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The Effectiveness of Recrystallization of Pearlitic Steels in the Regards of the Change the Annealing Time

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Abstract— **Although many heat treatment schemes have been developed for pearlitic steel, in the literature there is still little information about the influence of the different heat treatment parameters on the percentage, properties and morphology of the phases. Neither there is any information on matching the parameters, taking into account the different degree of deformation of the steel wires, the particular applications and operating conditions of the products. The aim of this research was to optimize the parameters of the interoperation annealing used during the cold plastic working of pearlitic steel intended for cold-drawn wires. The results of the mechanical properties and microscopic observations, presented in this paper clearly show that there is no need to apply long recrystallization treatments to small diameter wires. This finding is highly significant from the economic point of view and it clearly shows the importance of the individual matching of heat treatment parameters to specific industrial applications**

Index Terms— **pearlite, cementite, recrystalization, wire.**

I. Introduction

Pearlitic steels containing from about 0.8 to 0.95% C belong to a group of unalloyed steels intended for cold working, particularly drawing and rolling [1]. Since in comparison with the other low-alloyed steels their strength is the highest, they are used in the production of patented steel wires for tyre reinforcing cords (PN-EN 10323:2005 (U)), hoses (PN-EN 10324:2006) and ropes (PN-EN 10264-1:2005) [2].

Plasticity, i.e. formability, is one of the principal characteristics of metals, practically exploited to produce finished and semi-finished mill products, such as steel sheets, bars, wires and strips [3], amounting to over 80% of all the metal products produced by the industry [2]. The consequence of cold (below the recrystallization temperature) plastic deformation is a change in nearly all the properties of the metal [2]. The changes manifest themselves in mainly the strain hardening of the metal, i.e. in its higher strength, yield point and hardness and so in its lower elongation and impact resistance [3]. The considerable strain hardening of the deformed metal makes its further forming through hot working impossible since the latter leads to the failure of the material due to its decohesion [5-7].

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The recrystallisation annealing takes place during heating the steel subjected to cold plastic working. Squeeze strengthens metals changing almost all material properties, and therefore usually it is not possible to give the final shape of objects in one operation [6-8]. In order to enable further working the inter-operation recrystallisation annealing is applied which removes the effect of squeezing and restores the primary properties of metal.

The recrystallisation process however is influenced by many factors which greatly hinders optimization of that process; among others these are [8,9]:

- chemical composition of an alloy, metallurgic purity, phase composition and the structure before deformation,
- conditions of deformation; degree of squeeze, temperature and process rate,
- conditions of heat treatment and mainly: the heating period, heating rate and cooling rate.

Influence of the structure and purity of alloy is the subject of many research works, which however do not clarify this phenomenon unequivocally [11-13]. Course of recrystallisation depends on the form of additive or contamination contained in the steel, as well as on mutual relations between foreign atoms and grain boundaries. The foreign atoms first of all decrease mobility of grain boundaries, i.e. lower the rate of recrystallisation. They also change temperature of recrystallisation beginning and influence size of the recrystallised grains.

Among the conditions of plastic deformation the strongest impact on the course of recrystallisation, in particular on its temperature, makes deformation [7, 14-16]. At higher values of squeeze as a result of strong increase in nucleation rate, with relatively small increase in the rate of growth, the size of grain is decreasing. In addition an increase in the nucleation rate enables termination of recrystallisation at lower temperatures at the same annealing time.

The influence of temperature in which deformation was performed on phenomena taking place during recrystallisation is still relatively not enough explored, however it has been found that recrystallisation rate is all the greater the lower is the temperature of deformation [17]. Also little explored is the impact of the method of performing the plastic working leading to obtaining the finished product.

Numerous studies of the recrystallisation process of cold worked steel show clear influence of the heating rate applied during annealing [11, 15, 18]. The higher is the heating rate the higher is the temperature of beginning and the end of primary recrystallisation. It results from the fact that high rates of heating impede the healing course so, that the whole excess of the retained energy becomes released during primary recrystallisation. Steels after such process are characterised with better mechanical and plastic properties, fine-grain structure and lack of recrystallisation texture.

The most important and at the same time the most difficult in selecting conditions of the inter-operation annealing of steels is the fact that all the described factors interact with each other [9]. For that reason it is very often difficult to foresee the course of recrystallisation processes for a given finished product.

However, in the subject literature there is lack of data on influence of the described above parameters on share, properties and morphology of phases, which in the first order decide on mechanical and utility properties of that steel group [11-18]. There is also no information on selection of heat treatment parameters, taking into account different sizes of steel wires, specific application of products and conditions of their operation.

Thus, it results that performing optimisation of the process parameters for the inter-operation annealing applied during cold plastic working processes of pearlitic steels designated for wires is extremely important. There is a need for the optimisation considering sizes of the tested intermediates and the finished steel products, as well as their chemical composition, properties and phase morphology for that group of steels. Results from these observations will contribute to selection of the proper heat treatment technology during and after operations of the cold plastic working leading to obtaining of wires with possibly highest mechanical and plastic properties.

II. MATERIALS AND METHODS

Purpose of the research presented in the work was optimization of parameters, mainly the time of the inter-operation annealing process, applied during cold plastic working operations for the pearlitic steel designated for cold-drawn wires. As a result of the studies the influence of the heat treatment on the percentage, the properties and morphology of the phases which are mainly responsible for the mechanical and utilitarian properties of the steels belonging to this group, was determined.

The object of the studies was pearlitic steel with the chemical composition and properties consistent with standard PN-EN 10323:2005 (U). Specimens for tests had the form steel wires obtained after the successive stages of cold working from the diameter of 3.15 mm to 0,8 mm. The final stage in the preparation of the specimens consisted in recrystallization annealing at a temperature of 700°C for successively 15, 30, 45 and 60 minutes (Table 1). The heat treatment temperature was selected on the basis of earlier studies [12, 13] and it ensured the complete recrystallization of the deformed material.

Metallographic sections of specimens were prepared by means of mechanical grinding and polishing, as well as chemical etching with 3% Mi1Fe. For evaluation of microstructure of the tested steel a Phenom G2 scanning electron microscope was applied. Observations were performed on the etched material at magnifications from the range of 1000x ÷ 5000x. Each sample was mechanically ground on SiC abrasive papers (1000 and 1200) to thicknesses of 70 μm and after that polished. Samples were rectangular in shape and size of the samples ranged within 1,5 x 2 mm. Samples were electropolished using a solution consisting of 25 vol.% HNO3 and 75% methanol at 10 V voltage in a TenuPol. Overview of the microstructure studies by SEM was the basis for detailed TEM analyses, using Hitachi H-800 transmission electron microscope.

Microhardness measurements of the tested specimens were performed with the Vickers method using the MMT-X3 microhardness tester according to the PN-EN ISO 6507-2:1999 standard. Measurement time amounted to 15s, under the load of 300g.

Static tensile test was performed at the base of the binding standard PN-EN ISO 6892-1:2010. The tests were performed at the testing machine type MTS 858 Mini Bionix. Specimens were prepared of the wire of the initial gauge length L_0 = 100 mm. Tensile tests were conducted with the constant tensile rate controlled at the base of strain rate (method A according to the standard) equal to $\dot{e}_{Lc}$ = 0,0067 1/s until the fracture. The basic strength properties of the material were determined: tensile strength R_m, Young's modulus E, as well as the percent reduction of area after fracture Z.

Table 1. Tested specimens

SPECIMEN	MATERIAL CONDITION
No. 1	MATERIAL AFTER PLASTIC WORKING
No. 2	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/15minutes
No. 3	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/30minutes
No. 4	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/45minutes
No. 5	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/60minutes

III. RESULTS AND DISCUSSION

Microscopic observations of the tested material in the non etched state have shown presence of the very small number of non-metallic inclusions in the form of oxides (see fig.1). Impurities were distributed punctually and appeared in quantities not exceeding the standard No. 1 according to the PN-64/H-04510 standard. Thus, it was assumed that such small number of non-metallic inclusions does not influence mobility of grain boundaries, and by that does not decrease the recrystallisation rate. Further metallographic tests performed in the etched state at the transverse section of the tested wires have shown presence of the structure typical for the non-alloy pearlitic steel.

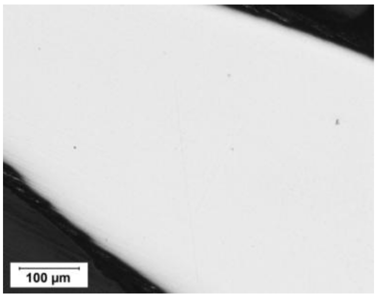

Fig. 1. Specimen No. 1, visible small number of non-metallic inclusions in the form of oxides. Non etched state, LM.

The microscopic tests performed at microsections made in accordance with the plastic working direction have shown that the applied cold drawing scheme of the tested wires enabled obtaining strong plastic deformation amounting to some 90% (fig. 2 and 3).

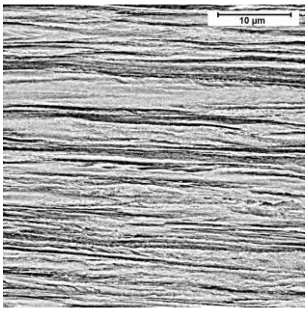

Fig. 2. Material after cold working, specimen no. 1, visible 90% material reduction. SEM

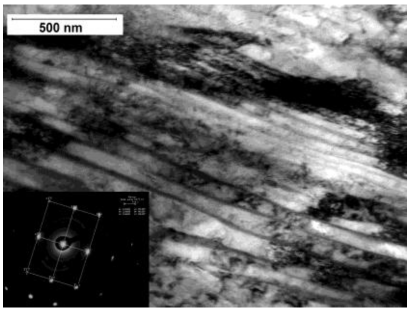

Fig. 3. Magnified area of structure shown in Fig. 2, visible strong plastic deformation of material. TEM

The effect of cold plastic deformation, i.e. below the recrystallisation temperature, is change in almost all properties of the metal. First of all the changes manifest themselves with metal strengthening, that is increasing its strength, yield point and hardness, and by that with decreasing of elongation and impact resistance. The strong deformation of the tested metal observed in microscopic tests inhibits its further shaping by cold plastic working, as it leads then to destruction of the material caused by its decohesion. In order to continue the cold plastic working between successive forming operations the recrystallisation annealing has to be performed. The heat treatment operation is aimed at removing the squeeze strengthening of the material and increasing its plastic properties which enables its further cold plastic working.

According to US patent no. 4,759,806, which describes in detail the manufacturing of patent steel wires out of pearlitic steel, the temperature of interoperation annealing should be in a range of 520-680°C and recrystallization annealing time should not exceed an hour [9,10]. In the present research recrystallization annealing at a temperature of 700°C for successively 15, 30, 45 and 60 min was used. The temperature of the heat treatment was selected on the basis of the research on this group of steels carried out by the present team, which had clearly shown that the most proper recrystallization temperature of plastically deformed pearlitic steel is 700°C since heat treatment at lower temperatures does not ensure the complete removal of the lamellar character of the microstructure, the consequence of which is the material's high strength and high E-modulus [16, 19].

Microscopic tests of the heat treated specimens No 2-5 have shown that at 700ºC full recrystallisation of material takes place, in that temperature radically disappear the structure banding and the precipitated cementite has typical lamellar form indicating the full and correct recrystallisation of the material. However micro observation did not show any differences in material structures obtained as a result of recrystallization annealing over different times (see fig. 4 and 5). The grain size estimated for all the tested materials on the basis of standard PN=84/H-04507 conformed to comparison chart no. 10 [2-4].

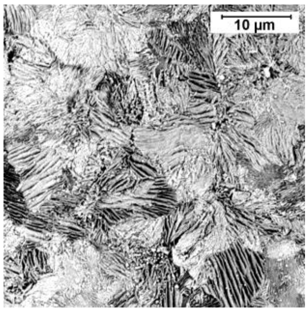

Fig. 4. Material of specimen no. 2, after 15 min long recrystallization annealing visible recrystallized fine-grained structure of pearlitic steel. SEM

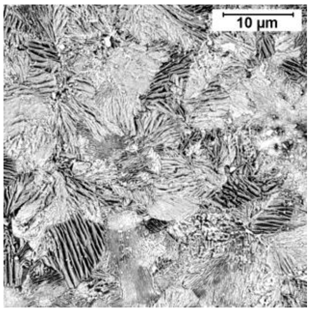

Fig. 5. Structure of material of specimen no. 5 after 60 min long recrystallization annealing, visible distinct lamellar structure of pearlite. SEM

Examinations of the structures under greater magnifications confirmed that all the adopted recrystallization annealing times were sufficiently long for proper material recrystallization to occur. A typical lamellar structure, in which the resistant to etching, hard cementite protrudes above the soft ferrite, was observed in all the tested materials. Length scales of the observed structures in this specimen were similar: pearlite colony 4 µm to 9 µm and cementite lamellas about 50-80 nm (see Fig. 6 and 7).

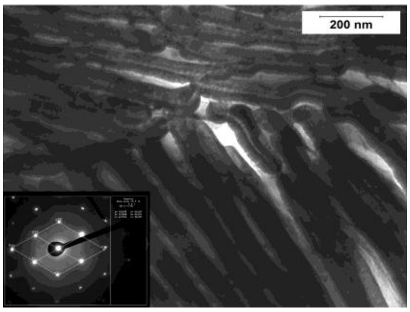

Fig. 6. Specimen no. 2, visible distinct lamellar structure of cemnetite length scales of the lamellas about 50-80 nm. TEM

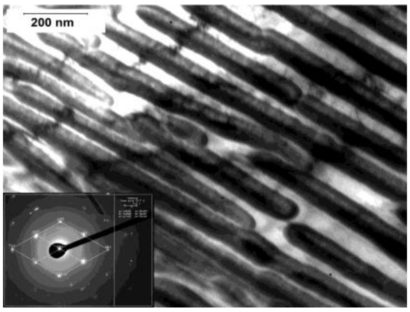

Fig. 7. Specimen no. 5, visible distinct lamellar structure of cemnetite length scales of the lamellas about 50-80 nm. TEM

Microscopic examinations of the structure of a material subjected to different heat treatment schemes can show only whether the parameters have been matched properly. In order to get a full picture of the results of the particular processes one should analyze the mechanical properties of the specimens. In industrial practice, the measurement of material hardness is usually used to determine whether the selected heat treatment is proper, mainly because of the ease of measurement and the fact that there are no special requirements for specimen preparation. For this reason Vickers hardness tests were used to preliminarily assess the optimization of the parameters of the interoperation annealing process used in the cold working of pearlitic steel intended for patented steel wires.

The test results showed that material plasticization occurs for all the adopted recrystallization annealing times. The hardness of specimen No. 1 (plastically deformed steel) amounts to 577 HV0,3 while that of the material of the specimens after heat treatment ranges from 364 to 369 HV (Table 2). It is also apparent that the hardness of the specimens subjected to annealing over different times is similar (the differences are within the margin of measuring error). This corroborates the previous microscopic observations and makes it even more certain that the time of 15 minutes is sufficient for the proper recrystallization of plastically deformed wires made of pearlitic steel.

Table 2. Results of measurements of strength and plastic properties of the tested specimens

No.	R_m [MPa]	Young's modulus E [MPa]	$R_{0,2}$ [MPa]	Z [%]	HV 0,3
No. 1	1998	$1{,}70 \cdot 10^5$	1321	25,6	577
No. 2	741	$1{,}76 \cdot 10^5$	701	38,7	363
No. 3	669	$1{,}61 \cdot 10^5$	534	43,0	365
No. 4	667	$1{,}76 \cdot 10^5$	583	41,3	369
No. 5	685	$1{,}60 \cdot 10^5$	634	35,0	364

Hardness measurement does not forejudge on ductility and strength of a material. In order to unmistakably determine which of the proposed heat treatment schemes is the most effective, as a result of the static tensile test the remaining properties of the tested specimen material were determined, the tensile strength R_m, $R_{0,2}$, Young's modulus E, as well as percent reduction of area after fracture Z.

Results of the studies have confirmed that the most suitable time for recrystallization annealing is 15 minutes, the process finally leads to obtaining steel of distinctively high tensile strength, R_m = 741 MPa and at the same time high ductility, area reduction Z for specimen No. 1 amounted to 38,7 % (Tab. 2). In case of recrystallisation annealing of steels in longer times 30-60 minutes similar high ductility Z = 35-43 % was observed and at the same time similar high material strength, R_m within the range of 669-685 MPa, it means that there is no need to used so log time of recrystallisation.

IV. CONCLUSIONS

Pearlitic steels containing 0.8-0.95% C belong to a group of unalloyed steels intended for drawing or cold rolling. The steels are characterized by a low content of nonmetallic inclusions and a limited chromium and nickel content, contributing to the elongation of the pearlite reaction. They are used mainly as rolled steel wires for springs, tyre reinforcing cords and ropes. According to US patent no. 4,759,806, which describes in detail the manufacturing of steel wires out of pearlitic steel, the recrystallization annealing time should not exceed an hour, but what it exactly means, is 15 minutes is enough or maybe 45 minutes?

Microscopic tests of the heat treated specimens have shown that at all times of annealing full recrystallisation of material takes place, radically disappear the structure banding and the precipitated cementite has typical lamellar form indicating the full and correct recrystallisation of the material. However micro observation did not show any differences in material structures, grain size and length scales of pearlite colony and cementite lamellas, obtained as a result of recrystallization annealing over different times, were similar.

Results of hardness measurements showed that material plasticization occurs for all the adopted recrystallization annealing times. The hardness of specimen after cold working amounts to 577 HV0,3 while that of the material of the specimens after heat treatment ranges from 364 to 369 HV0,3. It is also apparent that the hardness of the specimens subjected to annealing over different times is similar and the differences are within the margin of measuring error.

Result of the static tensile test confirmed too that the most suitable time for recrystallization annealing is 15 minutes, the process finally leads to obtaining steel of distinctively high tensile strength, R_m = 741 MPa and at the same time high ductility, area reduction Z for specimen No. 1 amounted to 38,7 %. In case of recrystallisation annealing of steels in longer times 30-60 minutes similar high ductility Z = 35-43 % was observed and at the same time similar high material strength, R_m within the range of 669-685 MPa.

This corroborates the previous microscopic observations and hardness measurements makes it even more certain that the time of 15 minutes is sufficient for the proper recrystallization of plastically deformed wires made of pearlitic steel, it means that there is no need to used so log time of recrystallisation.

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REFERENCES

[1] Ashby M.F., Jones D.R.H., Engineering Materials: An Introduction to Microstructures, Processing and Design, Elsevier, Oxford 2005
[2] Dobrzański L.A., Materiały inżynierskie i projektowanie materiałowe, WNT, Warszawa 2006
[3] Dobrucki W., Zarys obróbki plastycznej metali, Wyd. Śląsk, Katowice 1975
[4] Langford G., Deformation of Pearlite, Metall Trans A, 8A (6) 1977
[5] Hertzberg R.W., Deformation and Fracture Mechanics of Engineering Materials, John Wiley & Sons, New York 1989
[6] Cordier-Robert, C. at. all, Influence of torsion deformation on microstructure of cold-drawn pearlitic steel wire, Journal of Materials Science, Vol. 43 Iss.: 4, pp. 1241-1248, 2008
[7] Izotov, V.I. at all, Influence of the pearlite fineness on the mechanical properties, deformation behavior, and fracture characteristics of carbon steel, Physics Of Metals And Metallography, Vol.: 103, Iss.: 5, pp: 519-529, 2007
[8] Henkel D., Pense A.W., Structure and Properties of Engineering Materials, The McGraw-Hill Higher Education, Singapore 2002
[9] Vander Voort G.F. (ed), Atlas of Time-Temperature Diagrams for Iron and Steels, ASM International, Materials Park, OH 2004
[10] Ivanisenko Y., Lojkowski W., Valiev R.Z., Fecht H.J., The mechanism of formation of nanostructure and dissolution of cementite in a pearlitic steel during high pressure torsion, Acta Materialia, Vol.: 51, Iss.: 18, pp. 5555-5570, 2003
[11] Zhang M.X., Kelly P.M., The morphology and formation mechanism of pearlite in steels, Materials Characterization, Vol.: 60, Iss.: 6, pp. 545-554, 2009
[12] Grygier D., Rutkowska-Gorczyca M., Jasiński R., Dudziński W.: Microstructure evaluations in pearlitic steels wires resulting from modification of the interoperation annealing process, Journal of Machine Engineering, 2014, vol. 14, nr 1, s. 53-62
[13] Gerstein G., At all, Structural Evolution of Thin Lamellar Cementite during Cold Drawing of Eutectoid Steels, Procedia Engineering, Volume 81, 2014,
[14] Glover G., Sellars C.M., Recovery and recystalisation Turing high temperature deforming α-iron. Matallurg. Trans., 4, 1973, 765-771
[15] Sellars C.M., Whiteman J.A., Recrystalisation and grain growth in hot roling, Materials Science, 1979, 13, 187
[16] Petkovic R.A., Luton M.J., Jonas J.J., Grain size on high-temperature field strength of poycrystaline copper, Material Science, 1979, 13, 569
[17] Feng F., Texture inheritance of cold drawn pearlite steel wires after austenitization, Materials Science&EngineeringA618(2014)505–510
[18] Zhang M.-X., Kelly P.M., The morphology and formation mechanism of pearlite in steel, Material Characterization 60 (2009), 545-554