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THE STRUCTURAL AND STRENGTH CHANGES RESULTING FROM MODIFICATION OF HEAT TREATMENT OF HIGH CARBON STEEL

Pearlitic steels containing from some 0,8 to 0,95% C belong to the group of unalloyed steels intended for cold drawing or rolling. One of the problems discussed in literature is cracking of pearlitic steel subjected to plastic working, caused by high brittleness of the lamellar precipitations of hard cementite. This issue is extremely important because it affects significantly reduce fatigue strength. The paper presents proposals to modify the process of heat treatment, results in getting a steel with spheroidal structure characterized by better plastic properties, in order to eliminate this problem.

Keywords: pearlit, cementite, spheroidization, plastic working

1. Introduction

Pearlitic steels containing from some 0,8 to 0,95% C belong to the group of unalloyed steels of the quality class destined for cold drawing or rolling [1-3]. However, the steels have a very small percentage of nonmetallic inclusions and a limited chromium and nickel content, which factors contribute to the extension of pearlitic reaction time. The pearlitic steels in the annealed state have the highest strength in relation to other unalloyed steels, therefore they have found application mainly as wire rods for tyre reinforcing cords (PN-EN 10323:2005 (U)), hoses (PN-EN 10324:2006) and ropes (PN-EN 10264-1:2005) [2-5]. However one of the issues widely discussed in literature is cracking of pearlitic steel during plastic working caused by decomposition of cementite and the formation of nanocrystalline microstructure [6-16]. This issue is extremely important because it affects significantly reduce fatigue strength. The stability of cementite was studied by Gridnev and Gavrilyuk thanks to Mössbauer spectroscopy, they explain why carbon atoms leave cementite and spread in the ferrite [12-14]. Another explanation was proposed by Languillaume at al. They suggest that dramatic increase of the interfacial energy leads to the termodynamical destabilization of cementite [15]. Ohsaki et al. reported that the most of carbon atoms are located at the grain boundaries, forming an amorphous-like grain boundary phase [16]. Although the issue of fragmentation and decomposition of cementite in severely deformed pearlitic steel is well established and it is still a subject of controversy.

In the present paper it is proposed to modify the inter-operation heat treatment in order to eliminate such problems. Modification of the heat treatment will involve the hardening and tempering treatment, as the first stage of the manufacturing wire. The idea of modification was based at possibility of obtaining a steel of the sorbite or tempering troostite structures, which are a mixture of ferrite and dispersive cementite of high coagulation degree [1-3,17,18]. Structures obtained with such method as spherical in shape are to be featured by better plastic properties than lamellar structures, being created during the standard austenite diffusion transitions. Thus the above mention problem with decomposition of cementite in pearlitic steel subjected to cold working could be eliminated.

2. Experimental procedures

The aim of the tests was to show that by modifying of heat treatment parameters of pearlitic steel can obtain cold-drawn wires characterized by better plastic properties than the products manufactured with using standard processes.

Subject of the researches was pearlitic steel of chemical composition (see Table 1) and mechanical properties according the PN-EN 10323:2005 (U) Standard. Samples for tests were prepared in the form of steel wires of diameters ranging from 3.15 mm to 0.8 mm, obtained in the subsequent stages of standard cold working and simultaneously after the heat treatment involving hardening (in oil) and tempering with the 300°C, 400°C and 500°C temperature range as the first stage of the cold drawing (see Table 2).

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TABLE 1

Chemical composition of pearlitic steel acc. to the PN-EN 10323:2005 (U)

Chemical Composition	**C**	**Mn**	**Cr**	**Ni**	**Si**	**S/P**
%	0.83	0.4-0.6	<0.1	<0.15	<0.3	<0.025

TABLE 2

The sequence of samples investigated and documented within this article

Sample	**State of material**
No. 1	Matrial in the State of Delivering
No. 2	Sample no. 1 After Plastic Working
No. 3	Sample no. 1 After Hardening and Tempering at 300°C
No. 4	Sample no. 3 After Plastic Working
No. 5	Sample no. 1 After Hardening and Tempering at 400°C
No. 6	Sample no. 5 After Plastic Working
No. 7	Sample no. 1 After Hardening and Tempering at 500°C
No. 8	Sample no. 7 After Plastic Working

A NIKON ECLIPSE MA200 light microscope with the NIS Elements BR software and Phenom G2 electron scanning microscope were used to evaluate the microstructure of the tested steel. Both unetched and etched material was examined under magnifications ranging from 100× to 20 000×. Microsections of the subsequent samples were prepared in the direction compatible with the plastic working, using the mechanical processes of grinding and polishing, as well as the chemical etching with 3% Mi1Fe.

Measurements of Vickers hardness were executed on transverse section of samples, about five imprints on one sample, according to the PN EN ISO 6507-1 Standard. MMT-X3 micorhardness tester was used, with the load 500 g and working in the time of 15 s.

The static tensile test was carried out in according to the PN-EN ISO 6892-1:2010 Standard, MTS 858 Mini Bionix strength tester was used. Specimens with original length L_0 = 100 mm were got from the wire. The tensile tests were conducted at constant tension rate $\dot{e}_{Lc}$ = 0.0067 1/s (method A according to the standard) controlled on the basis the deformation rate, until failure. The basic material strength properties: R_m, Young's modulus E and percentage reduction of area after fracture Z were determined.

3. Results and Discussion

The observation of the sample No. 1, considered to be of an origin diameter of 3.15 mm, shown that this specimen is a pearlitic steel for drawing applications with 0.8% of carbon (see Fig. 1). Pearlite observed under the scanning electron microscope at greater magnifications as a clear lamellar structure appears, in which a hard, and hardly etching cementite lamellae protrudes over the soft ferrite (see Fig. 2).

The rectangular shape of pearlite colonies indicates that colonies' growth took place at a stress state in all the surrounding volume. A characteristic rotation around one axis during plastic deformation is revealed.

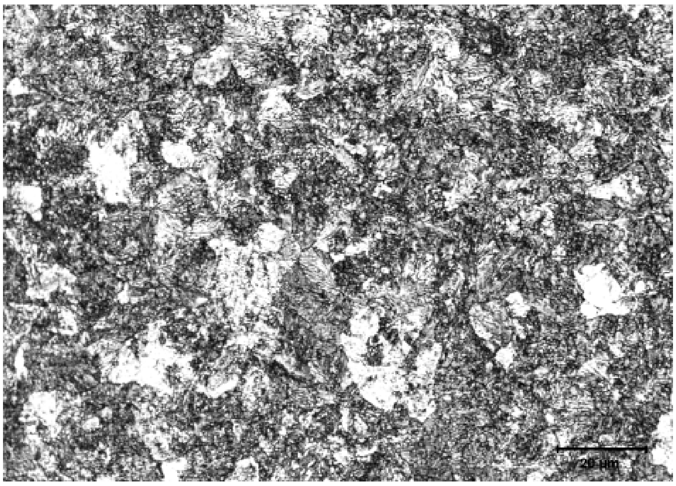

Fig. 1. Material in the state of delivery, visible typically steel for drawing applications with 0.8% of carbon. Etched condition, LM

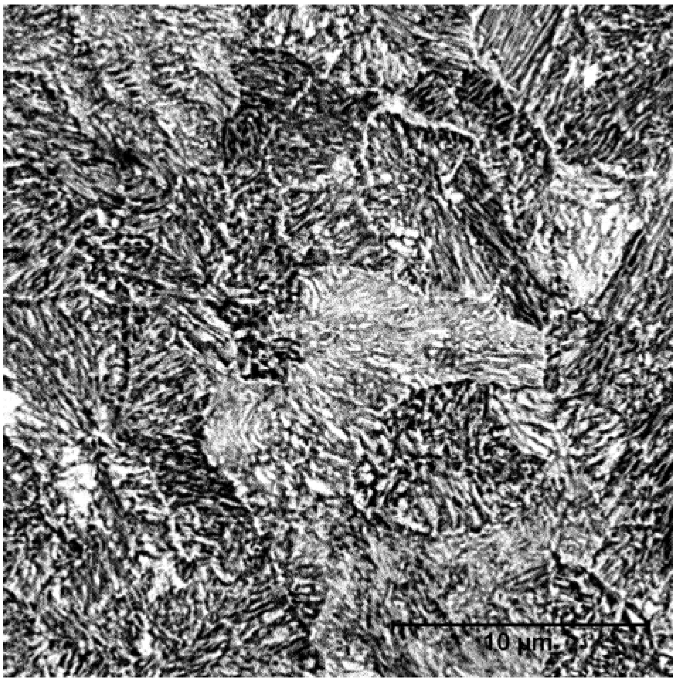

Fig. 2. Magnified area of structure shown in Fig. 1, visible hard, and hardly etching cementite lamellae protrudes over the soft ferrite. SEM

The microscopic examinations in the unetched sample made it possible to determine the degree of impurity and to describe in detail the nonmetallic inclusions. Only oxides, arranged point-wise, in the amount consistent with the TP1 according to the PN-H-04510:1964P Standard, occurred in the analyzed material (Fig. 3). The small amount of nometallic inclusions present does not affect the mechanical properties of the steel, but it can have a major effect on the spheroidization of the cementite during heat treatments [19].

However one of the important problems widely reported in the literature is cracking of pearlitic steel during plastic working,

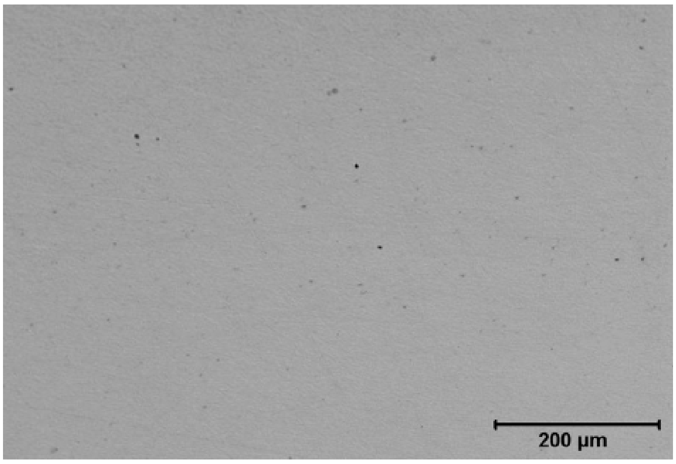

Fig. 3. Microstructure of unetched sample No. 1, visible small amount of oxides inclusions evenly located. Unetched condition, LM

due to the high brittleness of the lamellar precipitates of hard cementite [6-16]. The idea of the proposed modification was to obtain steel with a globular structure, characterized by better plastic properties than those of classic lamellar structures. The transformation of lamellar cementite structures into the globular form is called spheroidization. This process proceeds at higher temperatures or after longer times of tempering the steel as well as by long spheroidization annealing [2-4,19]. The modification included the hardening (in oil) from the temperature of 770°C and two-hour long tempering at a temperature of 300-500°C as the first stage of the cold drawing.

Although the literature data clearly show that the coagulation of cementite during tempering proceeds at temperatures above 400°C, the temperature range in the experiment presented in this paper was intentionally extended and ultimately it covered heat treatment at 300°C, 400°C and 500°C [1-5,20]. The temperatures were selected on the basis of the theory according to which the areas in which is the most often initiated the fragmentation of cementite are the places where ferrite subboundaries intersect cementite lamellas as well as concentrations of lattice defects in the cementite and concentrations of admixtures or non-metallic inclusions [19]. This means that the additional factors contributing to the effectiveness of coagulation are: the graininess of the structure, the metallurgical purity and the degree of plastic deformation of worked material. Therefore it seems necessary to individually select the temperature range for cementite coagulation in the particular material states.

Microscopic observation shown that the result of tempering with the 300°C the dissolution of carbide ε and the independent precipitation of fine-lamellar cementite take place (Figs 4 and 5). As the heating progresses over about 400°C a precipitation of carbon from martensite is taking place and creation of cementite, by very high dispersivity and a small degree of coagulation as well as decomposition of residual austenite. The specific volume decreases and the formed structure is called troostite (Figs 6 and 7). When the steel is heated to about 500°C further cementite coagulation in the ferritic matrix takes place, consisting in dissolving fine particles and growth of big ones, which begin to assume the shape close to the sphere. After such tempering the structure is called sorbite (Figs 8 and 9). Thus it turns out that the coagulation of cementite in the case of this particular tested group of the material occurs already at 400°C and directed spheroidization takes place at 500°C. As part of future research it would be worthwhile to closely study the structures tempered in a temperature range of 400-500°C as well as at higher temperatures.

Fig. 4. Microstructure of sample subjected to modification including hardening and tempering at 300°C, visible structure of troostite with small needles of martensite. SEM

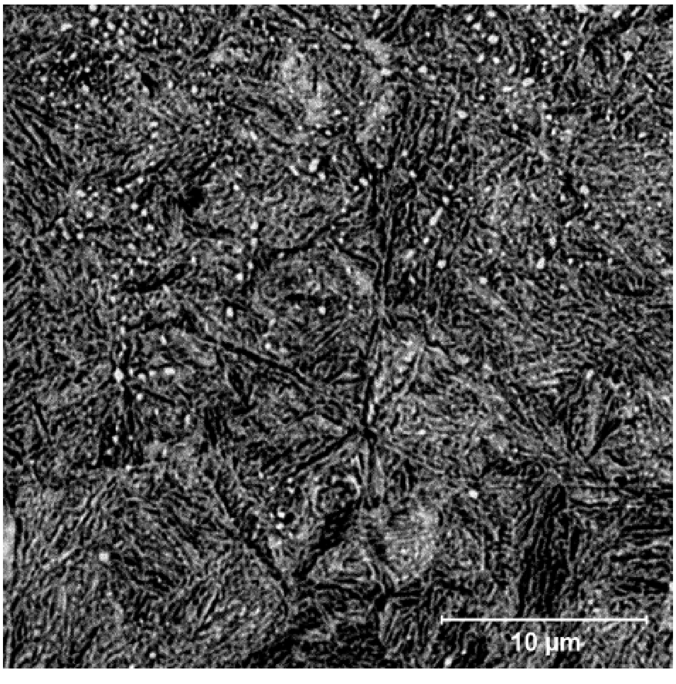

Fig. 5. Microstructure of the sample No. 5, visible a mixture of martensite and cementite of high dispersivity of coagulation. SEM

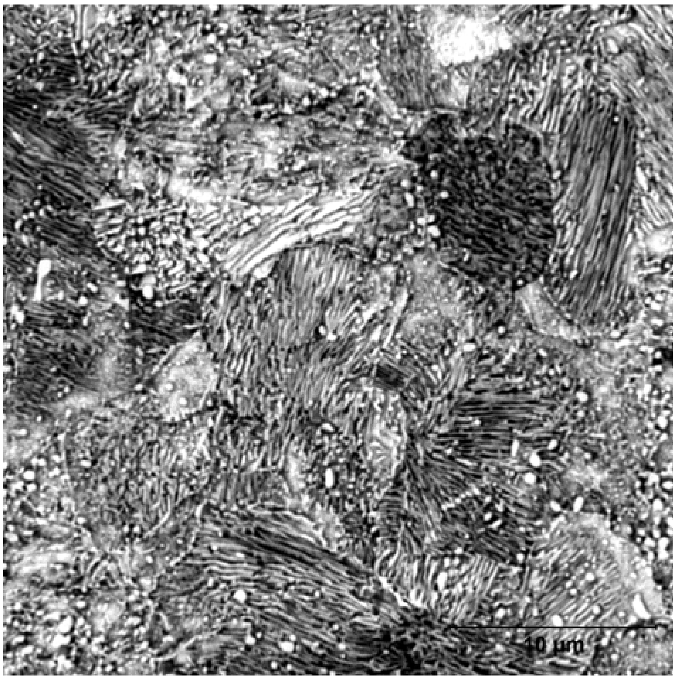

Fig. 6. Microstructure of material after modification at 500°C, visible numerous spherical disengagement of cementite in the ferritic matrix. SEM

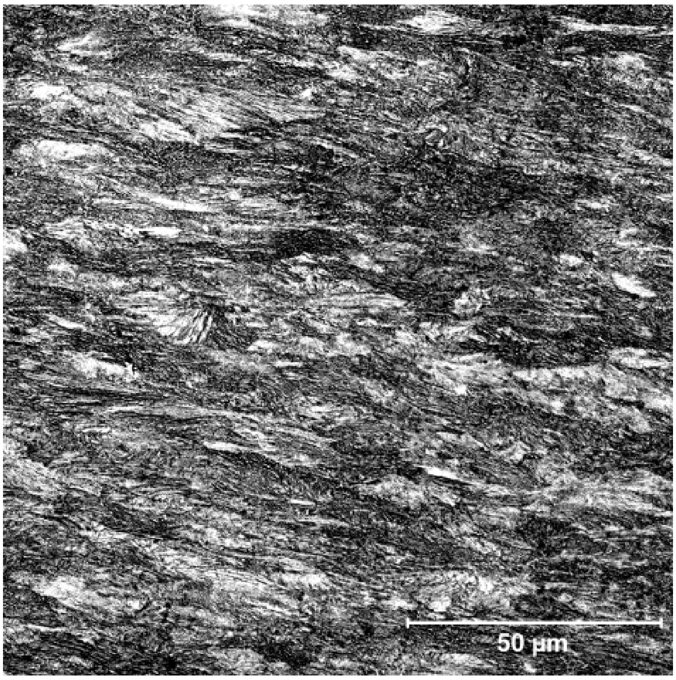

Fig. 7. Microstructure of unmodified material subjected to cold working treatments, visible distinct plastic deformation in order to 70%. SEM

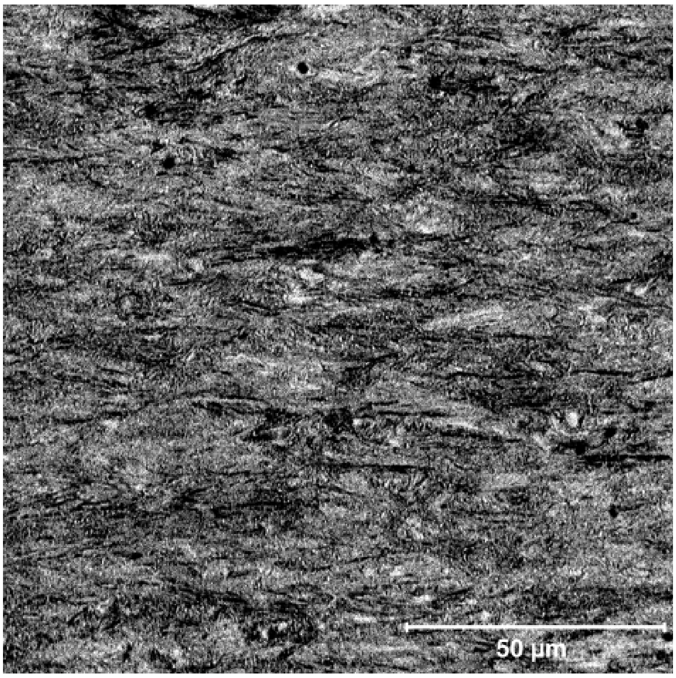

Fig. 8. Microstructure of sample No. 4, previously modified at 300°C, after cold drawing, visible 70% material reduction. SEM

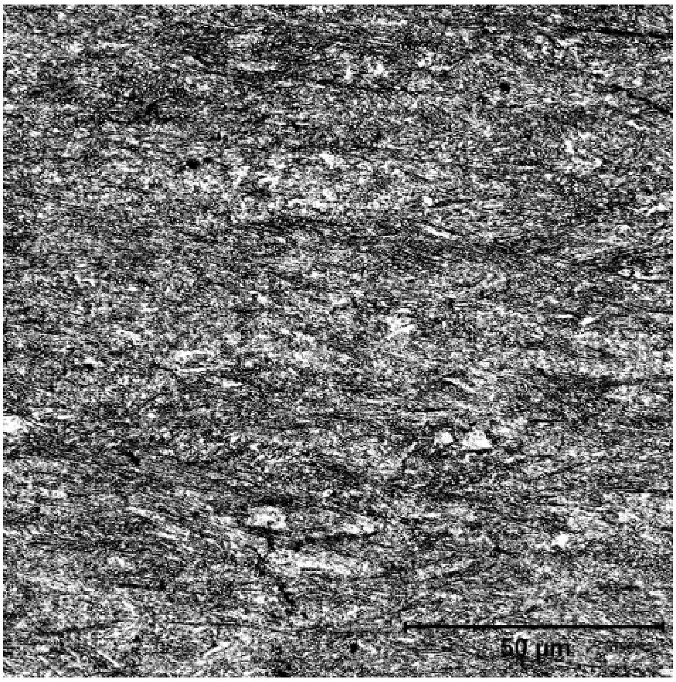

Fig. 9. Material tempered at the temperature 400°C, subjected to cold working treatments, visible 40% material reduction. SEM

Pearlitic steels, containing from about 0.8 to 0.95% of C, belong to the group of unalloyed steels of the quality class destined for cold drawing or rolling, this steel has the highest strength in relation to other unalloyed steels, therefore she have found application mainly as wire rods for hoses, wires for car tire reinforcement and cables [1-5]. In range of presented experiment plastic working process was carried out, samples for tests were prepared in the form of steel wires of diameters ranging from 3.15 mm to 0.8 mm, obtained in the subsequent stages of standard cold working.

The results of microscopic observations show that applied interventions had made possible obtainment structures with the diverse degree of plastic deformation. The highest degree of plastic deformation, amounting to 70%, characterized specimens

No. 2 and 4 in which structure of lamellar precipitations of cementite had been previously observed (Figs 10 and 11). After cold drawing, the microstructure of pearlitic steel tempered at 400°C and 500°C is characterized by a much lower degree of deformation of about 40% (Figs 12 and 13). This is due to the fact that the globular structures, which formed under the modified heat treatment configuration, are characterized by better plastic properties than the classic lamellar structures. Thus one can expect that the wires manufactured using this technology will be many times less susceptible to cracking during plastic working and it will be possible to use larger differences in diameters between the consecutive stages of drawing and the necessary interoperation annealing, which ultimately can result in a shorter overall process time.

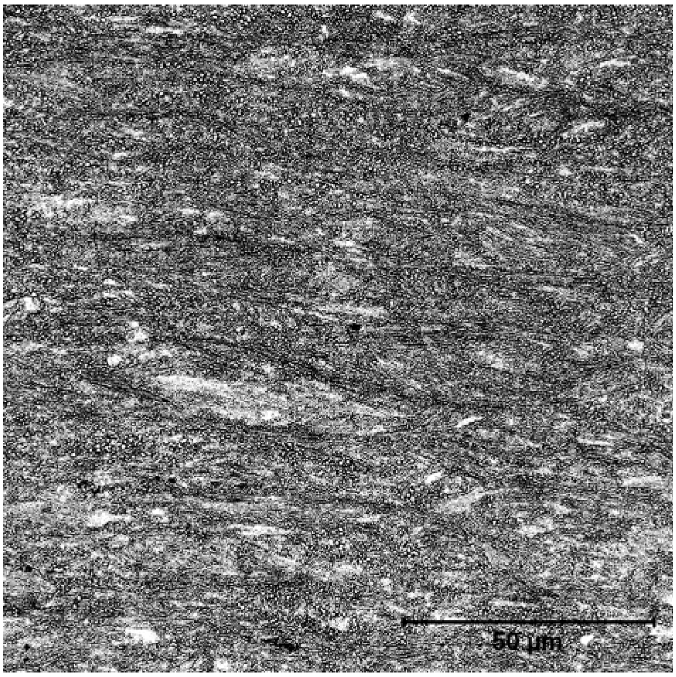

Fig. 10. Microstructure of sample No.8, after cold drawing, visible plastic deformation in order to 40%. SEM

The consequence of the plastic treatment strain, is a change in almost all metal properties [1-5, 21]. The changes manifest themselves, first of all, by consolidation of the metal, i.e. increase in its strength, yield point and hardness and, at the same time, by lowering the elongation and impact resistance. The modification of the heat treatment of cold drawn wires was aimed at obtaining steel characterized by better plastic properties. The confirmation of this can be clearly found in the results of microhardness measurements.

The microhardness of pearlitic steel with the classic lamellar structure, subjected to plastic working, amounts to about 416 HV while microhardness of the steel tempered at 500°C, characterized by distinctly spheroidal structure, was about 379 HV (see Table 3).

At the same time the measurement results indicate that there is no reason to carry out the modification at lower temperatures. The modification at the temperature of 300°C not only did not lead to the coagulation of cementite, but additionally increase the material hardness even to 629 HV. At this stage of the investigations it can be stated that the only temperature bringing considerable benefits as regards both material microstructure and properties is the temperature of 500°C.

TABLE 3

The results of Vickers hardness measurements

No.	Hardness hV					Average Hardness
Nr 1	212.5	235.1	220.6	218.6	238.4	225.1
Nr 2	399.5	413.2	435.7	427.2	408.7	416.9
Nr 3	300.5	305.9	314.1	322.7	310.7	310.8
Nr 4	369.2	363.2	383.7	382.1	400.8	379.8

Hardness measurements usually do not provide conclusive evidence about the ductility and strength of a material. Therefore the other properties of the material of the modified and unmodified cold drawn wires were determined in the tensile test. The test results showed the validity of the tempering at 500°C, process this leads to obtainment the steel about comparably high strength: R_m = 1420 MPa and 1202 MPa respectively before and after the modification and much higher ductility, area reduction for basic specimen amount Z = 13.9% and for modified specimen 32.9% (see Table 4).

TABLE 4

The results of static tensile test of tested samples

Specimen	R_m [MPa]	E [MPa]	Z [%]
No. 2	1420	$1.04 \cdot 10^5$	13.9
No. 4	1202	$1.08 \cdot 10^5$	31.6

Therefore we can expect that the material after preliminary modification with regard to heat treatment will be characterized by much lower susceptibility to cracking during product shaping processes and also during exploitation of finished products. In the case of the modification including steel tempering at lower temperatures of 300°C and 400°C, higher ductility Z, amounting to 25-31%, was observed, but the strength of the material was considerably lower: R_m – 930-936 MPa, which ultimately disqualifies this range of the modification of the cold drawing wires made of pearlitic steel.

4. Conclusions

Pearlitic steels, containing from about 0.8 to 0.95% of C, belong to the group of unalloyed steels of the quality class destined for cold drawing or rolling, this steel has the highest strength in relation to other unalloyed steels, therefore she have found application mainly as wire rods for hoses, wires for car tire reinforcement and cables [1-5]. One of the problems discussed in literature is cracking of pearlitic steel subjected to plastic working, caused by high brittleness of the lamellar precipitations of hard cementite [6-16]. This issue is extremely important because it affects significantly reduce fatigue strength.

The paper presents proposals to modify the process of heat treatment, results in getting steel with spheroidal structure characterized by better plastic properties, in order to eliminate this problem. The proposals modification included the hardening (in oil) from the temperature of 770°C and two-hour long tempering at a temperature of 300-500°C as the first stage of the cold drawing. Although the literature data clearly show that the coagulation of cementite during tempering proceeds at temperatures above 400°C, the temperature range in the experiment presented in this paper was intentionally extended and ultimately it covered heat treatment at 300°C, 400°C and 500°C [1-5, 20]. The temperatures were selected on the basis of the theory according to which the additional factors contributing to the effectiveness of coagulation are: the graininess of the structure, the metallurgical purity and the degree of plastic deformation of worked material [19].The results of the presented investigations clearly show that the temperature bringing considerable benefits, as regards both material microstructure and properties, is the temperature of 500°C, whereas the use of lower temperatures seems to be completely unnecessary. The idea of the proposed modification was to obtain steel with a globular structure, characterized by better plastic properties than those of classic lamellar structures. Owing to the heat treatment interoperation tempering in 500°C as a pre-preliminary intervention give the opportunity to get the steel with a spheroidal microstructure characterized by better plastic properties than the classic lamellar one. The hardness of pearlitic steel with the lamellar structure, subjected to plastic working amounts to about 416 HV and that after modification to about 379 HV. In the case of the other using temperatures disadvantageous increase (even to 629 HV) of material hardness was observed.Hardness measurements usually do not provide conclusive evidence about the ductility and strength of a material therefore the other properties of the tested specimens were determined. The test results clearly showed that owing the modification at 500°C leads to obtainment the steel about comparably high strength: R_m = 1420 MPa and 1202 MPa respectively before and after the modification and much higher ductility, area reduction for basic specimen amount Z = 13.9% and for modified specimen 32.9%. In the case of the modification including steel tempering at lower temperatures of 300°C and 400°C, higher ductility Z, amounting to 25-31%, was observed, but the strength of the material was considerably lower: R_m – 930-936 MPa, which ultimately disqualifies this range of the modification of the cold drawing wires made of pearlitic steel.

Thus one can expect that the wires manufactured using this technology will be many times less susceptible to cracking during plastic working and it will be possible to use larger differences in diameters between the consecutive stages of drawing and the necessary interoperation annealing, which ultimately can result in a shorter overall process time.

REFERENCES

[1] J. Adamczyk, Engineering of metal materials (in Polish), Wyd. Politechniki Śląskiej, Gliwice 2004.

[2] M.F.Ashby, D.R.H. Jones, Engineering Materials: An Introduction to Microstructures, Processing and Design, Elsevier, Oxford 2005.

[3] L.A. Dobrzański, Engineering materials and material design (in Polish), WNT, Warszawa 2006.

[4] D. Henkel, A.W. Pense, Structure and Properties of Engineering Materials, The McGraw-Hill Higher Education, Singapore 2002.

[5] R.A. Higgins, Materials for Engineers and Technician, Newnes 2006.

[6] D. Grygier, M. Rutkowska-Gorczyca, R. Jasiński, W. Dudziński, Journal of Machine Engineering **14**, 1 (2014).

[7] C. Cordier-Robert, at all, Journal of Materials Science **43**, 4 (2008).

[8] V.I. Izotov, at all, Physics Of Metals And Metallography **103**, 5 (2007).

[9] M.X. Zhang, P.M. Kelly, Materials Characterization **60**, 6 (2009).

[10] T. Tarui, N. Maruyama, H. Tashiro, Journal of the Iron and Steel **91**, 2 (2005).

[11] M. Suliga, Archives of Metallurgy and Materials **57**, 4 (2012).

[12] V.G. Gavriljuk, Materials Science and Engineering: A **345**, 1-2 (2003).

[13] V.G. Gavriljuk, S.P. Oskaderov, Neue Hütte **30**, 10 (1985).

[14] V.N. Gridnev, V.G. Gavrilyuk, Physics of Metals **4**, 3 (1982).

[15] J. Languillaume, G. Kapelski, B. Baudelet, Acta Materialia **45**, 3 (1997).

[16] S. Ohsaki at all, Scripta Materialia **52**, (2005).

[17] G.E. Totten, M.A.H. Howes, Steel Heat Treatment Handbook, Marcel Dekker, New York (1997).

[18] P. Matusiewicz, W. Ratuszek, A. Zielinska-Lipiec, Archives of Metallurgy and Materials **56**, 1 (2011).

[19] F. Staub at all, Materials science (in Polish), Wydawnictwo "ŚLĄSK", Katowice (1978).

[20] G.F. Vander Voort (ed), Atlas of Time-Temperature Diagrams for Iron and Steels, ASM International, Materials Park, OH 2004.

[21] P. Matusiewicz, W. Ratuszek, A. Zielinska-Lipiec, Archives of Metallurgy and Materials **56**, 1 (2011).