New approach to heat treatment of spring steel

Kotous J Salvetr P Procházka R
COMTES FHT a.s., Prumyslova 995, 334 41, Dobrany, Czech Republic
jakub.kotous@comtesfht.cz

Abstract. In the 54SiCr6 spring steel, a spheroidized structure was achieved by two processing methods: conventional soft annealing above the temperature A1 in a furnace and accelerated ASR annealing by an induction heating equipment. The ASR technology is much faster, even for materials with a worse tendency to the spheroidisation, such as steels with higher silicon content. Thanks to this, a finer structure is obtained. This is the basis of other advantages for the subsequent hardening. Quenching can be done at lower temperatures, higher yield and tensile strength is obtained while maintaining same or improved ductility and contraction. This also results in influencing of fatigue behavior.

1 Introduction
Quenching temperature and the time optimizing is an important state to achieving good resultant properties of products. An economic point of view is important too. The effort is to have temperature and time as lowest as possible. In this case, the optimal initial structure is very important [1-3]. The finer the structure, the faster the austenitization. The quenched structures achieved the fastest dissolution into austenite but it isn’t suitable for machining. The soft annealing is maybe the last in effective solution [4]. The different accelerated annealing in furnaces [5][6] are better option but the fastest spheroidisation annealing is achieved by an induction equipment [7]. The Accelerated Spheroidisation and Refinement (ASR) is based on fast-cycling about the temperature A1. It produced very fine globular cementite particles. The ASR treatment is able to achieve by thermo-mechanical processing too [8]. So it’s easy to integrate into hot forming and save more time. It is necessary to achieve at least the same properties after ASR as after conventional soft annealing to ensure applicability in the spring production. In this article, the influence of soft annealing (SA) and accelerated spheroidizing (ASR) on the spring steel 54SiCr6 grade was studied. Significant study values were resultant properties after hardening which were determined by the tensile tests. Important monitored attribute of spring steel is the fatigue limit therefore the fatigue behavior was also observed.

2 Materials and methods
The experimental material was the steel 54SiCr6 grade, which is used commonly for the spring manufacture. The steel was made at COMTES FHT a.s. It was casted in a vacuum induction furnace, forged, hot rolled and air cooled. Then follows normalizing annealing. The chemical composition is given in table 1. The samples were bars with 20 mm in diameter and length of 130 mm.

Table 1. The 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. |
The carbide spheroidisation was carried out using two different sequences. The process Accelerated Spheroidisation and Refinement (ASR) was completed using medium-frequency induction heating equipment. Induction heating was carried in a coil with 35 mm in inner diameter, 150 mm length and 14 threads. The temperature was measured with a welded thermocouple. The ASR sequence comprised of three cycles with follows regime: 820 °C/15 s + 725 °C/5 min/ air cooling to 600 °C. The second cycle was identical. In the third cycle, the hold at 725°C was extended to 10 minutes. The sequence is plotted in figure 1.

The soft annealing (SA) was performed in an electrical air furnace. The material was held just above the A_c1. The sequence was as follows: heating at 600 °C/hour 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. The total time until the removal of samples from the furnace was 27 hours.

The slow cooling at 5 °C/hour in the SA sequence and the isothermal holds at 725 °C in the ASR sequence were necessary for preventing formation of new cementite lamellae in this high silicon content steel.

The quenching and tempering (QT) were carried out in electrical air furnaces. Quenching temperatures were in the range from 810 to 910 °C, the soaking time 20 minutes, medium oil. Tempering was at 400 °C/2 hours/ air for all regime.

The evaluation of different spheroidisation was mediated by observing microstructures using a scanning electron microscopy (SEM), hardness measuring, tensile testing and fatigue testing of the selected regime.

3 Results

3.1 Initial state of material
Both spheroidized microstructures (post-SA and post-ASR) contained globular cementite particles in a ferritic matrix. Small cementite lamellae were rare observed. The ASR process led to densely-dispersed small globular cementite particles (max. 0.5 μm) in the ferritic matrix (figure 2). The soft annealing produced large globular particles up to 2 μm in size (figure 3).

Figure 2. The microstructure after accelerated annealing (ASR)
Figure 3. The microstructure after soft annealing (SA)
The hardness after the ASR was 224 ± 5 HV10. It was lower for SA - 207 ± 1 HV10. Both values meet the delivery specifications of spheroidized annealing of 54SiCr6 steel.

Table 2. The mechanical properties of initial states

| Heat treatment                      | Rp0.2 [MPa] | Rm [MPa] | As [%]  | As [%]  | Z [%]  | HV10  |
|------------------------------------|-------------|----------|---------|---------|--------|-------|
| ASR                                | 545 ± 11    | 803 ± 5  | 13.1 ± 0.3 | 24.0 ± 0.2 | 62.8 ± 0.1 | 224 ± 5 |
| SA                                 | 356 ± 1     | 688 ± 2  | 16.5 ± 0.2 | 27.9 ± 0.3 | 52.5 ± 0.1 | 207 ± 1 |
| Initial state with lamella cementite | 542 ± 1     | 987 ± 2  | 9.3 ± 0.1 | 16.7 ± 0.2 | 38.8 ± 0.6 | 290 ± 7 |

Big differences were in the results of tensile test, see table 2. The state after ASR had higher yield and tensile test with lower elongation. Surprisingly, the area reduction was better than after SA. The initial state before spheroidisation with lamellar pearlite was also included in results for interest.

3.2 Quenching and tempering

The quenching was performed in the range of temperatures from 810 to 910 °C. The soft-annealed specimens, which were quenched from 810, 830, and 850 °C, contained ferrite in the microstructure. With higher quenching temperatures, fully martensitic with undissolved cementite was observed (figure 4 and 5). The amount of undissolved cementite decreased with increasing quenching temperatures. Yet, undissolved globular cementite was found in the martensitic matrix after quenching from 910 °C (figure 5). The largest undissolved cementite particles had a size about 1000 nm. They remained in the material even after quenching from the highest temperature because they would need much more time for dissolving.

Figure 4. The quenched sample ASR 830 °C   Figure 5. The quenched sample SA 910 °C

The ferrite in the quenched ASR specimens was observed only after quenching from 810 °C. Fine globular cementite dissolved quickly. The maximal size of the undissolved cementite particles was 300 nm (figure 4). Following quenching from 870°C, the undissolved cementite was rare to find. During the tempering, cementite precipitated within and along martensitic plates. Apart from the size of undissolved cementite particles, no differences were found between tempered microstructures obtained after the ASR and the SA.
The hardness results are in agreement with the microstructure, see table 3. When the ferrite is completely transformed into austenite, the hardness reached maximum values. It wasn’t increase with dissolving rest of cementite. It was about 750 HV10 after quenching and 550 HV10 after tempering for both initial states. The dissolution of remaining cementite particles has not led to a major increase in overall hardness.

### Table 3. Hardness HV10

| Heat treatment | Quenching temperature [°C] |
|---------------|-----------------------------|
|               | 810 | 830 | 850 | 870 | 890 | 910 |
| Quenched      |     |     |     |     |     |     |
| ASR           | 683 ± 2 | 756 ± 4 | 752 ± 5 | 754 ± 6 | 757 ± 4 | - |
| SA            | 600 ± 11 | 667 ± 7 | 702 ± 11 | 742 ± 4 | 745 ± 4 | 746 ± 8 |
| Tempered      |     |     |     |     |     |     |
| ASR           | 533 ± 1 | 547 ± 2 | 554 ± 1 | 548 ± 6 | 546 ± 4 | - |
| SA            | 462 ± 2 | 515 ± 3 | 530 ± 4 | 544 ± 2 | 547 ± 4 | 534 ± 5 |

#### 3.3 Tensile tests

Room-temperature tensile tests were performed in accordance with ČSN EN ISO 6892-1. The gauge sections had a size of Ø10×60 mm. The specimens were tested in tempered condition. Results of the tensile tests are given in table 4.

### Table 4. Results of tensile tests

| Heat treatment | \(R_{p0.2}\) [MPa] | \(R_m\) [MPa] | \(A_g\) [%] | \(A_s\) [%] | \(Z\) [%] |
|---------------|-------------------|---------------|-------------|-------------|---------|
| ASR 830       | 1696 ± 19         | 1873 ± 13     | 3,1 ± 0,1   | 8,8 ± 0,1   | 34,9 ± 0,4 |
| ASR 850       | 1686 ± 11         | 1881 ± 1      | 2,9 ± 0,1   | 9,3 ± 0,1   | 38,8 ± 0,9 |
| ASR 870       | 1684 ± 19         | 1863 ± 10     | 3,1 ± 0,1   | 9,7 ± 0,2   | 41,7 ± 1,1 |
| SA 870 °C     | 1634 ± 7          | 1832 ± 12     | 3,4 ± 0,1   | 8,9 ± 0,2   | 31,4 ± 0,9 |
| SA 890 °C     | 1656 ± 10         | 1847 ± 1      | 3,2 ± 0,1   | 9,6 ± 0,2   | 34,0 ± 1,4 |
| SA 910 °C     | 1644 ± 18         | 1835 ± 16     | 3,2 ± 0,1   | 9,9 ± 0,1   | 35,2 ± 1,0 |

The states which were fully austenitized were tested. Although the hardness was the same, the dissolving rest of cementite carbides influences the mechanical properties. The results with both spheroidisation annealing had the same trend in elongation \(A_s\) and area reduction \(Z\). The trend was increasing with higher temperature and lowering content of undissolved cementite. The values of elongation were similar. However, the ASR achieved a higher area reduction than the SA. In the point of yield and ultimate strength, the values are similar for the individually initial state in the tracked range of quenching temperatures but all of them are higher after the ASR than the SA. Differences were about 30-40 MPa.

#### 3.4 Fatigue testing

The high-cycle fatigue testing was carried out on the magneto-resonance machine Vibrophore Rumul at the room temperature. The specimens were tested in the tempered state and there were compared between the ASR quenched from 830 °C and the SA quenched from 870 °C. The gauge sections had a size of Ø5 mm with a radius 64 mm. Loading was alternating with asymmetry cycle \(R = -1\). The results represent the determination of the fatigue limit and the Wohler curves, see figure 6.
The results had a large scatter. The fatigue limit of the SA regime was 900 MPa. The ASR fatigue limit was 925 MPa. This result was expected thanks to the fact that ASR achieved higher ultimate strength. The area of timed fatigue limit after the ASR looks a little better too. It last more cycles at the same stress amplitude.

4 Discussion
The carbides after the ASR dissolved very fast almost as quickly as lamellar pearlite [4]. In the SA material, the austenitizing was retarded, as cementite was present in the large globules whose neighborhood was depleted of carbon. Hence, slower dissolution of the large particles and the long-distance diffusion of carbon poses greater demands on the austenitizing process before quenching.

The hardened ASR material had probably finer austenite grain, which was reflected in higher strength characteristics with higher area reduction while maintaining good elongation. The grain etching was done without success for quantitative grain size evaluation.

5 Conclusion
The effect of different spheroidisation annealing was observed. The article was focused on the influence of this effect on resultant mechanical properties in 54SiCr6 spring steel. Therefore the metallographic analysis and hardness measuring were supplemented by tensile and fatigue testing in the final tempered state.

Two initial states were achieved by the Accelerated Spheroidisation and Refinement (ASR) and the soft annealing (SA). The austenitizing was much faster post-ASR than post-SA. In the 54SiCr6 steel, the difference in optimal quenching temperatures is about 40°C. In the case of the SA, the large undissolved cementite particles were observed at the highest quenching temperature. It also leads to different mechanical properties. The ASR treatment improves the yield and ultimate strength and the area reduction while the elongation and maximal values of hardness remain the same as for that SA samples. The finer carbides after the ASR positively affected resultant fatigue behavior. The fatigue limit was higher by 25 MPa and Wohler curve looked more favorable.
In the conclusion, the ASR annealing combines the advantages of the soft annealing and the state without annealing (lamellar pearlite). The globular morphology of cementite guarantees the reduction in hardness which is the purpose of the spheroidisation annealing. Thanks to the fine carbides, the austenitizing is rapid and lower quenching temperatures are sufficient. In addition, it leads to improving mechanical properties.

Acknowledgement
The result was supported from ERDF Research of advanced steels with unique properties, No. CZ02.1.01/0.0/0.0/16_019/0000836.

References
[1] Jirková H., Hauserová D., Kučerová L., Mašek B., Energy and Time Saving Low Temperature Thermomechanical Treatment of Low Carbon Plain Steel, Materiali in Technologije/Materials and Technology, 2013, vol. 47/3, pp. 335-9.
[2] Hauserová, D., Dlouhý, J., Nový, Z., Microstructure and Properties of Hardened 100CrMnSi6-4 Bearing Steel after Accelerated Carbide Spheroidisation and Long-duration Annealing. In: Bearing Steel Technologies: 10th Volume, Advances in Steel Technologies for Rolling Bearings, STP 1580, 2015, pp. 389-409, ISSN 2160-2050.
[3] Hauserova D, Dlouhy J and Kotous J 2017 Structure Refinement of Spring Steel 51CrV4 after Accelerated Spheroidisation Arch. of Metall. and Mater. vol 62 p 1473-77.
[4] Kotous, J., Dlouhy, J., Nacházelová, D., Influence of accelerated spheroidisation on quenching process and resultant mechanical properties in spring steel 54SiCr6, Materials Science and Engineering, 2019, vol. 461.
[5] Saha A., Mondal D.K., Biswas K., Maity J., Development of high strength ductile hypereutectoid steel by cyclic heat treatment process, Materials Science and Engineering A 2012, vol. 541, pp 204-15.
[6] Mishra S., Mishra A., Show B. K., Maity J., Simultaneous enhancement of ductility and strength in AISI 1080 steel through a typical cyclic heat treatment, Materials Science and Engineering A, 2017 vol 688, pp 262-71.
[7] Hauserová, D., Dlouhý, J., Nový, Z., Effect of Heating Rate on Accelerated Carbide Spheroidisation (ASR) in 100CrMnSi6-4 Bearing Steel, Archives of Metallurgy and Materials, 2014, Vol. 59, pp. 1199-1203, ISSN 1733-3490.
[8] Hauserová D., Dlouhý J., Nový Z., Zrník J., Duchek M., Forming of C45 Steel at Critical Temperature, Procedia Engineering, 2011, vol. 10, pp. 2955-60.