Influence of heat treatment on metallographic and mechanical properties of ductile iron

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Abstract: *Due to the mechanical properties (toughness, elongation, tensile strength) that characterize ductile iron, its application in foundry technology is becoming more pronounced every day. The chemical composition and heat treatment of ductile iron have a great influence on the required mechanical properties. Given the operating conditions, the main purpose of heat treatment of ductile iron is to change the desired mechanical properties. Since the specific mechanical properties of ductile iron are generally related to the regularity of the mined graphite nodules, the main objective in production is to produce ductile iron with the highest possible percentage of ductility. In the experimental part of the paper, microstructure and hardness tests were carried out on specimens of ductile iron NL 400 and NL 700 before and after heat treatment by soft annealing (ferritization) and improvement. It was found that the type and corresponding parameters of heat treatment significantly affect the microstructure and the achieved hardness values of the ductile iron test specimens.*

Keywords: *DUCTILE IRON, HEAT TREATMENT, STRUCTURES, HARDNESS*

1. Introduction

Casting is considered one of the oldest and most widely used technologies for forming metal objects. The principle of casting technology is based on molten metal, which solidifies by pouring into the mold, thus maintaining the internal dimensions of the mold cavity after cooling. The main advantages of this technology are certainly the great repeatability of the process and the possibility to produce the most complicated structural parts by casting. In the production of semi-finished products and products by casting technology, there are certain peculiarities in the process itself, which should be well known in order to obtain the required structure and, consequently, the desired properties. Since metal casting is a continuous process, complete control during the process itself is difficult [1]. Nowadays, ductile iron is increasingly used in casting technology, which is still considered a relatively new type of cast iron. Ductile iron has much higher strength than gray iron, but its workability and vibration damping are lower than those of gray iron. A particular advantage of ductile iron over unalloyed steel, as well as the widely used gray iron, is its high value of yield strength. The good mechanical properties of ductile iron are due to the extraction of graphite beads in the Fe-C alloy, which are still in the casting phase. The properties of ductile iron can be subsequently improved by mechanical and heat treatments [2]. In this paper, the heat treatment soft annealing (ferritization) and improvement (hardening + high temperature tempering) are applied to prepared ductile iron specimens NL 400 and NL 700, after which microstructure and hardness tests were performed.

2. Properties, heat treatment and application of ductile iron

From the second half of the 20th century until today, the production of ductile iron has increased exponentially. The mechanical properties (toughness, elongation, tensile strength) of ductile iron indicated its future successful application. Looking at the mechanical properties, ductile iron is classified between steel and gray iron. Ductile iron belongs to the group of castings with high carbon content, whose mechanical properties are characterized by the carbon precipitated in the form of graphite beads. The extraction of the graphite spheres from the original sheet shape is achieved by adding small amounts of e.g. cerium or magnesium to the melt just before the base melt Fe-C is poured into the mold. If the melt contains small amounts of nodulation inhibitors (e.g. lead, titanium) or more than the allowable amounts of sulfur, the carbon will not be precipitated in the form of pellets and ductile iron will not form [3].

In such a case, gray iron is formed, in which the carbon is formed in the form of flakes. Nodular graphite causes ductile iron to be more ductile and tough than gray iron, but the strength and toughness of such cast iron depends much more on the strength of the Fe-C base than, for example, gray iron. The tensile strength of ductile iron ranges from 400 to 800 MPa, but due to heat treatment and alloying of ductile iron, it can reach 1400 MPa. The properties and microstructure of ductile iron are influenced by various factors such as chemical composition, metallurgical processes in the melt, and cooling rate during solidification and solid cooling [2]. The shape of the graphite and the structure of the Fe-C matrix are significantly influenced by the chemical composition. The declared chemical composition of ductile iron is listed in Table 1.

Table 1: *Declared chemical composition of ductile iron* [4].

Chemical composition, %						
C	Si	Mn	P	S	Mg	Fe
3,2 ÷ 3,8	2,4 ÷ 2,6	0,1 ÷ 0,5	0,005 ÷ 0,045	0,002 ÷ 0,01	0,03 ÷ 0,05	rest

Because of its favorable mechanical properties, ductile iron is used as a substitute for cast steel or steel forgings. Typical examples of ductile iron are crankshafts and camshafts, cylinder liners for engines and compressors, gears, valves, piping, plain bearing caps, parts for wind turbines, pump rotors, etc. In the manufacture of ductile iron products, various heat treatment processes are used to simultaneously increase strength and toughness, such as isothermal and classical improvement (hardening and tempering). Ductile iron improvement processes also increase wear resistance. Also, the amount and shape of graphite formed in ductile iron cannot be affected by subsequent heat treatment. Nodularity is determined as the volume fraction of graphite nodule accumulations relative to the total number of graphite accumulations. During production, the aim is to produce ductile iron with as high a proportion of ductility as possible (usually above 90 %), since the specific mechanical properties of ductile iron are generally related to the regularity of the graphite nodules obtained [1]. The mechanical properties, i.e., tensile strength and dynamic resistance, decrease with decreasing percentage of nodularity. In addition, lower fissility affects the modulus of elasticity, lowers toughness and decreases electrical resistance, but increases the ability to dampen vibrations.

3. Experimental part

In the experimental part of the paper, samples of ductile iron NL 400 and NL 700 were tested. After preparation of the test specimens, heat treatments such as soft annealing (ferritization) and improvement (hardening + high temperature tempering) were performed. Microstructure and hardness tests were also carried out.

3.1. Chemical composition test

The chemical compositions of the test specimens were determined using a spectrometric analyzer. Table 2 shows the measured chemical composition of the NL 400 ductile iron specimen, while Table 3 shows the measured chemical composition of the NL 700 ductile iron specimen.

Table 2: *Measured chemical composition of ductile iron NL 400.*

Chemical composition, %						
C	Si	Mn	P	S	Cu	Mg
3,6	2,1	0,14	0,037	0,009	0,077	0,043
Cr	Ni	Mo	Al	V	Ti	Sn
0,036	0,045	0,007	0,012	0,008	0,018	0,007

Table 3: *Measured chemical composition of ductile iron NL 700.*

Chemical composition, %						
C	Si	Mn	P	S	Cu	Mg
3,54	2,072	0,676	0,035	0,008	0,0673	0,05
Cr	Ni	Mo	Al	V	Ti	Sn
0,055	0,051	0,011	0,010	0	0	0

Specimens made of ductile iron NL 400 and NL 700 with dimensions Φ40x20 mm and specified chemical compositions were selected for the test. Before starting the test, the specimens were cut on a metal cutter, which has the possibility of intensive heat dissipation by means of cold water circulation. The MC-80 specimen cutter is shown in Figure 1.

Fig. 1 *Device for cutting specimens.*

3.2. Heat treatment of ductile iron

After cutting the specimens, they were marked according to Table 4, which also lists the parameters of heat treatment (ferritization and improvement) and the designations of certain specimens made of ductile iron NL 400 and NL 700.

Table 4: *Inscriptions and heat treatment parameters (ferritation and improvement) for the specimens NL 400 and NL 700.*

Specimen	Initial condition (at 20 °C)	Ferritization (at 725 °C, cool in the oven to 345 °C	Improvement (quenching from 870 °C and tempering at 565 °C)
NL 400	1.0	1.1	1.2
NL 700	2.0	2.1	2.2

Before heat treatment, metallographic examinations of the microstructure and hardness tests of the initial condition of test specimens 1.0 NL 400 and 2.0 NL 700 were performed. Test specimens 1.1 NL 400 and 2.1 NL 700 were heat treated by soft annealing (ferritization). These specimens were heated continuously from room temperature to 725 °C, at a heating rate v_h = 250 °C/h. After heating to 725 °C, it was necessary to hold 1 h of 1" thickness at the soft annealing temperature.

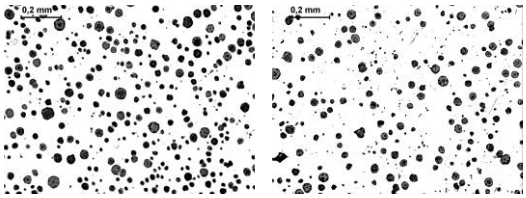
After holding at the soft annealing temperature, the specimens were cooled to 345 °C in an oven at a cooling rate of v_c = 55 °C/h and then in still air. Specimens 2.1 NL 400 and 2.2 NL 700 were processed by the heat treatment process of improvement (hardening + high temperature tempering). Quenching consisted of heating the specimens to an austenitizing temperature of 870 °C, holding at this temperature for 1 h, and then quenching in oil. Quenching of ductile iron castings is usually performed in oil because the castings may crack when cooled in water due to the high stresses. High temperature tempering consisted of heating the specimens from room temperature to 565 °C, heating rate v_h = 250 °C/h. The holding time at 565 °C was 2 h, followed by cooling in an oven to 240 °C (with the aim of relieving stresses, but also hardness), cooling rate v_c = 55 °C/h and cooling from 240 °C to room temperature in still air.

3.3. Microstructure test

Microstructure testing was performed using a Leica DM 2500 M laboratory light microscope connected to a computer to save photos after testing, Figure 2.

Fig. 2 *Light microscope Leica DM 2500 M.*

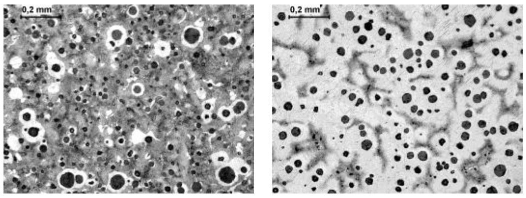
Figure 3.a shows the microstructure of ductile iron in the initial unheated and unetched condition for test specimen 1.0 NL 400, while Figure 3.b shows test specimen 2.0 NL 700. In both cases, it can be seen that the percentage of nodule formation during casting exceeded 90 %.



a) *b)*

Fig. 3 *Microstructure of ductile iron*
a) test specimen 1.0 NL 400; b) test specimen 2.0 NL 700.

Figure 4.a shows the microstructure of ductile iron in the initial unheated and etched condition for test specimen 1.0 NL 400, while Figure 4.b shows test specimen 2.0 NL 700. Etching was performed using a three percent solution of nital. Figure 4.a shows the pearlitic structure of ductile iron NL 400 with characteristic ferritic areas around the sphere (the microstructure consists of pearlite and the area around the sphere is called the "bull's eye"). In Figure 4.b, ductile iron NL 700 has a pure pearlite matrix.



a) *b)*

Fig. 4 *Microstructure of ductile iron*
a) test specimen 1.0 NL 400; b) test specimen 2.0 NL 700.

Figures 5.a and 5.b show the microstructures of ductile iron after soft annealing (ferritization) in the etched condition for specimen 1.1 NL 400, while Figures 5.c and 5.d show specimen 2.1 NL 700. Etching was performed using a three percent solution of nital. In Figure 5.a it can be seen for the specimen of ductile iron 1.1 NL 400 that the size and shape of the nodules did not change after heat treatment by soft annealing (ferritization). A similar, predominantly ferritic microstructure as in Figure 5.a was observed in Figure 5.c for the specimen of ductile iron 2.1 NL 700. Ferrites with traces of pearlite were observed in Figures 5.b and 5.d for the test specimens of ductile iron 1.1 NL 400 and 2.1 NL 700.

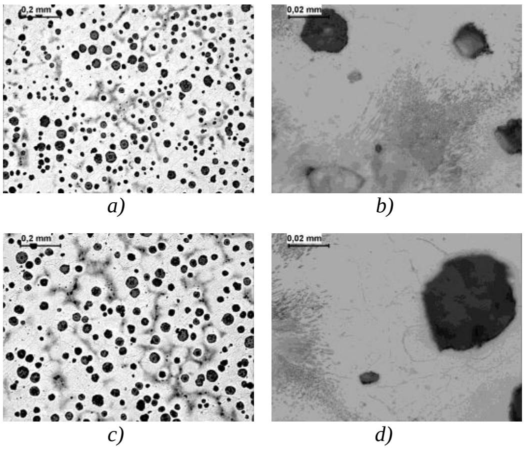


Fig. 5 *Microstructure of ductile iron*
a) b) test specimen 1.1 NL 400; c) d) test specimen 2.1 NL 700.

Figures 6.a and 6.b show the microstructures of ductile iron after heat treatment of the etched improvement for test specimen 1.2 NL 400, while Figures 6.c and 6.d show for test specimen 2.2 NL 700. Etching was performed using a three percent solution of nital. Loose martensite can be seen in the above images.

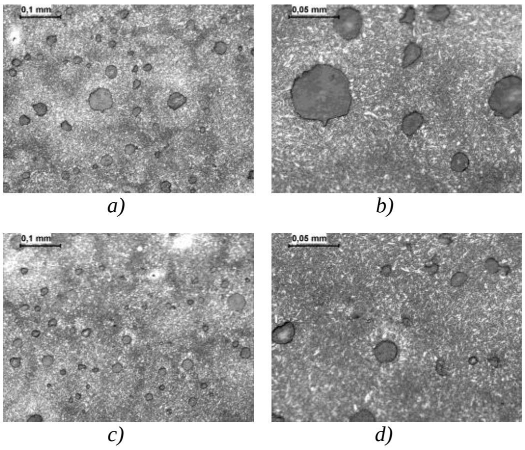


Fig. 6 *Microstructure of ductile iron*
a) b) test specimen 1.2 NL 400; c) d) test specimen 2.2 NL 700.

3.4. Hardness test

The hardness test was performed according to the Brinell method. A steel ball with a 5 mm cross-section was used for the hardness measurements. The diameter of the ball and the injection force in the Brinell hardness measurement are determined using the condition F/D2 = const. which is 30 for steel. The injection force is 750 N for 10 seconds. Table 5 shows the results of hardness measurements on test specimens 1.0 NL 400 and 2.0 NL 700, while Table 6 shows the results of hardness measurements on test specimens 1.1 and 1.2 (NL 400) and 2.1 and 2.2 (NL 700).

Table 5: *Results of hardness measurement on test specimens 1.0 NL 400 and 2.0 NL 700.*

Specimen	Ordinal number of measurements	Hardness HB 5/750/10	Arithmetic value HB
1.0	1	173	172
	2	171	
2.0	1	219	220
	2	221	

Table 6: *Results of hardness measurement on test specimens 1.1, 1.2 (NL 400) and 2.1, 2.2 (NL 700).*

Specimen	Heat treatment	Ordinal number of measurements	Hardness HB 5/750/10	Arithmetic value HB
1.1	Soft annealing	1	143	145
		2	147	
1.2	Improvement	1	288	285
		2	282	
2.1	Soft annealing	1	154	153
		2	152	
2.2	Improvement	1	349	349
		2	349	

4. Analysis of the results and conclusion

In the experimental part of the paper, microstructural investigations and hardness tests were carried out on specimens of ductile iron NL 400 and NL 700 before and after the heat treatment of soft annealing, i.e. subcritical ferritization and improvement. When analyzing the microstructure of ductile iron specimens NL 400 and NL 700 after the heat treatment of soft annealing (ferritization), a change in the size and shape of the ferrite was observed, around which traces of pearlite were observed. Measurement of hardness after heat treatment of soft annealing (ferritization) on test specimens of ductile iron NL 400 and NL 700 showed a decrease in the hardness value compared to the test specimens in the unheated condition. Also, analysis of the microstructure of ductile iron specimens NL 400 and NL 700 after heat treatment of improvement revealed relaxed martensite. Measurement of hardness after heat treatment of improvements on ductile iron specimens NL 400 and NL 700 showed an increase in hardness as a result of formation of the martensitic structure. Particularly high hardness values were observed in ductile iron specimen 2.2 NL 700, where the hardness value after the improvement was 349 HB, compared to heat-treated ductile iron specimen 2.0 NL 700, whose hardness value was 220 HB. In the continuation of the research, the analysis of the influence of certain factors in the improvement (austenitizing temperature and quenching agent) on the microstructure and properties of ductile iron is proposed. As the production of ductile iron castings increases intensively every year, it is important to know how certain chemical elements affect nodule formation and, consequently, the structure of ductile iron. The structure of ductile iron affects the required mechanical properties (toughness, elongation, tensile strength) in certain industries such as automotive. The aim of heat treatment of ductile iron is to change the desired mechanical properties depending on the operating conditions. Heat treatment parameters such as temperature and duration as well as heating and cooling rates determine the type of heat treatment of ductile iron.

5. References

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