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Isothermal Austenitization of Spheroidal Eutectoid Steel

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Abstract

Isothermal austenitization mechanism of a spheroidal, alloyed and near eutectoid steel will be discussed in this paper. Difference in activation enthalpies determined experimentally at different temperatures using the Arrhenius formula indicates that the austenitization at higher temperatures is a diffusion-controlled process, while at lower temperatures, enthalpy necessary for austenite nuclei formation brakes the process. It is not necessary at higher temperatures because austenite crystallites developed from ferrite act as nuclei, they are much larger than the necessary size of nuclei.

Introduction

The term combined reaction is used for solid-state reactions which consist of more than one elementary reaction such as phase transformation, decomposition or recrystallization. Eutectoid transformation combines the $\gamma \rightarrow \alpha$ transformation with decomposition of carbon to form carbide [1 to 3]. The reverse reaction is austenitization of perlite. It consists of several partial processes like decomposition of cementite, diffusion of carbon across the phase boundary for the formation of γ-nuclei. The particular processes follow a different temperature dependence. It is stated in the literature that austenitization is a carbon diffusion-controlled process, i.e. it determines the rate of austenitization [2 to 4]. The role of nucleus formation is open for discussion and investigated in this paper.

The low temperature limit of the austenite stability is indicated by the GOS- SE curves in the binary system of Fe-C (Fig. 1). The alloy with eutectoid composition can transform to austenite at the temperatures above the point S (eutectoid temperature T_E). However, in case of overheating, it may transform differently into austenite at $T >> T_E$. At temperatures of about 900°C the ferrite phase of eutectoid austenitized extremely rapidly, primarily without dissolution of carbides. The mechanism is a local massive transformation which requires minimum diffusion.

This austenite starts with a very low carbon content. It dissolves carbon from cementite through secondary diffusion. Finally, the alloy reaches a homogeneous austenitic condition. At temperatures only slightly above the eutectoid temperature T_E the process of austenitization is more complicated. At the ferrite-cementite boundaries, austenite nuclei must form of equilibrium carbon content. Their growth also leads to a homogeneous austenitic condition. In this process, diffusion-limited nucleus formation plays an essential role.

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There exists an analogy between the formation of the homogeneous austenite from an eutectoid and eutectic melting. At minor overheating, a melt of eutectic composition must form by diffusion from the two crystalline phases. They can melt individually at high overheating.

Discussion

Isothermal austenitization of a slightly hypereutectoid unalloyed steel (C=0,96%, Mn=0,70%, Si=0,17%, S=0,015%) has been investigated. In their initial condition the specimens had a spheroidized microstructure, produced by tempering at 700°C for 8 hours. In order to avoid surface oxidation and decarbonization, specimens were embedded in aluminium-oxide powder during the spheroidizing heat treatment. This type of microstructure is not very sensitive to the composition of the steel. Fig. 2 shows the spheroidal eutectoid microstructure of specimens before austenitization. Heat treatment for isothermal austenitization was conducted at 730, 740, 750, 760, 840 and 860°C, exposure time was 2 to 420 s. At the beginning of the experiment series, thermoelements of the annealing furnace were calibrated by setting melting points of lead, zinc, aluminium, sodium chloride and copper as secondary and primary temperature taking reference points. Thickness of specimens was about 1,5 mm. Austenitization was carried out by dipping them into aluminium melt after preheating to $T=700°C < T_E$. Subsequently they were water-cooled. This method differs from the "classical" one [4] as aluminium is used as heating medium instead of lead, and preheating is applied with more favourable thermophysical parameters. Instead of metal melt, also salt bath can be used as heating medium of the isothermal austenitization [5]. Surface of specimens taken off the alumina was not cleaned chemically prior to the austenitization, and preheating occurred in the furnace, thus assuring that at the boundary surface of the aluminium melt and the specimen no intermetallic compounds form and avoiding adherence of considerable aluminium, which would somewhat brake the rapid cooling of the specimens. Melt mass was about 6 kg, so its temperature remained practically unchanged after short lifting of the cover for placing the specimens of masses of 1.3 to 1.5 g.

At higher annealing temperatures ($T > T_E$), the austenitic portion of the microstructure transforms into martensite. Then the microstructure consists of martensite and a mixture of ferrite-cementite which is not yet austenitized. Their volume fractions depend on temperature and duration of austenitization. These microstructures were investigated by optical microscopy. The change of amount of martensite in specimens austenitized at different temperatures was followed by an image analyser „Quantimet 570C". The measurements were carried out at least in ten areas for every specimen. For microscopic investigation, a more concentrated Nital (6%) than usual was used. Martensite is etched stronger than the untransformed components. Fig.3 demonstrates some typical microstructures, Fig.3/a shows a microstructure containing 21% martensite after a heat treatment of 740°C and 180 s and Fig.3/b a microstructure with 46.2% martensite after a heat treatment of 760°C and 12.5 s in a magnification of 400x. (Their austenitization parameters (T, t) and the Vickers hardness (HV_{10}) are also contained in Fig.4).

To follow the kinetics of the reaction, hardness of specimens was determined by Vickers hardness measurements (using a load of 98.1 N ≡10 kp). The change of specimen hardness as a function of isothermal austenitization parameters resulted in regular sigmoid

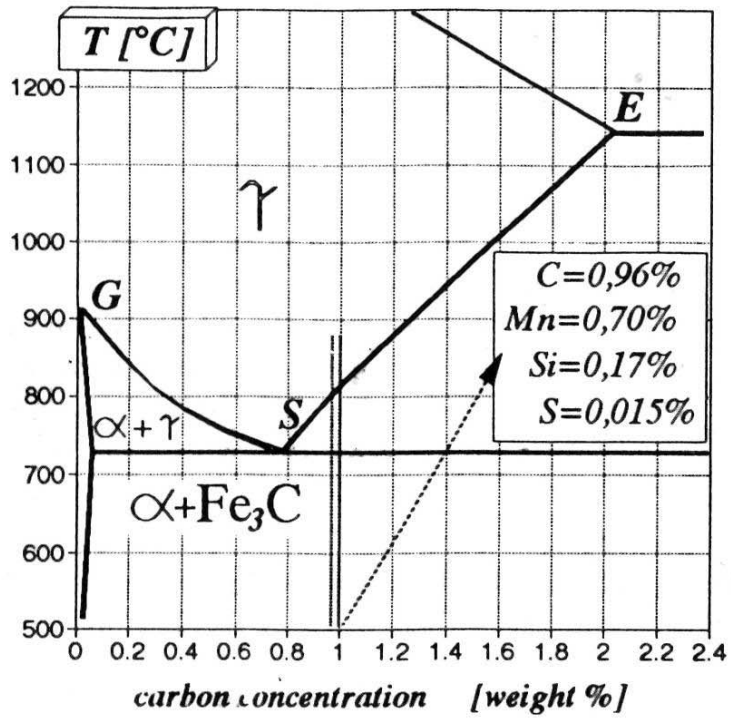

Fig. 1. Part of the Fe-C equilibrium diagram with the eutectoid transformation

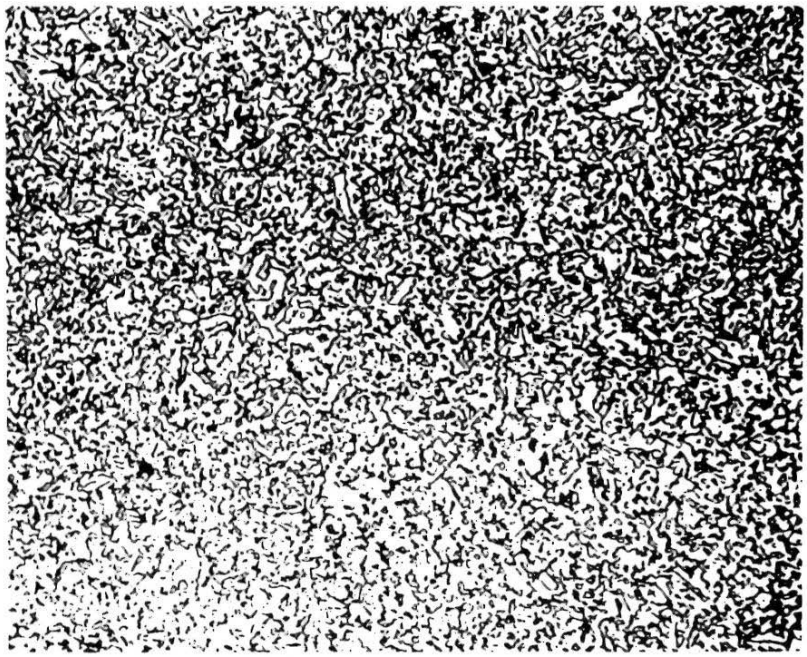

Fig. 2. Spheroidized microstructure, initial condition, obtained at 700°C : $T<T_E$

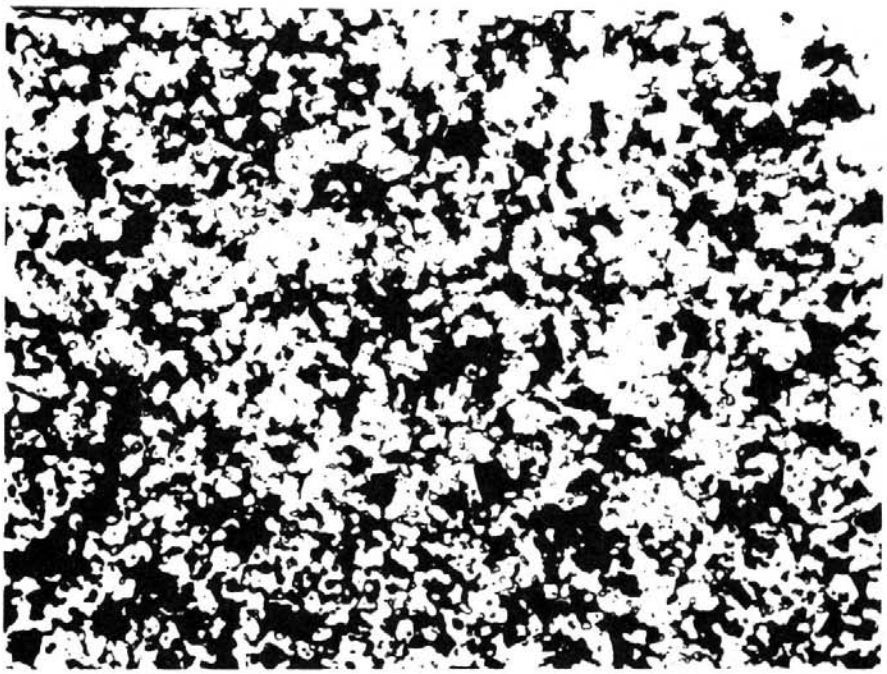

Fig. 3/a. Microstructure after partial austenitization and water cooling, untransformed ferrite and cementite (white) and transformed austenite (black, martensite; 21%).
The parameters of austenitization: T=740°C, t=180s

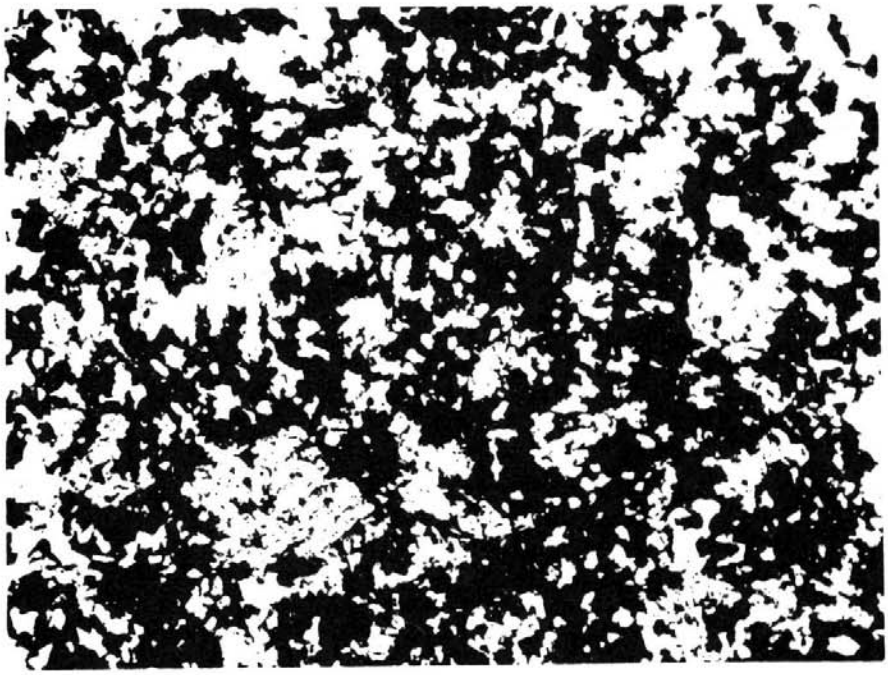

Fig. 3/b. Microstructure after partial austenitization and water cooling, untransformed ferrite and cementite (white) and transformed austenite (black, martensite; 46.2%).
The parameters of austenitization: T=760°C, t=12,5s

curves (Fig.4). Hardness measurements are sensitive to the process of austenitization because austenite had transformed into martensite. The formation of perlite must be prohibited by rapid cooling. Then the microstructure consists of a non-transformed ferrite-cementite mixture (i.e. spheroidite) and of martensite originating from transformed austenite. However, for a quantitative evaluation it has to be considered that its carbon content is variable. This means that hardness does not depend unambiguously on the microstructure. The results of a large number of tests have been summarised in Fig. 4. As symbols of the individual sigmoid curves indicate, the austenitizing heat treatments were several times repeated in order to enhance accuracy of hardness measurement, and in order to lower the differences due to inhomogeneity of the austenite developed, which is usually plotted in different austenitization diagrams [4]. Specimens were water cooled after austenitization, embedded as metallographic section and ground half thick, polished, and microscopic tests and hardness measurement were carried out on specimens prepared this way. Hardness measurement yields authentic results at the load applied because even the largest indentation deepness (diagonal 290 to 300μm) did not reach one tenth of the thickness.

From these sigmoid curves, phenomenological activation enthalpies of austenitization were determined. The activation enthalpy is characteristic of the process. If the activation enthalpy of two processes is the same, the processes may be similar, however, if the activation enthalpies differ, the processes may not be similar (their character is different). This requires coherent T, t-functions for data at a particular stage of the process. From the slope of the log t-, reciprocal T-functions Q/R values characteristic of the process are obtained (where Q is the activation enthalpy of austenitization and R is the gas constant). For determination of the activation enthalpy, the time and temperature data at the inflection points are plotted in Fig. 5. This is the condition for the maximum rate of austenitization.

For the temperature range of 740°C to 760°C (i. e. just above T_E), a value of 1126 kJ/mol was computed. This is much higher than the 140 kJ/mol indicated in literature for carbon diffusion in austenite [2, 3, 6]. The difference in activation enthalpy indicates that the austenitization mechanism is not the same at higher temperatures and at lower ones. The difference shows unambiguously that this process is not controlled simply by diffusion of carbon in austenite.

The difference clearly indicates that an additional process plays an important role in austenitization at lower temperatures. Obviously, this process must be the formation of austenite nuclei at the ferrite-carbide phase boundary.

In literature, similar investigation of austenitization of a cast iron with 2.49 wt-% silicium, 3.57 wt-% carbon, some copper, magnesium and phosphorus is described. A similar difference can be observed in the determined value of activation enthalpy [7, 8].

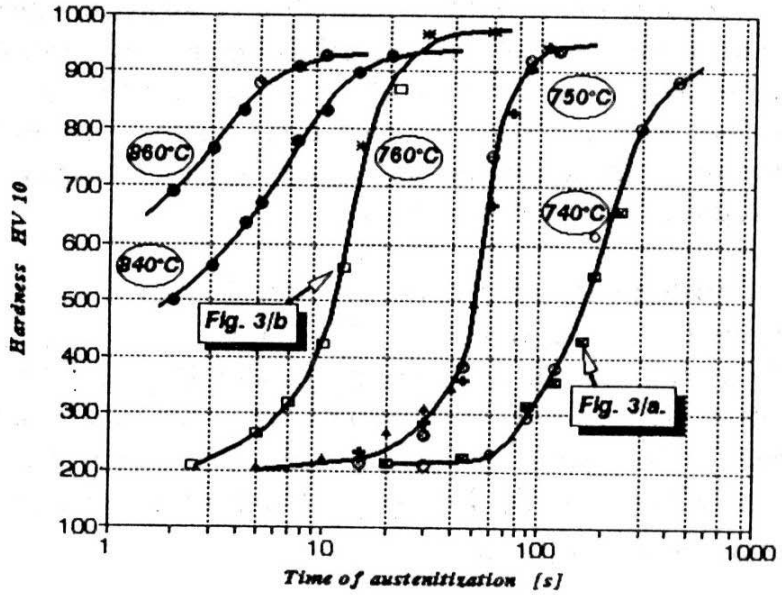

Fig. 4. Change of hardness as function of duration of isothermal austenitization. Hardening is due to martensitic transformation of reformed austenite.

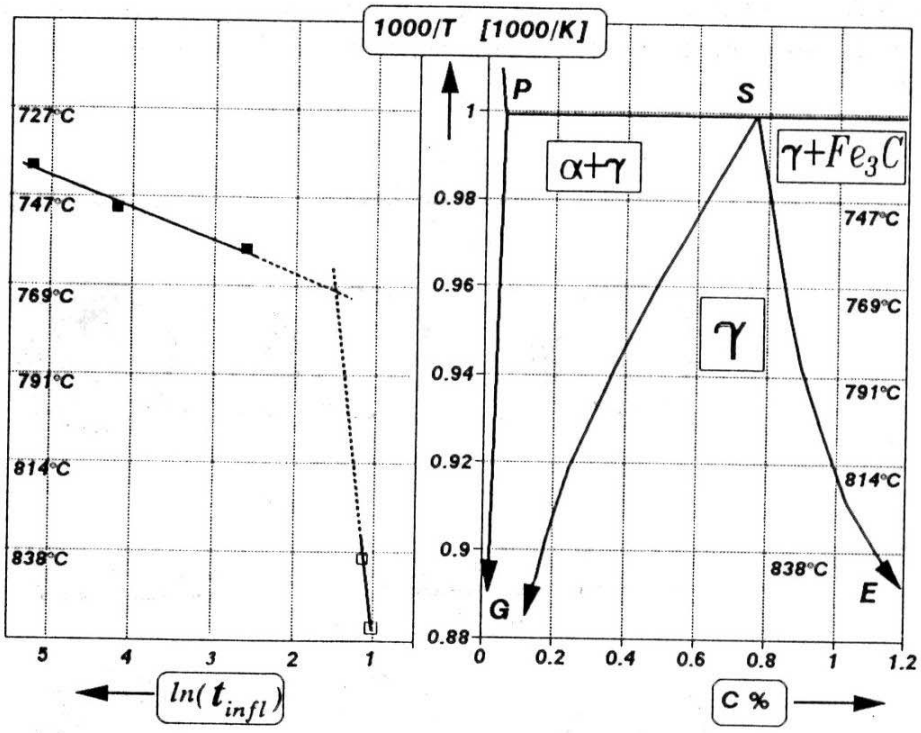

Fig. 5. Time and temperature data from the inflection points compared with the Fe-C diagram drawn with the same I/T scale.

Summary and Conclusion

According to our results, the activation enthalpy of austenitization at higher temperatures T > 770°C coincides with that of carbon diffusion in austenite. Obviously, nuclei of austenite crystals form from ferrite crystals by massive transformation. They are bigger than the required nucleus size. At lower temperatures austenitization is a more complex process. Nucleus formation has a stronger influence, actually retards the process and increases the phenomenological activation enthalpy.

Thus, kinetics of austenitization can be described by two mechanisms, one for low and one for higher overheating above T_E. The limit ($T \approx 770°C$) is determined by the necessity for carbon diffusion into austenite nuclei (Fig.5). This is demonstrated in the schematic model which shows both mechanisms of austenitization (Fig. 6). The high temperature reaction is sequential (Eq.[1]), α transforms into the γ-phase with the carbon content of the α-phase (Fig. 1), then Fe_3C dissolves and raises the carbon content from c_α to equilibrium concentration c_γ.

$$\alpha\text{-Fe}\ (c_\alpha) + Fe_3C \rightarrow \gamma\text{-Fe}\ (c_\alpha) + Fe_3C \rightarrow \gamma\text{-Fe}\ (c_\gamma) \quad [1]\ \text{sequential}$$

$$\alpha\text{-Fe}\ (c_\alpha) + Fe_3C \rightarrow \gamma\text{-Fe}\ (c_\gamma) \quad [2]\ \text{simultaneous}$$

For the low temperature reaction both proceses (phase transformation and homogenisation) must take place simultaneously (Eq.[2]). This implied a carbon content c_γ in the γ-nuclei, a large critical nucleus size as the eutectoid temperature T_E is approached and, consequently, a higher activation barrier.

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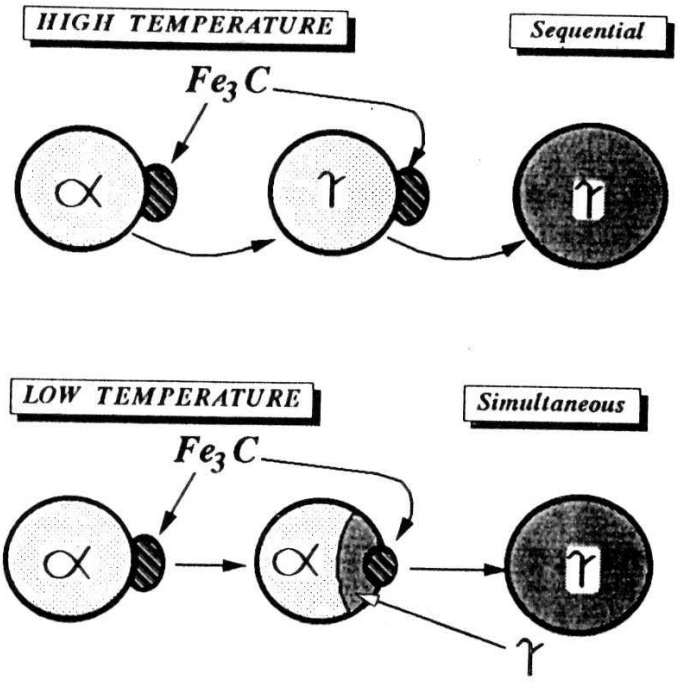

Fig. 6. Model for the two mechanisms of austenitization.

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