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MODIFICATION OF HEAT TREATMENT PARAMETERS FOR PEARLITE STEEL INTENDED FOR COLD PLASTIC WORKING

One of the problems widely discussed in the relevant literature is the fatigue cracking of pearlite steel subjected to cold plastic working, as well as of that being currently in operation. Studies presented in the work were aimed at selection of the parameters, and mainly the time, of heat treatment operations of pearlite steel enabling obtaining cold drawn wires characterised with better plastic properties and higher fatigue resistance than the products achieved during standard processes. The idea of modification was based at possibility of obtaining steel of the sorbite, which are a mixture of ferrite and dispersion cementite of high degree of coagulation. The structures obtained with this method, as being globular, should be featured with better plastic properties than the lamellar structures created during standard diffusion transformation of austenite. In addition the discussed change was to enable decreasing the total surface of interphase boundaries at which the highest delaminations of structure were observed, regardless of whether the cause of the change resulted from presence of non-metallic inclusions or pile-up of dislocations.

1. INTRODUCTION

Pearlite steels containing from about 0.8 to 0.95% C belong to the group of unalloyed steels of the quality class intended for cold drawing or rolling [1],[2]. They have found application mainly as wires designated for production of steel cord mainly applied for reinforcement of tires (PN-EN 10323:2005 (U)), hoses (PN-EN 10324:2006) or for production of ropes (PN-EN 10264-1:2005) [4]. Their chemical composition as well as mechanical properties should comply with the PN-EN 10323:2005 (U) standard [3],[4]. The mechanical properties, mainly the tensile strength are closely related with percent content of the steel components, in particular from the content of silicon and manganese. In the annealed state the pearlite steels have the highest strength in relation to the remaining unalloyed steels [4]. At the same time the steels are characterised with especially low share of non-metallic inclusions and the limited content of chromium and nickel that affect elongation of the pearlite transformation time [3],[4].

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One of the problems widely discussed in the literature is cracking of pearlite steel subjected to plastic working or that being in operation. The problem is the more serious as it concerns many industrial branches where the steel cord is used, and therefore it has been researched for many years in numerous scientific centres around the world.

The fatigue strength of steel wires highly depends on the metallurgic purity of the material, and especially on the content of sulphur and phosphorus, as well as precipitations of pure metals, chromium, nickel and manganese. The presence of non-metallic inclusions in steel strongly decrease the fatigue strength of wires, as the stress concentrations are created around them resulting in cracking of components [5],[13].

According to the literature data the maximum permissible content of non-metallic inclusions should not exceed the standard No. 2 according to the EN 10247:2007 standard and the GOST 1778-70 standard [8],[9]. Moreover, the non-metallic inclusions, especially including those not plastic, lower the wire ductility making the technological processes difficult. Presence of non-metallic inclusions in steel is a cause of lowering the strain preceding the process of wire cracking, as the material discontinuities created in them are capable of exceeding the critical size of the failure and, as a consequence, the wire cracking takes place [13].

The research conducted by Zelin [21] have shown that as a result of elastic strains preceding the permanent plastic strain of the material, the horizontal micro-delamination of singular cementite lamellae takes place. The changes were observed both, during tension and torsion of the wires. During the growth of the applied force the coalescence of the created micro-delaminations takes place and the crack propagation resulting in decohesion of the whole part.

Sauvage and Ivanisenko [14],[20] have shown instead, that the cause of cracking of pearlite steel subjected to plastic working is segregation of carbide precipitations at phase boundaries. The research have confirmed the theory discussed earlier by Gridnev and Gavriluk [10] according to which, as a result of plastic strain, the atoms of carbon occupy vacancies in the cementite lattice causing increase in concentration of carbon atoms at the ferrite-cementite phase boundary which, as a consequence, results in brittleness of those structures. Both teams have shown the simple relation between the degree of plastic strain and susceptibility of pearlite steel to cracking. It is known that the increase in strain is accompanied with increase in density of vacancies and by that the intensity of carbide precipitations segregation increases at the phase separation boundaries [10],[14].

The structural analysis performed by Izotov [15],[16] and the team have shown at the same time that cracking of cementite lamella follows as a result of dislocation pile-up at pearlite phases separation boundary. As a result of the applied force an edge dislocations movement follows in ferrite precipitations and dislocation of fragments of a crystal takes place in the slip planes, and due to the fact that each ferrite lamella has different crystallographic orientation, the dislocations are intersecting at the cementite lamellae finally initiating by that the microcracks creation. The research has confirmed the earlier literature reports, the works of Langeford, Wilson, Embury and Fisher [17], indicating that during plastic straining of pearlite steel the highest concentration of lattice defects takes just place at the cementite lamellae, which in consequence is a cause of cracking of those structural components.

The different theory explaining the cracking mechanism of cementite lamellae was presented by Languillaume and his team [18]. According to the research of that team, as a result of plastic strain of pearlite the uncontrolled and very strong increase in energy takes place at the contact of both phases that is in the inter-lamella space of pearlite. The observed increase in energy leads to thermodynamic destabilising of cementite resulting in cracking of its lamellae. The results of the research have been repeatedly confirmed by the other research teams including, among others, by Danoix and Sauvage [19],[20].

2. PURPOSE AND METHODOLOGY OF THE RESEARCH

The presented studies were aimed at development of modification of the heat treatment parameters for the pearlite steel which would enable obtaining cold drawn wires characterised with better plastic properties and higher fatigue strength than the products achieved in standard processes. The idea of modification was based at a possibility of obtaining steels of the temper sorbite structure which are a mixture of ferrite and dispersion cementite of high degree of coagulation. The structures obtained with this method, as being globular should be featured with better plastic properties than the lamellar structures which are created in standard diffusion transformations of austenite. The discussed change was in addition to enable lowering of the total surface of interphase boundaries at which the highest delaminations of structure was observed regardless of whether the cause of the change resulted from presence of non-metallic inclusions or pile-up of dislocations.

The object of the research was pearlite steel of chemical composition and mechanical properties in compliance with the PN-EN 10323:2005 (U) standard. Specimens for the tests were prepared in the form of steel wires obtained after subsequent stages of cold plastic working involving drawing from the diameter of 3.15 mm to 0.8 mm. The last stage of the specimens preparation was hardening from the temperature of 770ºC and tempering in the temperature of 500°C in times of 1, 2 and 3 hours in sequence (see table 1). Temperatures of the heat treatment process were selected at the base of earlier studies of the team and they warranted obtaining the structure of sorbite, being a mixture of ferrite and coagulated cementite of high dispersion [7],[11],[12].

Table 1. Tested specimens

SPECIMEN	MATERIAL CONDITION
No. 1	MATERIAL IN AS SUPPLIED CONDITION
No. 2	SPECIMEN No. 1 AFTER HARDENING AND TEMPERING AT 500°C/1 h
No. 3	SPECIMEN No. 1 AFTER HARDENING AND TEMPERING AT 500°C/2 h
No. 4	SPECIMEN No. 1 AFTER HARDENING AND TEMPERING AT 500°C/3 h

For evaluation of microstructure of the tested steel the scanning electron microscope Phenom G2 was applied, and the observations in the etched material state were performed

with magnifications from the 1000x ÷ 10 000x range. Detailed microscopic observations were performed also with the use of transmission electron microscopy method. The specimens were prepared in the form of thin films of the 1 5x2 mm size, and the preparation involved mechanical grinding and polishing up to the thickness of 70 μm, as well as electrolytic etching (in the solution of 25% HNO3 and 75% methyl alcohol) with application of the TenuPol equipment. For observation the Hitachi H-800 transmission microscope with maximum accelerating voltage of 200 kV was used. Images from the transmission microscope were recorded with the CCD Olympus Quemesa camera.

Hardness measurements of the tested specimens were performed with the Vickers method using the microhardness meter MMT-X3 in compliance with the PN-EN ISO 6507-2:1999 standard. Measurement time was 15s under the load of 300 g.

The nanoindentation measurements were performed with the use of the Indentation Release Candidate „SBO" tester. The measurement consisted in pressing-in the Berkowicz indenter under the maximum load of 300 mN within 15 second time, giving the impression in the shape of regular tetrahedron. At the base of the measurements the following values have been defined: microhardness HV_{IT} and the indentation hardness H_{IT}. The instrumental Young's modulus E_{IT} has been determined with the use of the Olivier and Pharr method. Three measurements were performed for each specimen while changing the measurement location by several hundred micrometres. The tests were conducted in the central part of a specimen.

3. RESULTS AND DISCUSSION

The first stage of the research involved microscopic analysis of the material in the as delivered state, in the form of wire rod of the 3.15 mm diameter, obtained as a result of earlier hot plastic working. The performed microscopic tests in the non-etched state have shown the presence of non-metallic inclusions, exclusively in the form of oxides distributed punctually in quantity not exceeding the TP1 standard according to the PN-H-04510:1964P

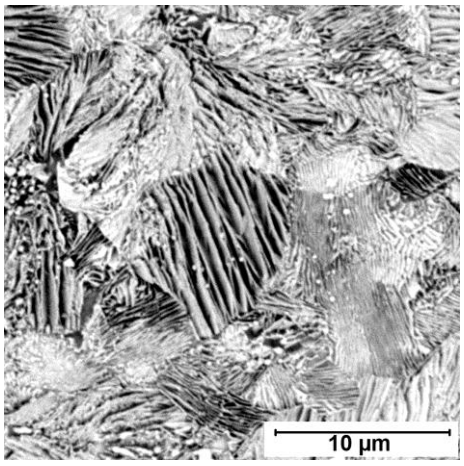

Fig. 1. Microscopic image of specimen No. 1, visible distinct lamellar build of pearlite. SEM.

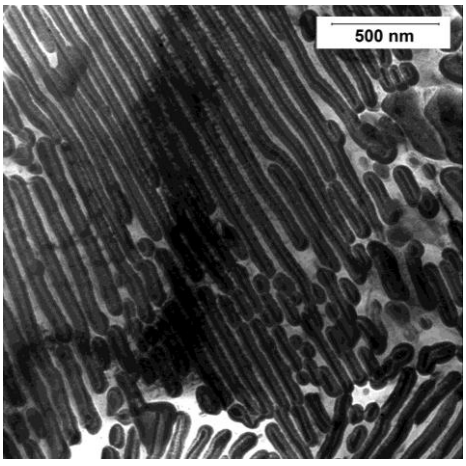

Fig. 2. Magnified area shown in Fig. 1, visible cementite lamellae of 50-70 nm thickness. TEM

standard. It has been found that such small number of non-metallic inclusions has no effect on mechanical properties of the tested steel and should not influence the process spheroidisation of cementite during tempering operations.

Microscopic observations of the material in the etched state have shown the presence of structure typical for unalloyed pearlite steel. The pearlite observed by means of metallographic microscope at low magnifications is etching itself into a grey colour, and instead, at greater magnifications its distinct lamellar build is already marked, where the hard, difficult to etch cementite protrudes over the soft ferrite. Sizes of the observed structural components amounted in sequence: pearlite colony 2-6 μm, cementite lamellae thickness about 50-70 nm (Fig. 1 and 2).

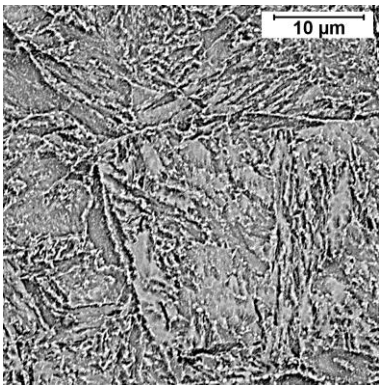

Fig. 3. Microstructure of material of the specimen No. 2, subjected to tempering in temperature of 500°C/1h. Visible microstructure of temper sorbite. SEM

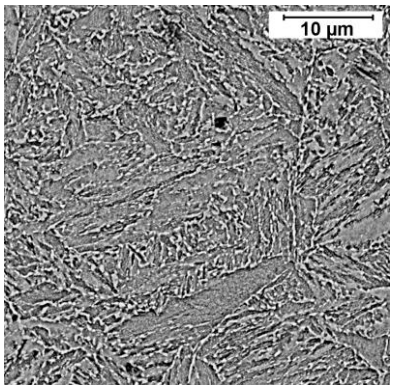

Fig. 4. Microstructure of material of the specimen No. 3, subjected to tempering in temperature of 500°C/2h. Visible microstructure of temper sorbite. SEM

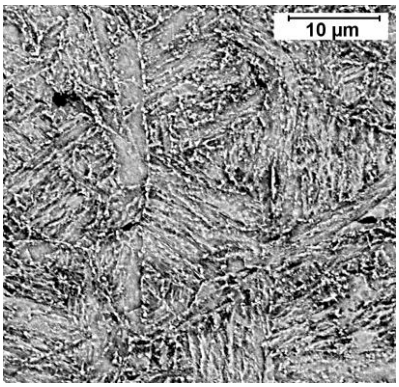

Fig. 5. Microstructure of material of the specimen No. 4, subjected to tempering in temperature of 500°C/3h. Visible microstructure of temper sorbite. SEM

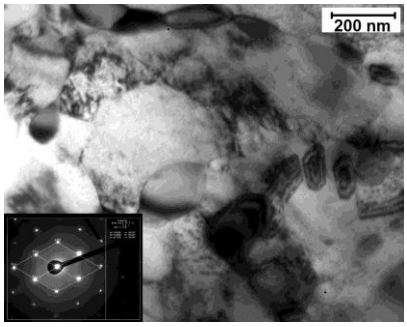

Fig. 6. Magnified area shown in Fig. 3, visible precipitations of coagulated cementite of 200-250 nm sizes. TEM

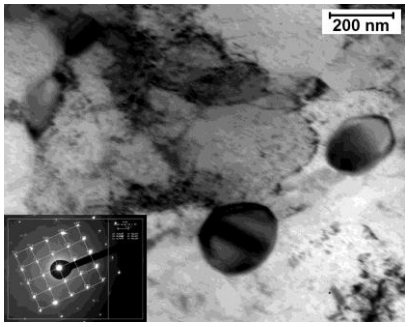

Fig. 7. Magnified area shown in Fig. 4, visible precipitations of coagulated cementite of 200-250 nm sizes. TEM

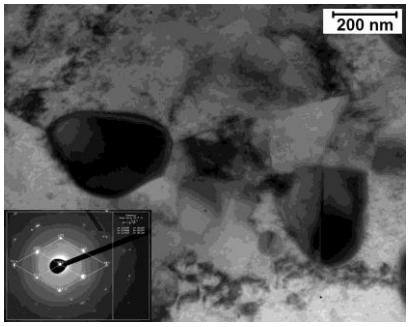

Fig. 8. Magnified area shown in Fig. 5, visible precipitations of coagulated cementite of 200-250 nm sizes. TEM

Results of the microscopic observations of the specimens subjected to heat treatment operations have shown lack of differences in structures of the materials obtained as a result of tempering in different times, each of the proposed schemes of heat treatment enabled

obtaining correct structure of temper sorbite with visible coagulated precipitations of cementite in the ferritic matrix (Fig. 3-5). Sizes of the observed dispersion cementite precipitations were similar in all the tested specimens and they amounted to 200-250 nm (Fig. 6-8). Thus, it can be concluded that the sufficient time warranting the proper tempering of cold drawn wires of pearlite steel is one hour.

The confirmation for the theory could be found in results of the mechanical properties tests. The obtained measurement results have shown that in all the applied tempering times the material plasticisation assumed in the modification theory takes place, hardness of the plastically strained specimen amounts to 477 HV0.3, and of the specimens material after the heat treatment is within the range of 298-387 HV0.3 (Table 2). At the same time the attention is drawn by the fact, that hardness of the specimen subjected to tempering in one hour time is clearly lower from the remaining cases and it amounts to about 298 HV0.3, where hardness of the specimen tempered for two hours equals as much as 387 HV0.3.

Table 2. Results of hardness measurements for tested specimens

SPECIMEN	HARDNESS HV0.3					AVERAGE HV0.3
No. 1	482	476	488	463	475	477
No. 2	295	308	296	289	301	298
No. 3	389	396	387	367	398	387
No. 4	359	347	354	356	343	352

Similar relationship has been observed in measurement results of nanoindentation, also in those microhardness measurements HV_{IT}, as well as the indentation hardness H_{IT} for the specimen subjected to tempering in one hour time was the lowest and amounted to HV_{IT} = 432 and H_{IT} = 4140 MPa correspondingly (Table 3). Additionally, the attention is drawn by the value of the instrumental Young's modulus E_{IT}, which in case of the discussed specimen is the lowest and amounts to 69 GPa, where in case of the specimens subjected to tempering in longer times is equal to about 72-76 GPa.

Table 3. Results of the nanoindentation measurements for the tested specimens

SPECIMEN	H_{IT} [MPa]	E_{IT} [GPa]	HV_{IT}
No. 2	4140	69	432
No. 3	6258	76	594
No. 4	4665	72	500

It therefore seems clear that in the studied case the heat treatment within one hour is fully sufficient for the full steel tempering to take place and the assumed in the discussed theory of modification the material plasticisation. This is the very important notice from the economic point of view and it clearly shows how extremely important is the individual selection of the heat treatment parameters for the specific industrial applications.

4. CONCLUSIONS

The fatigue strength of steel wires is highly dependent on metallurgical purity of material; too high content of non-metallic inclusions in steel is not permissible as around them the stress concentrations are taking place leading in effect to cracking of parts. The above theory is one of many explaining the issue of cracking the pearlite steel subjected to operation process. In addition, the literature data indicates that the cause for cracking could be dislocation pile-ups at the lamellar phase's separation boundary and the violent increase in energy related with it leading to thermodynamic destabilising of cementite.

The research conducted by the Author was aimed at developing modification of heat treatment parameters for pearlite steel enabling production of cold drawn wires characterised with better plastic properties and higher fatigue strength than the products obtained during standard processes. The idea of modification was based at a possibility of obtaining steel of sorbite or temper troostite structure, which are a mixture of ferrite and dispersion cementite of high degree of coagulation. The structures obtained with this method, as being globular should be featured with better plastic properties than the lamellar structures, which are created during the standard diffusion transformations of austenite. The described change in addition was to enable lowering the total surface of interphase boundaries at which the highest structure delaminations were observed, regardless of whether the cause of the changes resulted from presence of non-metallic inclusions or the pile-up of dislocations.

The results of earlier performed tests have shown that the temperature bringing benefits from the microstructure point of view and the mechanical properties for pearlite steel is 500 ºC. Further studies of the steel wires modification process were aimed at determining the impact of tempering time change on structure and mechanical properties of steel, and by that at the effectiveness of the modification. The applied scheme of heat treatment at the stage of the research involved hardening from temperature of 770ºC and tempering in the temperature of 500°C and the time of 1, 2 and 3 hours in sequence. The results of microscopic observations and hardness measurements have shown that in the studied case the heat treatment within one hour is fully sufficient for the full tempering of steel to take place. The hardening and tempering process performed that way enabled obtaining the proper temper sorbite structure of the lowest hardness of all the specimens equal to 298 HV0,3.

The confirmation for the theory could be found in results of the nanoindentation measurements also in those microhardness measurements HV_{IT} and the indentation hardness H_{IT} for the specimen subjected to tempering in one hour time was the lowest and amounted

to HV_{IT} = 432 and H_{IT} = 4140 MPa correspondingly. In addition the notice is drawn to low value of the instrumental Young's modulus E_{IT} amounting to about 69 GPa, where in case of specimens subjected to tempering in longer times it was equal about 72-76 GPa. This is the very important notice from the economic point of view and it clearly shows how extremely important is the individual selection of heat treatment parameters for specific industrial applications.

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