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Optimization of the Inter-Operation Annealing of Cold-Drawn Eutectoid Steels

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The test of the results presented in this paper has demonstrated that the most suitable recrystallizing annealing temperature for cold drawn eutectoid steels is 700 °C. The microscopic examinations, with the use of scanning electron microscopy and transmission electron microscopy and the results of mechanical properties tests have shown that only at this temperature it is possible to obtain the fully recrystallized homogeneous fine-grained structure of the pearlitic steel.

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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.

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, structure before deformation, conditions of deformation.

Influence of the structure and purity of alloy is the subject of many research works, which however do not clarify this phenomenon unequivocally [2, 3]. 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 [4].

Among the conditions of plastic deformation the strongest impact on the course of recrystallisation, in particular on its temperature, is made by deformation [4, 5]. 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 [6].

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. Numerous studies of the recrystallisation process of cold worked steel show clear influence of the heating rate applied during annealing [7, 8]. 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.

Object of the research described in the work was eutectoid steel of 0.8% C designated for production of colddrawn wires applied for reinforcement of car tires, hoses, and ropes. In accordance with the patent US4759806 A the inter-operation annealing temperature should range from about 520 to 700 °C, and the time of annealing should not exceed one hour [1]. 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 [2–5].

2. Materials and methods of investigations

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 patented wires. The studies conducted enabled determining of influence of heat treatment 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 of 0.8 mm diameter, obtained after subsequent stages of cold plastic working and after heat treatment processes involving onehour inter-operation recrystallisation annealing in temperatures ranging within 550–700 °C.

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3. 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.



Fig. 1. Fig. 1. Microstructure of the tested material in non-etched state, visible small number of non-metallic inclusions in the form of oxides. LM.



Fig. 2. Microstructure of pearlitic steel after cold plastic working operations, visible 90% squeeze of the material. SEM.

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. The rectangular shape of pearlite colonies indicates that colonies growth took place at a stress state in all the surrounding volume. 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). Length scale of the observed structures in this specimen was: ferrite lamellas of about 40–50 nm and cementite lamellas of about 7–10 nm (Fig. 3).



Fig. 3. Magnified area of structure shown in Fig. 2, visible light ferrite lamellas and dark cementite lamellas. TEM.



Fig. 4. Microstructure of specimen subjected to recrystallisation annealing in temperature of 600 °C, visible coagulation of cementite and the texture of recrystallisation. SEM.

Lamellar structure of pearlitic steel previously deformed plastically, subjected to recrystallisation annealing in the lower range of temperature, i.e. in 550 °C, 600 °C, and 650 °C clearly undergoes coagulation (Fig. 4). Size of the observed coagulated precipitations of cementite was diversified and ranged from 50 nm to 500 nm, which indicates for additional influence of unknown and at the same time not analysed in the work factors on growth of individual particles and course of the spheroidising process (see Fig. 5). Moreover, it was observed that in this temperature range a distinct texture of recrystallisation appears which may result with anisotropy of mechanical properties of the material and even lead to wire cracking during further cold plastic working operations. In summary, the microscopic tests performed show that heat treatment in the lower temperature range, as indicated by the standard, being supposedly the recrystallisation annealing, became the spheroidising annealing.



Fig. 5. Magnified area of structure shown in Fig. 4, visible coagulated precipitations of cementite. TEM.



Fig. 6. Microstructure of specimen subjected to recrystallisation annealing in temperature of 700 °C, visible correct structure of pearlite. SEM.

Further microscopic tests of the heat treated specimens have shown that full recrystallisation of material takes place only at 700 °C, in that temperature the structure banding radically disappears and the precipitated cementite has typical lamellar form indicating the full and correct recrystallisation of the material (Fig. 6). Length scales of the observed structures in this specimen were different like materials in the as delivered condition: pearlite colony 4 μ m to 9 μ m and cementite lamellas about 50–70 nm (see Fig. 7). 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.



Fig. 7. Magnified area of structure shown in Fig. 6, visible typical lamellar form of cementite precipitated. TEM.

Results of hardness measurements have shown that in all the proposed temperatures of annealing a decrease in hardness from 527 Hv0.5 to even 288 Hv0.5 is following. It has even been observed that hardness of specimens heat treated in lower temperatures is significantly lower from those obtained for 700 °C, however it results from the fact that cementite structures of spheroidal character are much more plastic than the lamellar ones (see Table I).

TABLE I

Results of mechanical properties of tested specimens.

Specimen	Rm	E [MPa]	Z	Hardness
	[MPa]		[%]	Hv0.5
mater. after plastic working	2221	1.70×10^{5}	15	527
mater. after anneal. $550^{\rm o}{\rm C}/1{\rm h}$	971	1.85×10^{5}	29	294
mater. after anneal. $600^{\rm o}{\rm C}/1{\rm h}$	1071	1.88×10^{5}	25	288
mater. after anneal. $650^{\rm o}{\rm C}/1{\rm h}$	1042	1.84×10^{5}	27	305
mater. after anneal. $700^{\rm o}{\rm C}/1{\rm h}$	685	1.60×10^{5}	35	352
mater. after anneal. 650 °C/1h mater. after anneal. 700 °C/1h	1042 685	1.84×10^{5} 1.60×10^{5}	$\frac{27}{35}$	305 352

In order to univocally determine at that stage of the research which of the proposed schemes of heat treatment is proper for cold drawn wires it was necessary to perform static tensile test and determine remaining properties of the material. Results of the studies have conformed that the most suitable temperature for annealing is 700 °C, the process finally leads to obtaining steel of

distinctively lover tensile strength, material after plastic working $R_m = 2221$ MPa, material after annealing in 700 °C $R_m = 685$ MPa and at the same time higher ductility, area reduction for specimen after plastic working Z = 15% and for specimen after annealing in 700 °C Z amounted to 35% (see Table I).

4. Conclusion

Research results presented in the work have shown that from all the proposed schemes of heat treatment of the plastically deformed pearlitic steel the most suitable temperature of recrystallisation annealing is 700 °C. Microscopic observations have shown that only in that temperature it is possible to obtain typical, recrystallised lamellar structure of pearlite without recrystallisation texture. Heat treatment in lower temperatures from the range of 550–650 °C, has not fully eliminated the banding character of microstructure, the effect of which could be anisotropy of mechanical properties of finished products, and first of all it led to coagulation of cementite precipitations, changing by this the operation of recrystallisation annealing into the spheroidising process.

The results of mechanical properties tests have confirmed that the most suitable annealing temperature is 700 °C, the process finally leads to obtaining steel of clearly lower tensile strength in relation to the material after treatment and at the same time of high ductility. In case of steel recrystallisation annealing operations in lower temperatures of 550-650 °C a high ductility has been indeed observed, but at the same time high material strength and clear increase in the Young modulus value were observed which finally disqualifies that range of heat treatment temperatures for cold drawn wires of pearlitic steel.

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