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CHARACTERIZATION OF NANOBAINITIC STRUCTURE OBTAINED IN 100CrMnSi6-4 STEEL AFTER INDUSTRIAL HEAT TREATMENT

CHARAKTERYZACJA STRUKTURY NANOBAINITYCZNEJ WYTWORZONEJ W HANDLOWEJ STALI ŁOŻYSKOWEJ – 100CrMnSi6-4 W PRZEMYSŁOWEJ OBRÓBCE CIEPLNEJ

The aim of the work was to produce a nanobainitic structure in the commercial bearing steel – 100CrMnSi6-4 and to characterize its structure and mechanical properties.

In order to produce this structure the austempering heat treatment was performed, with parameters that have been selected on the basis of dilatometric measurements of phase transformation kinetics in steel. The heat treatment process was performed in laboratory as well as in industrial furnaces. The obtained structure was characterized using transmission electron microscopy. In order to investigate the effect of the microstructure parameters on the material's mechanical properties, the hardness, impact strength and static tensile tests have been conducted.

Keywords: austempering, nanobainitic structure, bearing steel

W niniejszej pracy podjęto próbę wytworzenia struktury nanobainitycznej w handlowej stali łożyskowej – 100CrMnSi6-4.

W celu wytworzenia tej struktury przeprowadzono proces hartowania z przystankiem izotermicznym, którego parametry zostały określone w oparciu o badania dylatometryczne. Warto podkreślić, że proces ten przeprowadzono w piecach przemysłowych. Uzyskaną strukturę nanobainitu scharakteryzowano za pomocą transmisyjnego mikroskopu elektronowego. W celu zbadania wpływu wytworzonej mikrostruktury na właściwości mechaniczne przeprowadzono badania twardości, udarności i statyczną próbę rozciągania.

One of the steel development directions still pursued nowadays is the strive to obtain a material with high strength combined with high ductility and fracture toughness. The works carried out in the teams of Bhadeshia, Caballero and coworkers prove that it is possible to obtain steels with a highly advantageous combination of mechanical properties by creating a special type of bainitic structure in steel. As an example there are results described in the patent no. US20110126946 (2011): hardness of 590-690HV, ultimate tensile strength (UTS) up to 2100 MPa, impact strength: 4-7J and the total elongation (TE) 3-11% [1]. The other reference papers reveal that by using higher content of alloying elements it is possible to obtain even greater values of mechanical parameters, such as e.g. the ultimate tensile strength up to 2500 MPa, fracture toughness up to 130 MPa$\sqrt{m}$ (steel with 3.5% nickel addition) or the total elongation up to 30% [2-7].

The structure guaranteeing those unique mechanical properties is the nanocrystalline bainite significantly differing from the traditional bainite in terms of its microstructure. The nanobainitic structure is a mixture of two phases: a hard bainitic ferrite and ductile carbon enriched retained austenite. Such structure does not contain a cementite precipitations which are present in lower or upper bainite. In the nanobainitic structure the width of bainitic ferrite plates as well as retained austenite layers is below 100nm. This type of structure can be obtained in steels with a specific chemical composition containing, among others, increased amount of carbon (0.6-1.1%) and silicon (1.5-2% Si) that hinders the cementite precipitation [8-9].

The nanobainitic structure is created by thermal treatment, and more specifically during the isothermal quenching process. For example, in steels with high carbon and silicon content, a carbide-free nanobainitic structure with the ferrite plates about 20-40nm thick [10] was formed during the austempering process in temperatures ranging from 200 to 300°C.

The formation of a nanobainitic structure takes place during the austempering process which is the final stage of heat treatment. It means that this process can be carried out for finished components of any shape.

Moreover, there are the first attempts made at obtaining the nanobainitic structures in large size components at the industrial scale [11].

The present work is aimed at obtaining the nanobainitic structure in a commercial 100CrMnSi6-4 steel by use the austempering process with specially designed parameters in

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industrial furnaces. The obtained microstructure has been characterized and the mechanical properties has been determined.

1. Material and methodology of investigations

The material used for the investigations was commercially available 100CrMnSi6-4 steel used in production of rolling bearings components, with the chemical composition presented in Table 1. It should be noted that this steel has high carbon, but low silicon content.

TABLE 1

Chemical composition of 100CrMnSi6-4 steel according to PN-EN ISO 683-17[12] in weight percentage

C	Si	Mn	Cr	P	S	Mo
0.93 - 1.05	0.45 - 0.75	1.00 - 1.20	1.40 - 1.65	Max. 0.025	Max. 0.15	Max. 0.10

The steel bars were delivered in the softened state which is confirmed by the structure composed of spheroidal carbides in the ferritic matrix.

The isothermal process parameters were determined basing on the dilatometric tests thanks to which it was possible to control many parameters of heat treatment with high precision. The heat treatment consisted on austenitization at 930°C for 30 minutes, with subsequent cooling to 320°C, with the rate which enabled to avoid diffusional transformations (perlite transformation), followed by isothermal holding in that temperature for 5 hours and slow cooling down to ambient temperature. The isothermal holding time enabled for the total completion of the bainitic transformation. The heat treatment process with analogous parameters was applied to the large-size specimens to be used for mechanical tests. At first, the process was carried out in laboratory furnaces. In order to check the repeatability of the technology developed, the process was also carried out under industrial conditions. For the process, a SecoWarwick VPT15 4022/24IQN single-chamber vacuum furnace was used, with the work chamber sized 400×400×600 mm. The maximum charge for this furnace is 200 kg, with about 2 kg of this charge being the tested steel. The entire process (austenitization and isothermal holding) took place in a single chamber of the furnace, with no need to move the charge.

The procedure of cooling down was performed by nitrogen injecton (high pressure below 15 bar). Following the isothermal process, the specimens were subjected to microstructural investigations and to mechanical tests.

The microstructure was investigated using a JEM3010 transmission electron microscope (TEM) with the accelerating voltage of 300 kV. The specimens observed using TEM was sampled from two spots, at the edge and from the central part of a cylindrical specimen 15 mm high and with the diameter of 25 mm (Fig. 1a). The specimens were prepared by electropolishing method after cutting into slices 3 mm in diameter and the thickness of 70-100 μm with an electrical spark and wire saw. The structural characterization was performed for several thin foils from different heights of the specimen in order to check the degree of microstructural homogeneity in the volume of a large-size specimen (Fig. 1b).

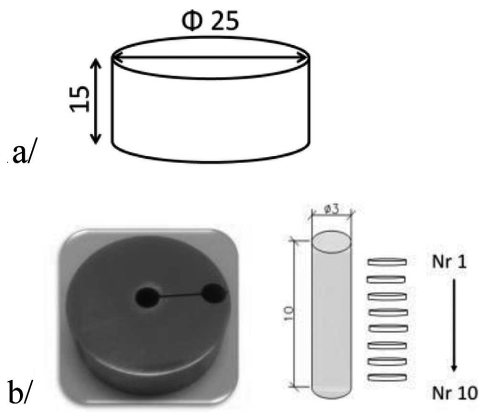

Fig. 1. The scheme of: a) specimen dimensions in millimeters and b) sites where the material for tests was sampled from

Basing on the images obtained, the widths of bainitic ferrite plates (Lα) and austenite layers (Lγ) were measured. The measurement was made perpendicularly to the plate length which made it possible to determine their actual thickness basing on the equation Eq. 1:

$$d = L \cdot \frac{2}{\pi} \tag{1}$$

where:

d – real thickness of the plate;

L – width of the plate measured on the image obtained by TEM. [13]

About 100 measurements were made for each phase on each of the analysed images. Those values were averaged and d values were calculated in accordance with the above-mentioned formula.

Basing on the images taken with the transmission electron microscope, the volume fractions of various structural constituents present in the obtained microstructure were determined.

The tests of mechanical properties included the measurement of hardness, impact strength and static tensile test. All the tests were carried out in ambient temperature. The hardness was measured by Vickers method with the load of 2 kg. The impact strength test was carried out using the Charpy impact testing machine for specimens with a V notch. The static tensile test was carried out on a Zwick/Roell Z250 universal testing machine using an extensometer.

2. Results and discussion

2.1. Microstructure characterization

The process parameters were designed on the basis of the dilatometric tests which is why the structure obtained in the process carried out in the dilatometer was used as a reference one.

TEM images of the microstructure formed through the austempering processes conducted in the dilatometer and in the industrial furnace are presented on Fig. 2a and Fig. 2b respectively. In both cases the nanobainitic structure was formed.

This microstructure was composed of the mixture of two phases, that is bainitic ferrite (light phase) and carbon enriched retained austenite (dark phase).

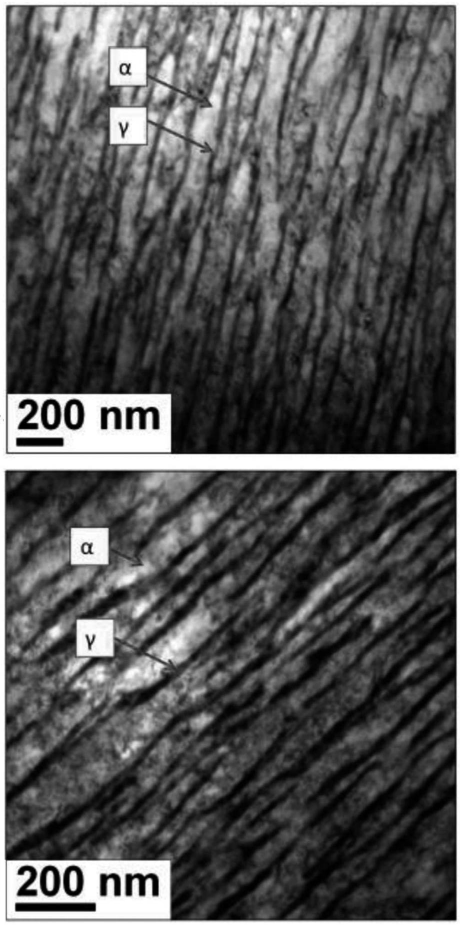

Fig. 2. The nanobainitic structure (light phase – bainitic ferrite, dark phase – retained austenite) obtained in the processes with identical treatment parameters in two various devices: a) in dilatometer, b) in industrial furnace

The analysis of the TEM images indicates that process carried out in the dilatometer led to a microstructure with about 80-90% of a nanobainite (the structure where both the width of bainitic ferrite and austenite plates is smaller than 100 nm), 8% of a carbide-free bainite (the structure where the width of bainitic ferrite or austenite plates is above 100 nm – example, Fig. 3a), and the remaining 2% were carbides. For the structure obtained in the industrial furnace, the content of a nanobainitic structure was slightly reduced and was about 70-80%, while the carbide-free bainite structure content was increased to about 10%, with the remaining 10% for carbides. It should be emphasized that no blocky austenite was observed in the examined microstructures.

Small spheroidal carbides, as revealed in TEM observations, with the diameter below 500 nm (Fig. 3b) are chromium enriched cementite particles, which were not dissolved completely during the austenitization process performed at 930°C. This can result from high chromium content (about 1.5%) in 100CrMnSi6-4 steel which leads to a high volume fraction of carbides. These carbides are hard and contribute to an increase in the hardness and in the wear resistance of steel, especially when it underwent a quenching and tempering heat treatment [14]. It is, however, worth to notice that theses carbides do not make notches as they are spherical and relatively well embedded in the nanobainitic structure.

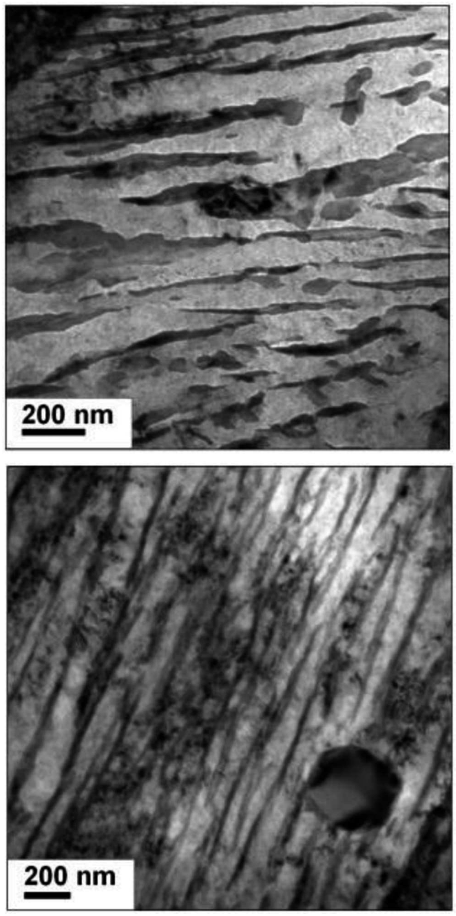

Fig. 3. a) Sample carbide-free structure (that is the structure with the bainitic ferrite or austenite plate width above 100nm), b) carbide found in the nanobainitic structure formed in the industrial furnace process

The analysis of TEM images revealed also, that the retained austenite volume fraction amounts below 35%.

In Table 2 there are the values of bainitic ferrite and retained austenite plates/layers ($L\alpha$ and $L\gamma$) widths measured on recorded TEM images. The evaluated real plates/layers thickness values ($d\alpha$ and $d\gamma$) are also indicated.

TABLE 2

Width of bainitic ferrite (α) and austenite (γ) layers in TEM images (L value) and the evaluated actual plate width (d value) basing on the images of the structures obtained in the dilatometer and industrial furnace process

	Dilatometer	**Industrial furnace**
Lα [nm]	69.2±20.6	53.7±20.0
Lγ [nm]	25.0±7.5	22.6±5.7
dα [nm]	44.1±13.1	34.2±12.8
dγ [nm]	15.9±4.8	14.4±3.6

The obtained results clearly indicate, that in both thermal processes a nanobainitic structure was obtained as the plate widths of the bainitic ferrite plates and retained austenite layers were well below 100nm in both cases. What is more, according to Tab. 2, a finer structure was obtained in the process in industrial furnace. It should be underlined, that the nanobainitic structure has been documented in all investigated areas of steel samples. It means that the microstructure formed during the austempering heat treatment is homogeneous across the thickness of the samples.

2.2. Mechanical properties

Microscopic observations confirmed that the nanobainitic structure was formed in the large-size specimens subjected to isothermal quenching with the adopted parameters. This permitted us to assess the mechanical properties of steel with the formed nanobainitic structure.

The measurements of hardness and impact strength revealed the following values: hardness=540±3.4HV2 and impact strength 9±1.5J.

In Fig. 4 there are the results presented for the static tensile test for 5 specimens. The analysis of the results obtained proves that the mechanical behavior of all specimens was similar, that is they were broke under similar strain and stress values.

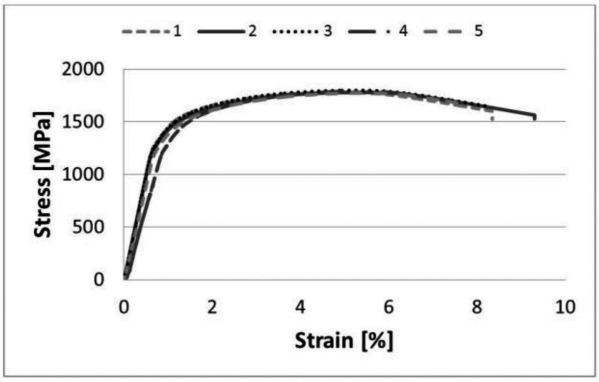

Fig. 4. Diagram of the interdependence between the stress and strain for specimens from 100CrMnSi6-4 steel with a nanobainitic structure

Summing up the results of mechanical tests for 100CrMnSi6-4 steel with a nanobainitic structure, it can be stated that this material is characterized by the hardness of 540HV2, impact strength of 9J and the yield strength YS=1370 MPa, ultimate tensile strength UTS = 1780 MPa and the total elongation of 7%.

3. Summary and conclusions

The results presented in this work proved that a nanobainitic structure has been obtained in 100CrMnSi6-4 steel by the use of isothermal quenching process with appropriately designed thermal treatment parameters. The TEM observations revealed that the applied thermal treatment conditions ensure formation of a similar structure both in the dilatometer furnace and in the industrial furnace. The TEM images of specimens derived from various depths of the large-size specimens showed that a repeatable microstructure, composed primarily of the mixture of bainitic ferrite and retained austenite was obtained in the entire volume of specimens investigated. The thickness of both phases was well below 100nm and equal to 30-50 nm for bainitic ferrite plates and 14-20 nm for retained austenite layers. Moreover, 100CrMnSi6-4 steel with the nanobainitic structure was characterized with relatively high strength properties with the acceptable ductility and impact strength.

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