

Chapter 3

The Components and Phases of Systems 'Boron-Iron' and 'Boron-Carbon-Iron'

Abstract Basic chemical and physical properties of the components and phases in B-Fe and B-C-Fe systems are analyzed and the conditions of their existence and formation during the process of boriding of iron and its smelts are studied. Different types of iron borides (Fe_3B , Fe_2B , FeB , FeB_2), their crystall lattices, concentration and temperature formation conditions are thoroughly investigated. The chapter also provides the values of hardness and chemical properties which form the exploitation characteristics of boronized layers.

The plasticity of boronized layers depends on the type, content, quantity, size and mutual position of phases in the layers. One more additional factor that influences the properties is a substrate layer's material. This makes the description of existing phases in B-Fe and B-C-Fe systems especially important for the properties' evaluation.

The specifics of initial studies on iron and boron alloys was that scientists were not certain if the investigated iron-boron alloys actually contained boron. The reason was that the analysis methods of alloys' chemical contents were far from precise and were not appropriate for alloys that contained boron in small quantities. This is applied to the earliest studies of F. Osmond who was investigating allotropic metamorphosis in iron alloys generally and iron-boron alloys in particular in 1890–1895 [1]; J. O. Arnold's study on the physical influence of different chemical elements on iron (including boron) [2]; H. Moissan and G. Charpy's works on steels with different boron contents [3].

One of the earliest works based on the chemical analysis which confirmed the presence of residual boron was the paper of Guillet [4], published in 1907. The paper was devoted to changes in the structures of steels containing 0.2 and 0.5% C in dependence of the quantity of injected boron in the range of 0.2–1.5%. The result was the discovery of a specific structural component; its quantity was increasing with the rising quantity of carbon and boron. It was defined as an iron boron carbide.

The analysis of equilibrium diagrams for B-Fe and B-C-Fe systems, built on the base of various results provided by different scientists, reveals that the difference between them are defined by the diagrams' type but mostly by the positions of

critical points and the presence of certain phases. This is connected to the fact that in each case the result was influenced by the chosen study methods and their precision.

The further decades have made it possible to develop methods of investigation and deepen the knowledge of physical and chemical states of system components (boron, carbon and iron) and the phase composition and structure for double, triple and multiple iron and boron alloys.

3.1 Boron (B)

Boron is a chemical element №5 in Mendeleev periodic table (group III, period 2); its atomic weight is 10,811; boron atom diameter—1.78 kX [5]. There are 6 isotopes with mass numbers 8, 10–14; typical oxidation states—+III and –III; clear crystal boron has a greyish-black colour; the melting point = 2352 K; it is dissolvable in a concentrated HNO₃ bath; it oxidates into B₂O₃ at 973 K; it is concentrated on Earth in boric acid and various borates; it was isolated by Joseph Louis Gay-Lussac and Louis Jacques Thénard (France) in 1808.

Boron is used in metallurgy as an alloy element which increases the wear resistance and high-temperature strength of steels. Boron is combined with hydrogen to form hydrides (boranes); hydrides in solid objects (e.g., metal objects) are effectively used for hardening metal details' coatings. Boron interacts with metals to form solid solutions and chemical compounds—borides [6].

The special physical property of boron is its ability to absorb neutrons. The cross section area of heat neutron absorption for a natural isotopes mixture is 750; for isotope B¹⁰ – 3990 bn.

3.2 Carbon (C)

Carbon is a chemical element №6 in Mendeleev periodic table (group IV, period 2); its atomic weight is 12,01115; carbon atom diameter—1.54 kX [7]. There are 8 isotopes with mass numbers 9-16, typical oxidation states –IV, –II, 0, +II, +IV; the melting point = 4003 K (graphite). Carbon-12 is of particular importance as it is used as the standard from which atomic masses of all nuclides are measured: its mass number is 12 by definition.

Carbon is found in different allotropic modifications: graphite, diamond, carbyne, fullerene. The chemistry of carbon compounds is singled out into a separate field of science—organic chemistry.

Carbon is known since ancient times and has a wide-spread use: it can be a part of many alloys (steel, cast iron), it is used for producing electrodes, crucibles, black powder, metal smelting from ore (charred coal) and as an adsorbent (activated coal) in chemical industry; isotope ¹⁴C is used as a radioactive indicator [6].

3.3 Iron (Fe)

Iron (Fe) is a chemical element №26 in Mendeleev periodic table (group VIII, period 4); its atomic weight is 55,847; iron atom diameter is 2.54 kX [5]. There are 12 isotopes with mass numbers 49, 52–62, typical oxidation states are +II, +III, +VI; it is a silver-white ductile metal; the melting point = 1808 K (1539 °C); easily adaptable for forging and rolling; oxidates and covers in rust in dump air; interacts with most of chemical elements; easily dissolves in dilute acids and becomes passivated in concentrated acids; one of the most widespread elements; found in the form of ore; known from ancient times (the beginning of its use dates back to VIII-VI centuries B.C.—Iron Age); used as a base for most construction materials—cast iron, steel—as a component of special alloys, as a catalyst [6]. Iron is characterized by its polymorphism. The biggest significance for thermal and chemical-thermal processing is applied to α - and γ -Fe, which exist at the temperatures corresponding to these technological processes.

3.4 Boron Solid Solution in Iron

In 1917 N. P. Chizevsky discovered that iron, heated in boron or ferrobore powder, acquires a solid boron enriched surface layer [8]. Later boron's 'cementing' effect was confirmed and it was revealed that boron diffusion into a steel is similar to carbon diffusion in many ways [9, 10]. The diffusion of boron into iron and steel was first systematically studied in work [11]. It introduced the coefficient of boron diffusion in austenite:

$$D_{\gamma} = 2 \times 10^{-3} e^{-21000/RT}.$$

It was also established that D_{γ} doesn't depend on the carbon content below 0.43% and boron content lower than 0.003%. Later the coefficient of boron diffusion into Fe_{α} was determined as [12]

$$D_{\alpha} = 10^6 e^{-62000/RT}.$$

The comparison of obtained diffusion coefficients allowed to state that boron forms a solid interstitial solution in the lattice of Fe_{γ} and a solid substitutional solution in the lattice Fe_{α} . This conclusion was previously made by other authors [13] but based only on theoretical arguments. The implication that boron forms a solid interstitial solution in austenite could be also found in paper [14], where the researchers studied the speeds of deboronizing and decarbonizing of steel. It was established that the boron content in a decarbonized steel layer decreases with the fall in carbon concentration.

The boron solubility in iron is insignificantly low and many times less than the carbon solubility: at temperature 710 °C it is 0.0002%, at 906 °C—0.0082%, i.e. it largely depends on the temperature.

Till the 1960–1970s it was believed that boron, while dissolving in iron, can form solid interstitial solutions in Fe_γ and substitutional solutions in Fe_α . However, at that time, with the introduction of the internal friction method, it was shown that boron in areas of low content and iron can form only solid interstitial solutions [15–17]. The comparison of boron and iron electron shells' states leads to the same conclusion as boron in its electron structure differs from metals in such a way that it can't possibly take the metal's place in its lattice's point without changing the nature of mid-atomic bonds (i.e. without turning a metal in a metal-like compound—boride) [18–20]. Nevertheless, Japanese scientists, while studying the internal friction of alloys in Fe-B and Fe-Ni-B systems in order to recognize the nature of the solid solution in Fe_α , discovered that boron atoms in Fe_α grains are found in interstitial and substitutional positions [21].

Despite the low solubility in iron, boron is quite effectively used for alloying construction steels. Microsolidness of a boron-saturated α -solid iron solution is ~ 233 – 236 HV [9].

Boron in its influence on iron can be compared to carbon, but it is several times more active. Relatively small boron addition (0.02%) makes iron more adaptable for quench hardening and improves the milling of its crystal structure [22]. Boron in a solid solution significantly increases the steel's hardenability. This increase demands the boron content of 0.0005–0.0012% in a solid solution. Boron additives of more than 0.007% lead to hot shortness and complications during hot rolling and forging of steel [5].

The significantly important data for explaining increased hardenability of thermally processed steels was obtained in the study of boron distribution in Fe_γ grains; especially—the discovered inhomogeneity of such distribution. It was shown [23] that boron is adsorbed in Fe_γ : boron atoms try to segregate on borders of γ -grains; the quantity of segregating atoms increases with the rise of temperature. E.g., the number of boron on the borders of γ -grains in steel AISI86B40 ($\sim 40\text{XTH}$) increases in double with the rise of saturation temperature from 1800 °F (982 °C) to 2200 °F (1204 °C). It is well known that boron in steels under hardening slows the beginning of austenite decomposition. The increased concentration of boron doesn't effect it. Only saturation of grain borders up to the critical concentration leads to its appearance.

The introduction of boron into steel aimed at increasing its hardenability allows to save a significant number of rare and expensive alloying elements. The incision of 0.002% B (20 grams of boron for 1 ton of steel) allows to save 1,2–2.4% Ni or 0.15–0.35% Mo. Due to the large size of boron's atom diameter (1.78 kX) in comparison with carbon (1.54 kX), nitrate (1.40 kX) or oxygen (1.32 kX), its solubility in iron (atom diameter ~ 2.54 kX) is lower than other mentioned elements'. However, the influence of boron atoms on the iron lattice, due to its big atom diameter, is stronger than solid interstitial solutions of carbon, nitrate or oxygen with iron have. Moreover, a low carbon content in steel makes boron more

effective in increasing hardenability. Normally boron is added to low- or medium-carbonated steels which contain less than 0.60% C.

Boron doesn't increase the hardenability of eutectic and over-eutectic steels, thus it is not added to these kinds of steels. The addition of boron is most effective in steels which contain no more than 0.30% C [5].

3.5 Iron Borides

Iron borides are chemical compounds of boron and iron. The main peculiarity of iron borides is their high density and low ductility. Iron borides, when heated in ammonia currents at 400–600 °C, decompose into iron nitrides and boron; when heated at 700–768 °C, iron Fe_α and boron nitride BN are acquired [22, 24].

Borides found in the structure reduce the hardenability of steels as they are the center of austenite transformation, e.g. into perlite [5, 25, 26]. If borides are assembled in a row according to the increasing level of boron content, four borides will be found there— Fe_3B , Fe_2B , FeB and FeB_2 —all of them are studied in connection with iron and steel boriding.

Iron Boride Fe_3B The study [27] states that borides like Fe_3B exist only in the presence of carbon. This way they are the product of the substitution of carbon by boron in the cementite lattice [28, 29] and are defined by the formula $Fe_3(B,C)$. The data in the work [28] shows that boron can substitute more than 40% of carbon atoms in $Fe_3(B,C)$ phase without transforming its crystal structure. This fact allowed the authors in the paper [17] to doubt the legitimacy of attributing this compound to borides.

However other works [30–32] inform that Fe-B system contains a compound Fe_3B . It can be obtained by hardening of alloy $Fe_{76}B_{24}$ from liquid state at cooling speeds which suppress the equilibrium $Fe_\alpha + Fe_2B$ and allows to get a crystal structure. The obtained Fe_3B phase exists in the thermodynamic equilibrium at high temperatures. The temperature range of existence is established between 1150 and 1250 °C. Fe_3B Boride has two stable modifications.

Iron Boride Fe_2B Iron boride Fe_2B contains 8.83% (8.34% [33]) of boron and has a body-centered tetragonal structure type— $CuAl_2$ with 12 atoms in a cell and with periods of lattice: $a = 0.5109$ nm, $c = 0.4249$ nm, $c/a = 0.831$ [18, 27, 34–36] or $a = 0.5078$ nm, $c = 0.4249$ nm, $c/a = 0.836$ [37]. Microsolidness is 1800–2000 HV [37], or 1320–1680 HV [9], or 1500–1800 HV [38].

The phase equilibrium diagram of B-Fe doesn't clearly show [39, 40] if boride Fe_2B is a steady compound. It has the elastic modulus of $(285–295) \times 10^3$ MPa, the thermal-expansion coefficient— $7.65 \text{ mln}^{-1}/^\circ\text{C}$ and $9.2 \text{ mln}^{-1}/^\circ\text{C}$ in the temperature range from 200 to 600 °C and from 100 to 800 °C correspondingly [37, 41, 42], or $14 \times 10^{-6} \text{ grad}^{-1}$ [38], density— 7.43 t/m^3 . The general opinion is that boriding should better ensure the generation of a one-phase monolayer Fe_2B with columnar saw-shape morphology and not the generation of a two-phase layer with FeB phase

[33, 37, 42–48]. The formation of columnar structure is witnessed due to the anisotropy of the diffusion coefficient in borides.

A monophase layer Fe_2B may be obtained from a two-phase $\text{FeB-Fe}_2\text{B}$ layer by special vacuum processing or processing in a salt bath for several hours at the temperature $\sim 800^\circ\text{C}$ which can be followed by hardening in oil in order to improve the plasticity properties of a substrate [37, 49].

Iron Boride FeB Iron boride FeB (monoboride) contains 16.23% of boron and has an orthorhombic lattice with 8 atoms in a cell (4 boron atoms and 4 iron atoms) and with lattice periods: $a = 0.4061\text{ nm}$, $b = 0.5506\text{ nm}$, $c = 0.2952\text{ nm}$ or $a = 0.4053\text{ nm}$, $b = 0.5495\text{ nm}$, $c = 0.2946\text{ nm}$ [1, 6, 51]. It is characterized by polymorphism and exists in two variants: low-temperature and high-temperature (lower and higher 1135°C correspondingly). However this fact requires additional proof as it was the existence of only one variant which was experimentally proved—the modification of FeB which has a B27 structure type.

The microsolidness is 1900–2100 HV [33, 37, 46] and 1850–1900 HV [33] or 1800–2200 HV [38]. The melting point is 1390°C [33, 37]. The elastic modulus is $590 \times 10^3\text{ MPa}$, the density— 6.75 t/m^3 [37], the thermal-expansion coefficient— $23\text{ mln}^{-1}/^\circ\text{C}$ between 200 and 600°C [37, 41, 42] or $\sim 9 \times 10^{-6}\text{ grad}^{-1}$ [37]. Boride FeB is the most chemically stable phase of B-Fe system. It is stable and doesn't decompose under hot pressing in the mix with carbide B_4C up to 1450°C and in this environment there is no interaction between FeB and B_4C .

Boride FeB is easily formed under hot pressing ($\sim 1300^\circ\text{C}$) of powder mixtures of iron and its carbide (Fe_3C) with boron or boron carbide (B_4C) [7, 16]. Iron boride FeB dissolutes in nitric acid [22].

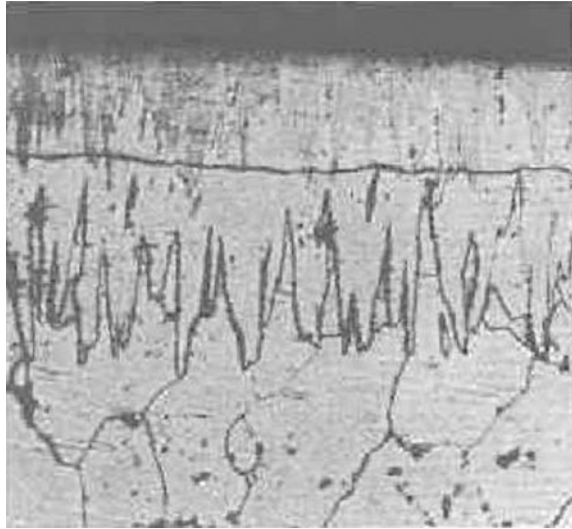
The hard exploitation, especially under alternating loading, makes the presence of a boron-saturated whole outer layer of a FeB phase in a boronized layer almost unacceptable [33, 37, 42, 46, 50]. The presence of the sharp variation of residual stresses on the border of these phases leads to formation of cracks in the layers with FeB and Fe_2B on or near the surface of transition $\text{FeB-Fe}_2\text{B}$ [13, 33, 37, 42, 51–53] (Fig. 3.1). The conclusion is that it is necessary to avoid or significantly limit the quantity of FeB phase in boronized layers. Good exploitation properties are characteristic for boronized layers where the correlation of phases $\text{FeB:Fe}_2\text{B}$ is no more than 1:3 [38].

The presence of carbon in steel doesn't affect its hardness; alloying elements Ti, Mo, W increase the hardness of boride FeB while the presence of Al or Cr decreases it [33, 54]. Under heating boride FeB is stable up to 800°C .

Iron Boride FeB_2 Iron boride FeB_2 (diboride) contains 27.9% of boron and can be attributed to hexagonal systems, structural type AlB_2 . The parameters of its elemental cell: $a = 3.045\text{ nm}$; $c = 3.035\text{ nm}$; $c/a = 0.997$. The melting point of FeB_2 is $2070 \pm 50^\circ\text{C}$ [33, 55].

The number of atoms in a rhombohedra lattice is 1 (3 cells—2 boron atoms and 1 iron atom). The atom coordinates are Fe (000), B1 ($1/3\ 2/3\ 1/2$), B2 ($2/3\ 1/3\ 1/2$). Boron atoms form graphite-shape lattices, which dimensions are

Fig. 3.1 The microstructure (x200) of a two-phase boride layer in a low-carbon steel (boriding at 900 °C for 4 h)



perpendicular to axis Z. Boron atoms' layers are alternated with iron atoms' layers which contain two times less boron atoms [17, 56].

The microsolidness is 2740–2800 HV [17] or 1650 HV₁₀₀ [54]. The elastic modulus is 25.3×10^3 kg/mm², the melting point is 2070 ± 50 °C. The presence of carbon or alloying elements Ti, Mo, W, Cr doesn't affect the hardness of FeB₂ boride [33, 54]. FeB₂ boride is stable under heating up to 1000 °C.

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