± 0.1**	**8.4 ± 0.1**	**35.8 ± 0.6**	**567 ± 4**
ASR 870 °C	1694 ± 22	1875 ± 13	3.1 ± 0.1	9.7 ± 0.2	22.0 ± 0.5	548 ± 6
HR 830 °C	1719 ± 3	1908 ± 7	3.1 ± 0.1	8.9 ± 0.3	17.5 ± 1.1	553 ± 2
HR 830 °C + CT	**1771 ± 11**	**1951 ± 12**	**2.4 ± 0.1**	**7.0 ± 1.1**	**28.0 ± 3.7**	**575 ± 3**
HR 870 °C	1666 ± 9	1867 ± 6	3.2 ± 0.2	8.6 ± 0.8	15.9 ± 0.4	534 ± 7

Cryogenic treatment led to higher ultimate strengths and yield strengths. The increases in yield strength were 129 MPa, 85 MPa and approx. 52 MPa in materials after SA, ASR and HR, respectively. To some extent, they compensated for the differences in strength characteristics (table 3). After ASR, the ultimate strength and yield strength were slightly higher than in other conditions, including the hot-rolled one. Greater differences remained in elongation and reduction of area. The best values were found after ASR: reduction of area was 35.8%. Following soft annealing, reduction of area was 30.5%. After hardening of the hot-rolled material with lamellar pearlite, the reduction of area was no more than 28%.

The quenched and tempered ASR material probably had finer austenite grain, which was reflected in its reduction of area. This was made even more conspicuous by cryogenic treatment. Differences between strength characteristics were reduced, probably by precipitation of very fine eta carbides. Nevertheless, the smaller prior austenite grains led to better performance under triaxial load, as seen in increased reduction of area after ASR.

4 Conclusion

Effects of three different initial microstructures on mechanical properties and microstructures upon quenching, tempering and cryogenic treatment were studied in 54SiCr6 spring steel. The initial microstructures included lamellar cementite obtained by hot rolling (HR, 290 HV10), fine globular cementite after Accelerated Spheroidisation and Refinement (ASR, 224 HV10) and coarser globular cementite produced by soft annealing (SA, 207 HV10).

Austenitizing was fastest in lamellar pearlite obtained by hot rolling. It was almost matched by the rate of dissolution of spheroidised carbides upon ASR. Austenitizing was much slower in soft-annealed material where the appropriate quenching temperature proved to be 40°C higher than for the other microstructures. Maximum hardnesses upon quenching were the same for all three initial conditions. Dissolution of remaining carbides in austenite has not led to an appreciable increase in hardness appreciably.

The ASR material showed higher tensile properties than the SA one. Quenching from 870°C led to higher yield strength and ultimate strength, by 59 MPa and 41 MPa, respectively. At the same time, elongation and reduction of area were higher by 0.8 points and 5.5 points, respectively. Quenched and tempered HR material (830°C) had slightly higher strength characteristics than the ASR material. However, its elongation and reduction of area was lower, similar to SA.

Cryogenic treatment (CT) led to improved mechanical properties, while reducing the differences between yield strengths and ultimate strengths of materials with different initial conditions. This was not the case with elongation and reduction of area which still reflected the microstructures before quenching. The highest reduction of area was found in the ASR material: 35.8%. With SA, it was 5.3 points less and with HR even 7.8 points less.

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