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The influence of different heat treatment cycles on controlled surface graphitization in CK45 steel

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ABSTRACT

Controlled graphitization has become known as a practical method for improvement of wear resistance and machining properties in steels. In this paper, the effect of heat treatment on microstructure of CK45 steel has been investigated. Austenitising was carried out at 920 °C for 5 h. Besides, isothermal transformation was conducted at 750 °C in the time range of 1–20 h. In this work, full potential carbon resource was tried to be used around samples as a factor to prevent the burning of the limited carbon in steel and also to increase the percentage of sample surface carbon in order to improve wear capability and machining. The microstructure of the steel considerably changes by this heat treatment process which exhibits the effects of temperature, appropriate austenitising duration and isothermal transformation. Conducted experiments show a suitable distribution of semi-spherical graphite particles especially on the surface of the steel. Also, analyses demonstrate that the amount of formed graphite in the austenitising temperature 920 °C is more than graphite in single heat treatment temperature of 750 °C.

1. Introduction

Iron and its alloys have been among the most commonly used metals and alloys in industry. The appropriate use of alloy elements along with iron can result in physical and mechanical properties. Carbon is the most important element in steels and cast irons. Steels made of iron and carbon are called plain carbon steels which are divided based on the percentage of carbon to low, average and high carbon steels. Due to limited properties of plain carbon steels, in most cases other alloy elements are also added.

Carbon in the structure of steels and cast irons can exist in the form of free (graphite), iron carbide (Fe_3C) and other alloy carbides. The formation of the graphite in the microstructure of iron alloys is called graphitization. Graphite can be formed during solidification, through deposition of carbon from solid solution (austenite) and also during Eutectic transformation [1–4].

Graphitization in steels and cast irons is done depending on desired application. Graphitization in steels is known to be a destructive factor in some cases. This kind of graphitization is observed in uncontrolled and especially in critical oxidation condition. Graphitization improves wear resistance and some of the mechanical properties of steels in cold and hot work operating condition and can be a very good substitute for free cutting steels. Machining capability and self-lubrication along with good wear resistance of this kind of alloys are improved as the percentage of graphite in steels increases.

In cast irons, due to the existence of alloy elements and its desired properties performing graphitization process is very common. An important example of this process is the formation of malleable cast iron from white cast iron. The existence of alloy elements has a very important effect on carbon solution rate in steels and cast irons and also in speeding up or slowing down the rate and time of graphitization. Silicon as an important element considerably decreases the chemical composition of Eutectic and Eutectoid points and maximum carbon solution in austenite. Adding silicon to iron carbon system leads to an increase in transformation temperature of Eutectic and Eutectoid. Additionally, in the presence of silicon, the above transformation is conducted in a range of temperature instead of one fixed temperature. This range of temperature depends on the percentage of silicon and increases as this element increases. Silicon significantly increases the tendency to graphitization by raising the temperature range of graphite formation [2,3].

A few researches have been done in this field so far. Generally, graphitization in steels has been carried out based on its similarity to cast iron. Graphitization in cast iron is accomplished in two stages. In the first stage, formation of the graphite phase is achieved by the combination of solution treatment and deposition in a way that carbides are gradually solved in austenite phase and in the

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meantime carbon diffuses toward graphite nucleus and deposits on them. The growth of graphite particles in this stage depends on the amount of carbide existing in the structure. When these carbides are finished, the first stage of graphitization is also ended. The time required for the first stage of graphitization depends mostly on the number of graphite nucleus, solution of free carbide in austenite and the rate of carbon diffusion at fixed temperature of heat treatment cycle called malleablizing. As temperature increases, the duration of the first stage in graphitization decreases. The temperature of this stage is usually chosen between 840 and 980 °C. It should also be noted that a very high temperature has disadvantages such as an increase in cost, distortion, plastic deformation of specimen and grain growth. An increase in the percentage of graphitizer elements like carbon and silicon reduces the time needed for the first stage of graphitization. On the other hand, carbide stabilizing elements like excessive manganese, chrome and molybdenum delay graphitization in the first stage or completely stop it. The research results show that the amount of chrome more than 0.03% is not desired. The percentage of molybdenum should be chosen according to the percentage of sulfur. Excessive molybdenum acts as a carbide stabilizer [4–7].

The second stage of graphitization includes cooling and keeping within the temperature range of Eutectoid transformation which is usually between 705 and 787 °C. The rate of cooling at this stage should be slow enough so that austenite does not have an opportunity to decompose into ferrite and free carbon (graphite). If the rate of cooling and the duration of keeping in this stage are excessive, austenite will change to pearlite and if specimen does not quench in this stage, martensite structure will be achieved. High cooling rates are useful when, at first stage, enough numbers of graphite spheres are formed or silicon percentage is rather high; otherwise, lower cooling rate should be used. In this case slow cooling usually continues under 650 °C because formed pearlite can change into a bunch of ferrite and graphite during cooling under the temperature of Eutectoid. Most of the factors that are effective in the first stage of graphitization show similar effects on the second stage [1–3]. The presence of graphite on the surface of steel increases the machining capability and wear resistance. Increase wear resistance and better machining, leaded steels are usually used. The reason for this use is self-lubrication property of lead. On the other hand, lead has some destructive environmental effect and also causes brittleness at high temperature.

In this paper, lead in steels has been substituted with graphite so that in addition to improving wear resistance and machining capability properties, destructive effects of the presence of lead is also eliminated [7].

2. Experimental

2.1. Preparing samples and determining the chemical composition

At first some pieces were cut from a bar with the thickness of 15 mm and the diameter of 30 mm. Then, each of them was divided into four equal samples. This is the first step of sample preparation process, and it is very important from the viewpoint of microscopic examination. Cutting the sample should be done from a place where it represents a real and appropriate surface and is a symbol of all specimens and its general microstructure. In addition, some parameter, such as pressure, temperature, cutting speed, produced heat (that may result recrystallization, local recovery and local melting), accuracy and so on must be noticed since they can affect microstructural properties of the sample. Therefore in order to prevent a rise in temperature and microstructural changes in the cutting stage, enough flow of water is used for cooling. After cutting, all samples were coded by punching Latin letters like A, B, C and E on the back side for future identification. Before heat treatment, the chemical composition of samples was determined by using Quantometry method given in Table 1.

2.2. Grinding and polishing

In order to prepare metallographical samples, rough to fine sandpapers can be used respectively. Polishing makes the surface smooth without any scratches and gets it ready for microscopic studies. In this study, in order to polish the surface, Italian polish apparatus (model: MP800V) was used. In the polish stage of samples, sandpapers with different mesh: 400, 800, 1000, 1500, 2000 and 2500 accompanied by aluminum oxide abrasive particles were used.

Table 1
Chemical composition of experimental steel (wt. %).

Element	C	Mn	Si	Cr	Mo	P	S	Fe
Wt. %	0.43	0.35	0.15	0.19	0.02	0.02	0.02	Balance

2.3. Heat treatment of CK45 steel specimens

For heat treatment and graphitization process, selected samples after passing preparation process were put into the prefabricated steel boxes with 5 cm in diameter and 7 cm height. Then graphite powder was poured around them to prevent limited burning of carbon on the surface and acting as a full potential source of carbon. In order to inhibit burning of graphite in furnace, the head of the boxes were isolated by fireproof materials and also clay so that oxygen could not enter. These boxes were put in the muffle electric furnace and the temperature was increased up to 920 °C and maintained for 5 h. Then the first box was taken out from the furnace and the sample was quenched in the water (sample A). Other boxes were transferred rapidly to another furnace with the temperature of 750 °C. After 1 h of holding the samples at 750 °C, another box was taken out, and its sample was quenched in water (sample B). Other samples after 3, 15 and 20 h holding at 750 °C were taken out and quenched in the same way (samples C, D, E). Fig. 1 schematically explains these processes.

3. Results and discussion

The overall experiments were conducted in two general ways before and after the heat treatment. This section addresses how these were carried out.

3.1. Before heat treatment

3.1.1. Microstructural analysis

The main purpose in this paper is the analysis of graphite formation procedure and its way of distribution of the surface of CK45 steel. To observe the graphite on the surface of specimen, after completing grinding and polishing stages, the optical microscope is used and there is no need to etching.

Because of the soft structure of graphite, the steps of polishing and rattling should be carried out accurately so that graphite particles are not destroyed, stretched or deformed. The most common method of grinding and rattling is mechanical method. This method is done by making use of fixed emery, mobile emery and

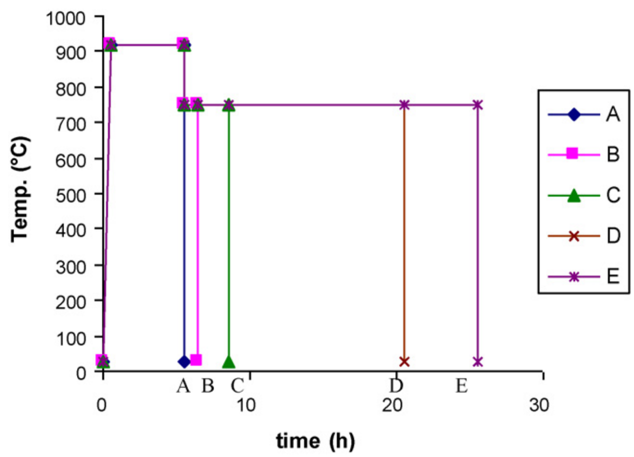

Fig. 1. Schematic diagram of CK45 steel samples, heat treatment process. The samples were maintained at 920 °C for 5 h. Then, they were transferred to another furnace with temperature of 750 °C and were taken out of the furnace at different times and quenched in water.

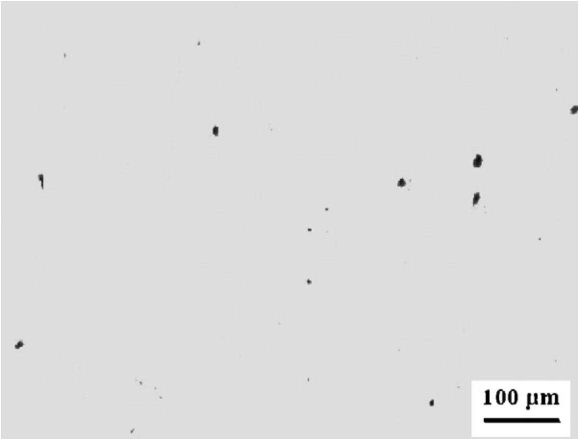

Fig. 2. Microstructure of CK45 steel sample before heat treatment (without etching). Black spots show surface oxides and inclusions which are eliminated by more polishing and increasing instance from the surface.

rotating plate. The goal of sanding, which generally decreases the roughness of emery, is to achieve an even surface without cleavages and deformation. The presence of different phases (ferrite and phases in metal matrix) with a large hardness difference in the structure of graphitized steel increases the sensitivity and is considered as difficult and lengthy process. And unfortunately, in spite of all efforts, it is not possible to completely get ideal images. The observation of CK45 steal sample before any kind of heat treatment or in a word in initial conditions under the optical microscope with different magnifications shows no graphite on the surface. Black spots observed in the figure belong to the surface oxides, impurities and inclusions (Figs. 2 and 3) which disappeared with further polishing (Fig. 3 shows fewer numbers of these spots after further polishing). Later, for microstructural diagnosis, matrix was etched by 2% Nital solution. The observed microstructure after etching is ferrite—pearlite which is the general structure of this kind of Hypoeutectoid steel (Fig. 4). Fig. 4a and b is produced in different magnifications.

3.1.2. Hardness testing

Hardness is one of the parameters representing mechanical properties. By using a hardness tester in Rockwell B scale, the measured hardness of CK45 sample before heat treatment was around 81 Rockwell B.

Fig. 3. Microstructure of CK45 steel sample before heat treatment and after more polishing (without etching). The spots which exist on the surface show a decrease in comparison with Fig. 2.

(a)
(b)

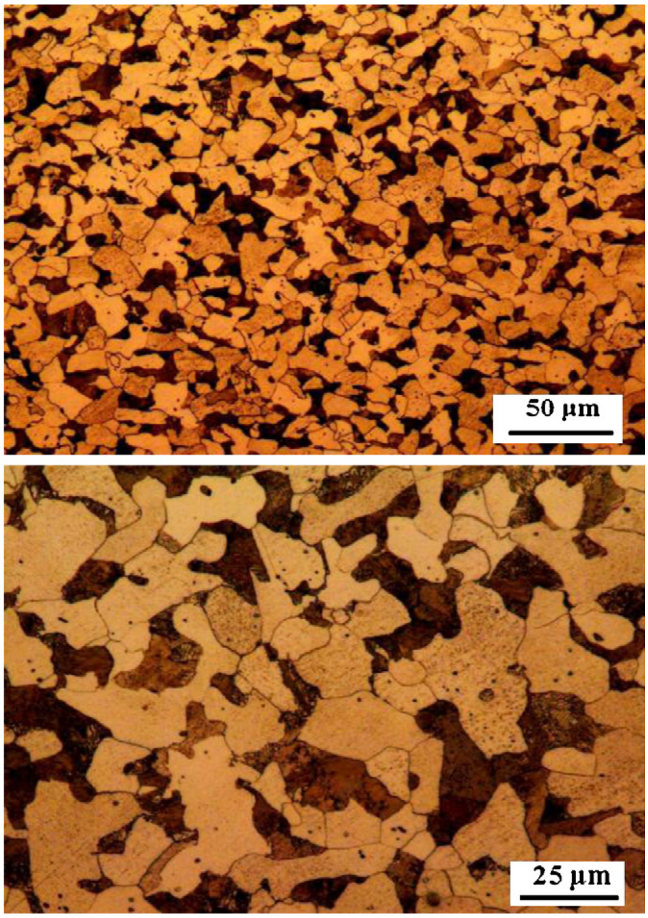

Fig. 4. Microstructure of CK45 steel sample before heat treatment in two levels of magnification. Nital 2% etchant.

3.2. After heat treatment

Lots of industrial specimens need hard surface and in the meantime good toughness or resistance to impact. One of the common methods in surface hardening is carburizing and surface hardening with chemical carburizing heat treatment which lead to chemical composition change of steel surface. In another method, without a change in chemical composition of surface, interstitial elements are concentrated in surface layer just with heat treatment and result in surface hardening. When a low carbon steel specimen is placed in carburizer materials such as char and is heated at high temperature, atomic carbon is released from the material having carbon and is diffused into the specimen surface. The resulting carbon monoxide is decomposed and releases atomic carbon which accelerates the carburizing process. As the temperature of the process increases, in fixed pressure the percentage of resulting carbon monoxide increases; therefore, the carburizing rate is augmented.

Although this process needs time, but the surface of the sample can absorb carbon in few hours. In this way a specimen was made whose center is a low carbon steel and the surface is formed by high carbon steel. In spite of putting the specimen into the matrix of carburizer materials, in this method the main factor for producing carbon is carbon monoxide (CO) which conveys atomic carbon on the surface of specimen. In the unbalanced condition, the hard cementite phase is formed which is the hardness factor and has special applications. Plain carbon steels which are used for surface hardening by the carburizing method have always 0.2% carbon. This carbon content causes toughness and good flexibility in the center of specimen. In this study, it is desired to form soft graphite in the

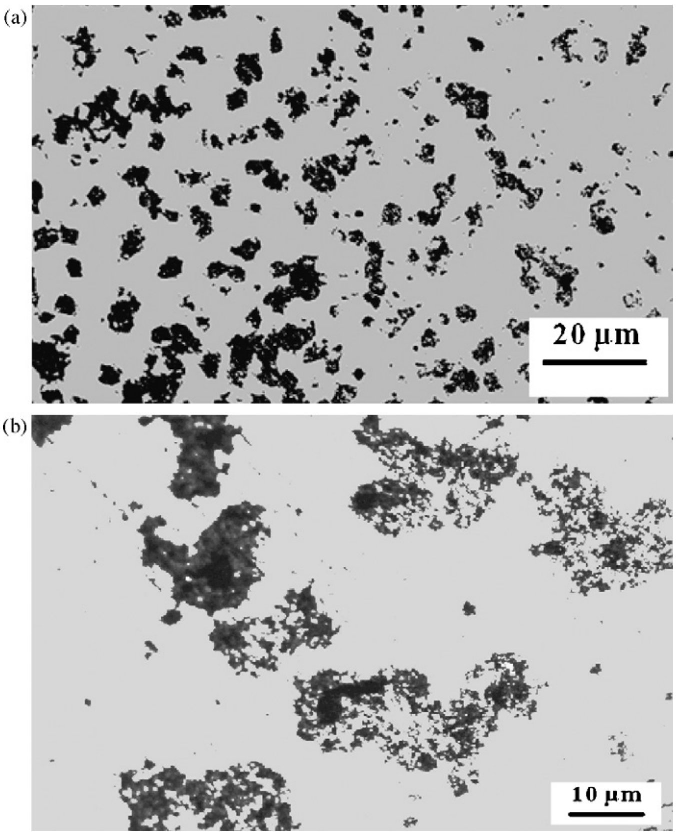

Fig. 5. Microstructure of CK45 steel sample in two different levels of magnification after austenitising heat treatment at 920 °C for 5 h and rapid quenching in water.

surface layers of steels without carbides that result excellent wear resistance and good condition for machining.

After the end of carburizing stage, to achieve the desired microstructure or mechanical properties, the specimen can be quenched in water or oil and even be cooled down to the room temperature or be cooled in open air.

3.2.1. Microstructural examinations

After heat treatment and quenching, the microstructure characterization and the distribution manner of graphite have been studied. The samples after cleaning were examined by an optical microscope, and some important changes were observed (Fig. 5). A high percentage of semi-spherical free graphite was formed on the surface of the sample. In sample A (5 h at 920 °C and quenching in water), a high percentage of graphite in the form of fine and outspread exists on the surface of the sample that shows the appropriate cycle for graphitization.

While heating and after passing the thermal range of Eutectoid transformation, the pearlite completely converts to Austenite. When the temperature increases to maximum in heat treatment cycle (920 °C), the structure includes austenite saturated with carbon from unsolved carbides and graphite nucleus. Graphite nucleuses are formed during the heating and the beginning of holding at fixed temperature. They are formed inside the primary pearlite, on the interface of cementite–austenite and upon non-metallic impurities. One of the effective parameters on nucleuses graphite is the cooling rate. When this rate increases, the number of nucleuses will decrease.

At this stage, graphitization is done by solution treatment and deposition. In this way, carbides are gradually solved in the austenite phase and carbon diffuses toward graphite nucleuses and deposits on them simultaneously. The growth of graphite particles

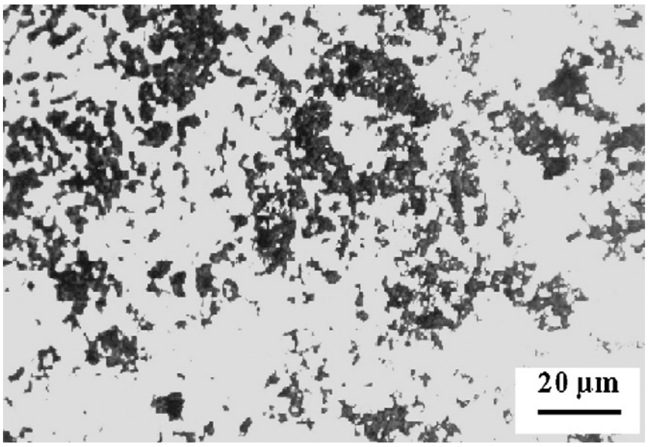

Fig. 6. Microstructure of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then putting it at 750 °C for 1 h and quenching in water. Without etching.

is attributed to the amount of existing carbides on the structure, and when the carbides finish, the first stage of graphitization will end. The required time for the first stage of graphitization is generally associated with the number of graphite nucleus, solution of free carbides and carbon diffusion rate at the fixed temperature of heat treatment cycle. As the temperature increases, the time of the first stage of graphitization is decreased. The temperature of this stage is usually chosen between 840 and 980 °C. It should be noticed that too much temperature has disadvantages like more expenses, distortion, plastic deformation and grain growth. An increase in the percentage of graphitizer elements such as carbon and silicon decreases the time of the first stage of graphitization. In addition, carbide stabilizer elements like manganese, excessive chrome and molybdenum delay the first stage of graphitization or even stop it. The results of researches exhibit that more than 3% chrome is not suitable. The percentage of manganese should be chosen in relation to the percentage of sulfur. Excessive manganese acts as a stabilizer of carbide [1,3,8].

There are not any important differences between graphite particles on sample B (at 950 °C for 5 h then 1 h at 750 °C and quenching in water) and sample A. The amount of the former graphite is just a little increased (Fig. 6). Graphitization is controlled by diffusion parameters, so time has an important role in this process and completing graphitization requires more reasonable time [9,10]. Anyway in some researches in the last decade, the addition of graphitizer elements to these alloys has been observed that accel-

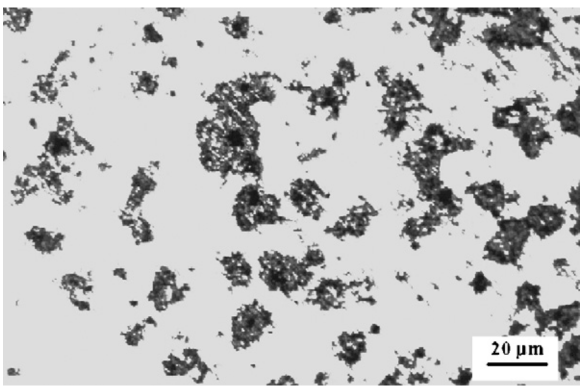

Fig. 7. Microstructure of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then putting it at 750 °C for 3 h and rapid quenching in the water. Without etching.

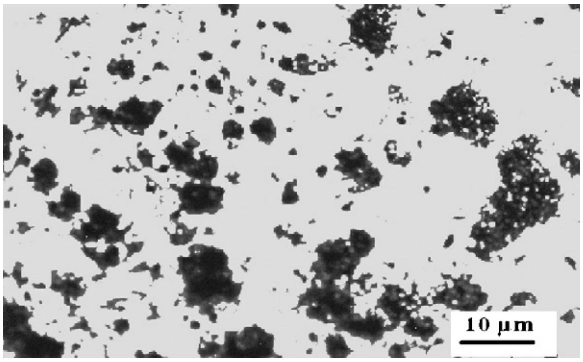

Fig. 8. Microstructure of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then putting it at 750 °C for 15 h and rapid quenching in water. Without etching.

erates this process and make the decomposition of cementite more possible [11,12].

In the sample C (5 h at 920 °C then a longer time at 750 °C and quenching in water), graphite particles are observed in the shape of semi-spherical on the surface and bigger than previous sample that due to holding at 750 °C and gathering of solute carbon on the surface upon the graphite nucleus were formed at 920 °C (Fig. 7). In the samples E and D (5 h at 920 °C then rather longer at 750 °C and quenching in water), a sensible change is not observed in numbers of graphite particles, but graphite spheres become bigger than the former case. Also, in these samples less fine graphite particles are observed (Figs. 8–10).

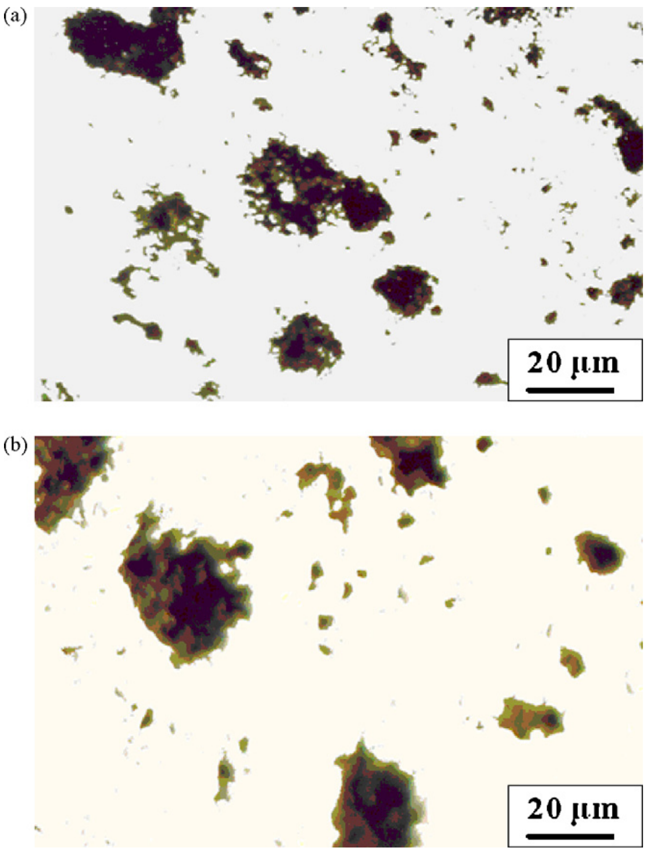

Fig. 9. Microstructure of CK45 steel sample from two different zones after austenitising heat treatment at 920 °C for 5 h then putting it at 750 °C for 20 h and rapid quenching in the water. Without etching.

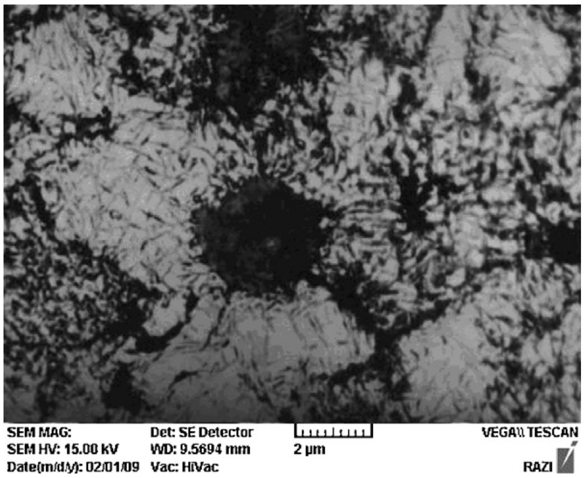

Fig. 10. SEM micrograph of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then rapid quenching in water. Nital 2% etchant.

In the malleablizing process of cast irons that is very similar to the method in this study, the second stage of graphitization includes slow cooling and holding at the thermal range of Eutectic transformation (usually from 705 to 787 °C). The cooling rate should be very slow so that austenite has enough time to decompose into ferrite and free graphite. If the cooling rate is high at this stage, austenite will transform into pearlite. If the specimen is quenched at this stage, martensite structure will be obtained. High rates of cooling are suitable for conditions in which enough graphite spheres are formed at the first stage or the percentage of silicon is high. Otherwise, slower cooling rates are used. In latter case, slow cooling rate is usually continued under 650 °C because the formed pearlite can transform into groups of ferrite and graphite under the Eutectoid temperature. A lot of parameters that affect the first stage of graphitization have similar effects on the second stage.

At the end, a number of SEM images taken at different heat treatment conditions have been presented with regard to their microscopic matrix (Figs. 10–12). These images show noticeable changes both in graphite sizes and microstructural matrix that with increasing the time of heat treatment, graphite sizes grow even partially. In the matrix, the percentage of ferrite increases gradually, so it has some points in common with the researches (Figs. 9 and 10).

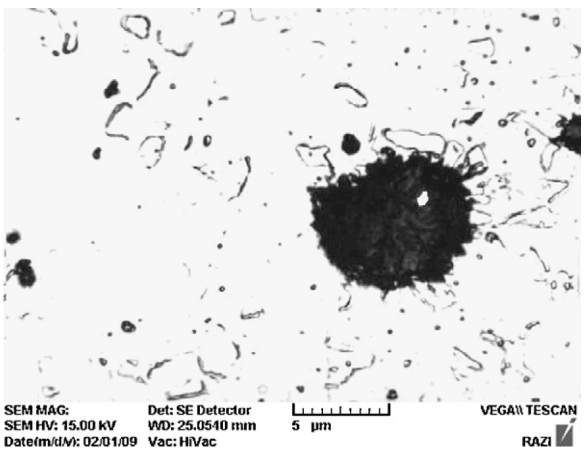

Fig. 11. SEM micrograph of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then holding it at 750 °C for 1 h and quenched in water. Nital 2% etchant.

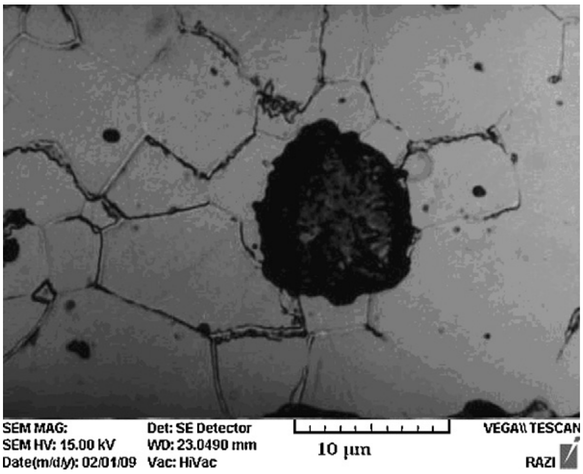

Fig. 12. SEM micrograph of CK45 steel sample after austenitising heat treatment at 920 °C for 5 h then putting it at 750 °C for 15 h and rapid quenching in water. Nital 2% etchant.

3.2.2. Hardness test

After heat treatment, the hardness test has been done on the samples to determine the amount of hardness variation as a result of carbon diffusion on the surface of the samples. The hardness number for sample A, 87 Rockwell B and for the other samples a mean amount of 84 Rockwell B were obtained. The increasing amount of hardness value compared with the samples without heat treatment is because of carbon diffusion into the iron network that generally exhibits a logical correlation between hardness and total carbon percentage. In the second step of graphitization, with an increase in time and formation of higher graphites volume, the hardness value decreases a bit. Fixed hardness at the second stage of graphitization regarding different time is due to providing the required carbon for graphitization from the solid carbon around it and to defusing the atomic carbon into the network.

4. Conclusions

(a) The obtained images from the surface of CK45 steel show that there is no graphite in the microstructure before heat treatment. The existence of black stains and spots exhibit oxides and inclusions which are observed limitedly.
(b) The goal of this study is to optimize the condition of graphitization. The presence of enough percentage of graphite after solution treatment at 920 °C for 5 h shows the correct time and temperature for graphitization process.
(c) Continuing heat treatment at 750 °C did not seriously change the size of graphite particles. Just some amount of saturated carbon deposited upon the primary graphite nucleuses and made them bigger. Also a new kind of graphite with a smaller size formed in matrix.
(d) All cases are based on making use of austenite supersaturated with carbon at high temperatures and deposition of graphite at lower temperatures. Our results show the effect of correct heat treatment in order to achieve a high percentage of graphite and uniform distribution of them specially on the surface that can significantly affect machining properties and wear resistance.

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