The effectiveness of recrystallization high carbon steel with regard to the condition of material

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Abstract: The main aim of the studies presented in paper was to optimize the parameters of the recrystallisation annealing process used in the course of cold plastic working of high carbon steel for patented wires. Object of the research described in the work was eutectoid steel of 0,8% content of C designated for production of cold-drawn wires applied for reinforcement of car tires, hoses and ropes. The results of tests show that the condition of the material and mainly degree of plastic deformation has a clearly influence on the effectiveness of pearlitic steel recrystallisation

Keywords: recrystallisation, pearlitic steel, annealing, cementite

1. Introduction

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 [1]. 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.

In industrial conditions the recrystallisation annealing consists in heating up the previously cold plastically deformed metal to the temperature higher from recrystallisation temperature, keeping it in that temperature and then cooling [2]. For selecting the proper annealing temperature the recrystallisation diagrams are helpful where the experimentally determined relationships between grain size, recrystallisation temperature and the degree of plastic deformation are presented [3]. These are the spatial diagrams and usually they are made for one-hour annealing. The recrystallisation temperature may also be determined from a curve presenting hardness changes for cold plastically deformed metal and next annealed for one hour depending on the annealing temperature [2]. Then, as the recrystallisation temperature the point of inflection at the hardness curve is assumed.

The recrystallisation process however is influenced by many factors which greatly hinders optimization of that process; among others these are: chemical composition of an alloy, metallurgic purity, phase composition, the structure before deformation and degree of squeeze [4, 5]. Influence of the structure and purity of alloy is the subject of many research works, which however do not clarify this phenomenon unequivocally [5, 6]. 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 [7]. 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 [8].

Among the conditions of plastic deformation the strongest impact on the course of recrystallisation, in particular on its temperature, makes deformation [9]. 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 [10].

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 [10, 11]. 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 [10-13].

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 patented 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 the condition of the material. 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.

2. Materials and methods

Purpose of the research presented in the work was optimization of parameters of the inter-operation annealing process, applied during cold plastic working operations for the pearlitic steel designated for wires. The studies conducted enabled determining of influence condition of the material and mainly degree of plastic deformation on share, properties and morphology of phases, which essentially decide on mechanical and utility properties of that group of steels.

Object of the tests was pearlitic steel of chemical composition and mechanical properties according to the PN-EN 10323:2005 (U) standard. Specimens for tests were prepared in the form of steel wires from 2,00 mm to 0,5 mm diameter, obtained after subsequent stages of cold plastic working and after heat treatment processes involving one-hour inter-operation recrystallisation annealing in temperatures of 700°C (Tab. 1).

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 NIKON ECLIPSE MA200 light microscope and a Phenom G2 scanning electron microscope was applied. Observations were performed at magnifications from the range of $100x \div 6000x$.

Tab.1. The sequence of samples investigated and documented within this article.

SAMPLE	STATE OF MATERIAL				
No. 1	material after plastic working with diameter 2.0 mm				
No. 2	material after plastic working with diameter 0.5 mm				
No. 3	sample No. 1 after annealing at 700°C/60minutes				
No. 4	sample No. 2 after annealing at 700°C/60minutes				

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 L0 = 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 Rm, Young's modulus E, as well as the percent reduction Z and extension A of area after fracture.

3. Results

Microscopic observations of the material of sample No. 1 and 2 in the non etched state have shown presence of the very small number of non-metallic inclusions in the form of oxides. 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. Pearlite observed under the scanning electron microscope at greater magnifications appears as a clear lamellar structure in which hard and hardly etching cementite lamellas protrude over the soft ferrite.



Fig. 1. Microstructure of specimen no. 1, pearlitic steel after cold working, visible 30% material reduction. LM.



Fig. 2. Magnified area of structure shown in Fig. 1, visible low plastic deformation of material. SEM.



Fig. 3. Microstructure of specimen no. 2, pearlitic steel after cold working, visible 85% material reduction. LM.



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

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 clearly plastic deformation amounting to some 30% for sample No. 1 and 85% for sample No.2 (see Fig. 1-4).

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 [1]. 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 the literature data describing technological process of patented wires of pearlitic steel, the recrystallisation annealing temperature should amount from about 520 to 700°C, and the time of the operation should not exceed one hour [8, 14, 15].

The microscopic tests of the heat treated specimens have shown that at 700°C 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 (Fig. 5, 7). Length scales of the observed structures in this specimen were different, grain size for sample No.3 was equal of standard No. 9 and for sample No. 4 to the standard No. 10 according to the PN-EN ISO 634:2005 (see Fig. 6, 8). Differences in the phase morphology result from the fact that the material of specimen No. 4 was strongly plastically deformed.

Microscopic observations of structure of the material subjected to various schemes of heat treatment enable only statement of correctness of the selected parameters. In order to fully express the opinion on the results of given processes the mechanical properties of the tested specimens should be analysed. In the industrial practice the basic parameter indicating for correctness of the selected recrystallisation annealing is measurement of the material hardness. The property is used particularly because of the relative ease of making measurement and lack of special requirements for specimen preparation. For that reason, in order to initially evaluate the influence of condition the material on mechanical properties of pearlitic steel designated for wires the Vickers microhardness was applied.



Fig. 5. Microstructure of specimen No. 3, visible correct structure of pearlite. LM



Fig. 6. Magnified area of microstructure shown in Fig. 5. SEM



Fig. 7. Microstructure of specimen No. 4, visible

fully recrystalised microstructure of pearlite. LM



Fig. 8. Magnified area of microstructure shown in Fig. 7. SEM

The obtained test results have shown that in the both cases the plasticisation of the material takes place. Microhardness of specimen No. 1, i.e. the 30% plastically deformed steel amounted to 387 HV0,3, and the material of specimens after heat treatment ranged was about 270 HV0,3. At the same time it was observed that microhardness of the specimen with 85% plastic deformation was 551 HV0,3, and the material of this specimens after heat treatment ranged was about 363 HV0,3 (Tab. 2). It results from the fact that smaller grain size are featured with significantly lower hardness than the coarse-grained structures, however, it has to be remembered that this does not mean they have better plastic and strength properties.

Tab. 2. Results of material microhardness measurements for tested samples.

SPECIMEN		HAR	AVERAGE HARDNESS			
No. 1	379	381	382	392	403	387
No. 2	552	544	550	575	536	551
No. 3	261	271	278	263	276	270
No. 4	359	364	349	368	377	363

As already mentioned above, hardness measurement does not forejudge on ductility and strength of a material. In order to unmistakably determine the influence of the material condition and mainly degree of plastic deformation, as a result of the static tensile test the remaining properties of the tested specimen material were determined the tensile strength Rm, Young's modulus E, as well as percent reduction Z and extension A of area after fracture.

Results of the studies clearly show that recrystalisation annealing of material with 85% degree of plastic deformation finally leads to obtaining steel of distinctively higher tensile strength Rm = 985 MPa and at the same time high ductility, area reduction Z = 37% and area extension A = 5,2% (Tab. 3). In case of recrystallisation annealing of steels in lower degree of deformation high ductility Z = 41% and A = 7,8% was observed but at the same time much lower material strength, Rm = 667 MPa (see Tab. 3).

Table 3. Results of measurements of strength and	
plastic properties of the tested specimens.	

No.	R _m [MPa]	E [MPa]	Z [%]	A [%]
No. 1	1382	1,75.105	35,6	3,9
No. 2	2221	1,70.105	30,5	2,0
No. 3	667	1,76.105	41,3	7,8
No. 4	985	1,78·10 ⁵	37,1	5,2

5. Conclusions

Pearlitic steels containing from about 0,8 to 0,95 % C belong to the group of unalloyed steels of the quality class designated for cold drawing or rolling. At the same time the steels are characterised with particularly low share of non-metallic inclusions and limited content of chromium and nickel which elongate the time of pearlitic conversion. They found application mainly as wire rods for rope and tire reinforcement wires.

Despite many known schemes of heat treatment for pearlitic steel there is still insufficient literature data on the influence of heat treatment working parameters on the share, properties and morphology of phases. There is also lack of sufficient information on selection of those parameters, taking into consideration different degrees of steel rod deformation, specific applications of products and their operating conditions.

Research results presented in the work have shown that the condition of the material and mainly degree of plastic deformation has a clearly influence on the effectiveness of pearlitic steel recrystallisation. The microscopic tests of the heat treated specimens have shown that at 700°C full recrystallisation of material in both cases 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 it was observed differences in the phase morphology, grain size for sample No.3 equal of standard No. 9 and at the same time for sample No. 4 to standard No. 10 according to the PN-EN ISO 634:2005 standard. The lower length scales of the structures in specimen No. 4 it result from the fact that the material of this specimen was strongly plastically deformed.

Results of hardness measurements have shown that for both cases the applied recrystalisation annealing decrease the hardness from 551 HV0,3 to even 270 HV0,3. The hardness of the specimen with 85% plastic deformation after heat treatment was about 363 HV0,3 and the same time for sample with 30% plastic deformation after heat treatment was about 363 HV0,3. It results from the fact that smaller grain size are featured with significantly lower hardness than the coarse-grained structures, however, it has to be remembered that this does not mean they have better plastic and strength properties.

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