pearlitic steel, annealing, cementite

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THE EFFECT OF A CHANGE IN RECRYSTALLIZATION ANNEALING TIME ON THE PROPERTIES AND STRUCTURE OF COLD-DRAWN WIRES

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 hardness measurements 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.

1. 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]. Thanks to the phenomenon of plasticity there is possible metalworking like rolling, drawing and pressing. Plastic metal working can be hot (above the recrystallization temperature) or cold (at a temperature below the absolute recrystallization temperature) [4].

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

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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],[6],[7]. Annealing recrystallization, which make plastic the material, needs to be carried out between the successive forming operations in order to continue the plastic working [2].

Although many heat treatment schemes have been developed for pearlitic steel, still little is known about the influence of the different heat treatment parameters on the percentage, properties and morphology of the phases which are the primary determinants of the mechanical and utilitarian properties of this group of steels [8],[9]. Neither there is any information on matching the parameters, taking into consideration the different dimensions of steel wires and their operating conditions [10],[11].

Thus it is essential to optimize the parameters of the interoperation annealing process used in the course of the cold working of pearlitic steel intended for patented wires. The results of such studies will help in the choice of a proper heat treatment technology during and after cold working, resulting in steel wires with possibly the best properties for industrial applications.

2. AIM AND SUBJECT OF STUDIES

The aim of the studies was to determine the effect of a reduction in recrystallization annealing time on the structure and mechanical properties of pearlitic steel for the production of 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 shown in Table 1 and having 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 3.15mm to 1.17mm. 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 2). The heat treatment temperature was selected on the basis of earlier studies [12],[13] and it ensured the complete recrystallization of the deformed material.

Table 1. Chemical composition of pearlitic steel acc. to standard PN-EN 10323:2005 (U)

CHEMICAL COMPOSITION	С	Mn	Cr	Ni	Si	S/P
wt. %	0.83	0.4-0.6	< 0.1	< 0.15	< 0.3	< 0.025

A NIKON ECLIPSE MA200 light microscope with the NIS Elements BR software and a Phenom G2 scanning electron microscope were used to examine the microstructure of the tested steel. The etched specimens were observed under magnifications in the $100 \times$ range.

The hardness of the specimens was measured by the Vickers method using an MMT-X3 microhardness tester in accordance with standard PN-EN ISO 6507-2:1999. The duration of the measurement, performed under the load of 300g, was 15s.

SPECIMEN	MATERIAL CONDITION				
No. 1	MATERIAL IN AS SUPPLIED CONDITION				
No. 2	MATERIAL AFTER PLASTIC WORKING				
No. 3	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/15minutes				
No. 4	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/30minutes				
No. 5	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/45minutes				
No. 6	SPECIMEN No. 2 AFTER ANNEALING AT 700°C/60minutes				

Table 2. Tested specimens

3. DISCUSSION AND TEST RESULTS

The microscopic examinations of the material (in as supplied condition) of the steel wires 3.15mm in diameter showed a structure typical of low-alloy pearlitic steel (Fig. 1). The structure is fine-grained and the size of its precipitates conforms to comparison chart no. 9 of standard PN-84/H-04507. Pearlite observed under metallographic microscope at low magnifications etches grey while at higher magnifications a clearly lamellar structure, in which the etching resistant cementite protrudes above the soft ferrite, becomes visible (Fig. 2). Microscopic examinations of the material of the ϕ 1.17mm wires (specimen no. 2) showed that thanks to the adopted cold drawing scheme strong plastic deformation, in the order of 65%, was obtained (Figs 3 and 4).

As a result of cold (below the recrystallization temperature) deformation almost all the properties of the material change. 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. 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. Annealing recrystallization, aimed at removing the strain hardening of the material and improving its plastic properties, needs to be carried out between the successive forming operations in order to continue the 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.

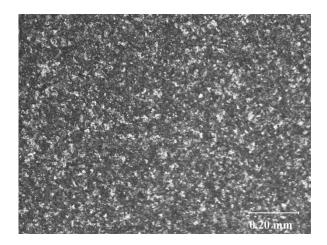


Fig. 1. Material in as supplied condition, specimen no. 1, visible fine-grained structure of pearlitic steel. LM



Fig. 3. Microstructure of specimen no. 2, pearlitic steel after cold working, visible 65% material reduction. LM

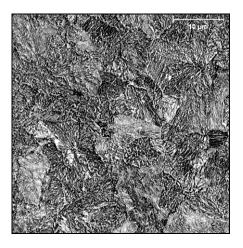


Fig. 2. Magnified area of structure shown in Fig. 1, visible distinct lamellar structure of pearlite. SEM

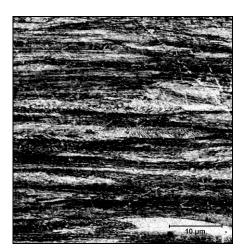


Fig. 4. Magnified area of structure shown in Fig. 3, visible strong plastic deformation of material. SEM

Microscopic examinations of specimens no. 3-6 did not show any differences in material structures obtained as a result of recrystallization annealing over different times (Figs 5-8). Each of the proposed heat treatment schemes resulted in a fully recrystallized homogenous fine-grained structure of the pearlitic steel. 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],[3],[4]. The lower grain size in comparison with that of the material in as supplied condition is the consequence of the recrystallization of the strongly deformed material and it is always consistent with the principle: the greater the reduction, the finer the grains [2],[3],[4].

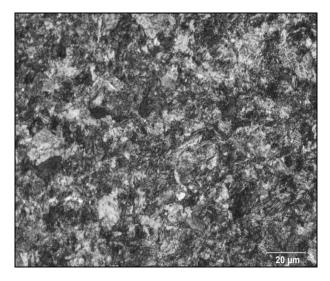


Fig. 5. Material of specimen no. 3, visible recrystallized fine-grained structure of pearlitic steel. LM



Fig. 6. Structure of material of specimen no. 4 after 30 min long recrystallization annealing, visible distinct lamellar structure of pearlite. LM

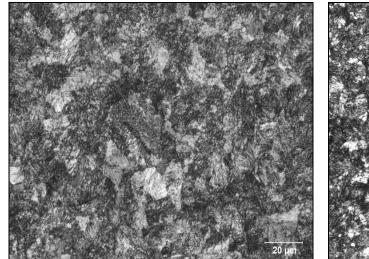


Fig. 7. Microstructure of specimen no. 5, pearlitic steel after heat treatment, visible full recrystallization of the material. LM

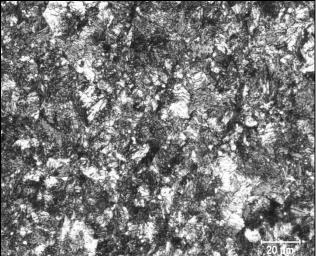


Fig. 8. Material of specimen no. 6 after 60 minutes of recrystallization annealing, visible fine-grained structure typical of pearlitic steel. LM

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. The size of the lamellas in all the specimens was similar, ranging from 0.29 to 0.48 μ m (Figs 9-12). Since the interlamellar distance in pearlite is a major structural parameter having a direct bearing on material hardness it could be expected that hardness measurements would indicate similar values for all the tested materials.

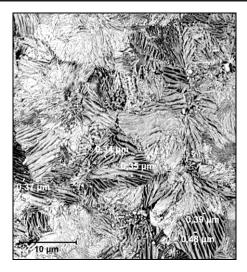


Fig. 9. Magnified area of structure shown in Fig. 5, visible distinct lamellar structure of pearlite and marked interlamellar distances ranging from 0.34 to 0.48μm. SEM

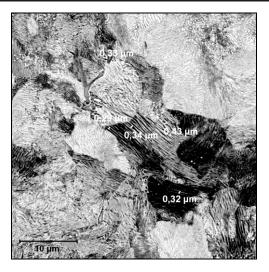


Fig. 10. Material of specimen no. 4, visible cementite lamellae sticking out of soft ferrite, and interlamellar distances ranging from 0.29 to 0.43µm. SEM

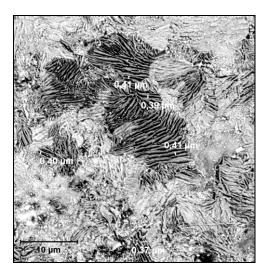


Fig. 11. Microstructure no. 5, pearlitic steel after heat treatment, visible lamellar structure typical of pearlitic steel, interlamellar distances ranging from 0.39 to 0.41µm. SEM.

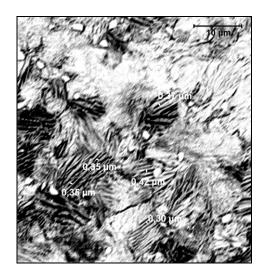


Fig. 12. Magnified area of structure shown in Fig. 8, visible full recrystallization of material, pearlite interlamellar distances ranging 0.30-0.42µm. SEM

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.

No.	HARDNESS HV					AVERAGE HV
Nr 1	312	335	320	318	338	325
Nr 2	552	544	550	575	536	551
Nr 3	352	371	334	325	348	346
Nr 4	353	358	371	383	368	367
Nr 5	350	365	346	371	352	357
Nr 6	359	364	349	368	377	363

Table 3. Results of hardness measurements for tested specimens

The test results showed that material plasticization occurs for all the adopted recrystallization annealing times. The hardness of specimen no. 2 (plastically deformed steel) amounts to 551 HV while that of the material of the specimens after heat treatment ranges from 346 to 367 HV (Table 3). 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.

4. 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.

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 results of the hardness measurements and microscopic observations, presented in this paper have shown that there is no need to apply long recrystallization treatments to such small-diameter elements as the tested steel wires. In the analyzed case, a 15-minute-long heat treatment is quite sufficient for the full recrystallization of the previously deformed material to take place. 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.

However, one should bear in mind that hardness measurements do not provide conclusive evidence about the ductility and strength of the material. In order to conclusively determine which of the proposed heat treatment schemes is proper for fine cold-drawn wires one would have to carry out tensile and fatigue strength tests and determine the other properties of the material. Only on the basis of such comprehensive results one could make a decision as to the technology of producing patented steel wires.

REFERENCES

- [1] ASHBY M.F., JONES D.R.H., 2005, Engineering Materials2: an Introduction to Microstructures, Processing and Design, Elsevier, Oxford.
- [2] DOBRZAŃSKI L.A., 2006, Materials engineering and design materials, WNT, Warszawa, (in Polish).
- [3] DOBRUCKI W., 1975, *Outline of metal forming*, Wyd. Śląsk, Katowice, (in Polish).
- [4] LANGFORD G., 1977, Deformation of Pearlite, Metall Trans A, 8A (6).
- [5] HERTZBERG R.W., 1989, *Deformation and fracture mechanics of engineering materials*, John Wiley & Sons, New York.
- [6] CORDIER-ROBERT, C., FORFERT B., FUNDENGERGER J-J., TIDU A., 2008, *Influence of torsion deformation on microstructure of cold-drawn pearlitic steel wire*, Journal of Materials Science, 43/4, 1241–1248.
- [7] IZOTOV, V.I., POZDNYAKOV V.A., LUK'YANENKO E.V., USANOVA O.YU., FILIPPOV G.A., 2007, *In-fluence of the pearlite fineness on the mechanical properties, deformation behavior, and fracture characteristics of carbon steel*, Physics of Metals and Metallography, 103/5, 519–529.
- [8] HENKEL D., PENSE A.W., 2002, *Structure and properties of engineering materials*, The McGraw-Hill Higher Education, Singapore.
- [9] VANDER VOORT G.F., (ed), 2004, *Atlas of time-temperature diagrams for iron and steels*, ASM International, Materials Park, OH.
- [10] IVANISENKO Y., LOJKOWSKI W., VALIEV R.Z., FECHT H.J., 2003, The mechanism of formation of nanostructure and dissolution of cementite in a pearlitic steel during high pressure torsion, Acta Materialia, 51/18, 5555–5570.
- [11] ZHANG M.X., KELLY P.M., 2009, *The morphology and formation mechanism of pearlite in steels*, Materials Characterization, 60/6, 545-554.
- [12] GRYGIER D., RUTKOWSKA-GORCZYCA M., JASIŃSKI R., DUDZIŃSKI W., 2014, Microstructure evaluations in pearlitic steels wires resulting from modification of the interoperation annealing process, Journal of Machine Engineering, 14/1, 53–62.
- [13] GERSTEIN G., et al, 2014, Structural evolution of thin lamellar cementite during cold drawing of eutectoid steels, Procedia Engineering, 81.