

Strain Aging in Boron Alloyed Multi-Phase C- Mn – Si – Steel Wire Rod

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Abstract

The specimens of C – Mn – Si - steel wire rod 5.5 mm in diameter with 0.005% B and without B additions from Moldova Steel Works had been studied. It was been established by crystallographic-geometrical analysis, that the boron atoms could be allocate in B – alloyed α – Fe only in the positions of a sub-interstitial solid solution. By study a static strain aging (SSA) and a dynamic strain aging (DSA) specimens of wire rod by C – Mn – Si - steel with B and without B (both with multi-phase (ferrite- martensite (bainite)-pearlite) microstructures) more expressed decreasing of strengthening properties and higher characteristics of ductility had been determined for C – Mn – Si - steel specimens with B. These results could be explained by “de - nitrogenous” and “de - carbonaceous” mechanisms, when boron atoms from α – Fe sub-interstitial solid solution by producing wire rod and by its strain aging thermal treatment with temperatures 150 - 450 °C generate boron- nitrogenous and boron - carbonaceous – nitrogenous precipitations. By realizing these mechanisms nitrogen and carbon atoms are partly excluded from the dislocation pinning’s process. This, in fact, explains to inhibit the development of strain aging (SSA and DSA) in boron- micro-alloyed C – Mn – Si - steel.

Keywords: static and dynamic strain aging, wire rod, boron micro-alloyed steel, mechanical properties.

1. Introduction

It was showed in (Frank & Kirkcaldy, 1998; Yalamanchili, Nelson, Power, & Lanham, 2001; Parusov., Sychkov, Zhigarev, & Perchatkin, 2004), that metal-roll of boron – micro-alloyed low – carbon and low-alloyed steels was characterized by a high level of ductility and deformability. Mechanical and physical properties of boron – micro-alloyed low – carbon and low-alloyed steels are determined to a large degree the peculiarities of boron atoms’ disposition in the crystalline lattices of iron’s γ - and α – phases (austenite and ferrite, respectively). A comparison the sizes of the octahedral and tetrahedral voids in the crystalline lattices of γ - and α – Fe with size of a boron atom ($r_B = 0.091\text{ nm}$) was showed (Table 1, data of (Lyakishev, Pliner & Lappo, 1986), that the boron atom, whose size in 1.26 times surpasses the size of nitrogen atom and in 1.18 times - the size of carbon atom, whether could be allocate in the positions of an interstitial solid solution (in the octahedron and tetrahedron voids) in both iron’s modifications.

However, accordingly data, for instance, in Table 2 (McBride, Spretnak, & Speiser, 1954), boron atoms have a decided solubility in γ - and α - Fe. Data of the X-ray structure analysis were showed (Shevelev, 1966), that a crystalline lattice parameter a of the B – alloyed α - Fe had a lower value, than such in α – Fe without B. As it in (Shevelev, 1966) was stated the boron atoms could be allocate only in the positions of a sub-interstitial solid solution in B – alloyed α – Fe.

A thermodynamically steady solid solution was found out (Brown, Garnish, & Honeycombe, 1974) in boron alloyed α – Fe by the method of autoradiography. A Snoek peak was not detected in internal-friction measurements, indicating that boron is not in an interstitial solid solution in α – Fe. Therefore authors (Brown, Garnish, & Honeycombe, 1974) asserted, that boron generate in α – Fe a sub-interstitial solid solution, but in γ - Fe, possibly, - an interstitial solid solution.

However, the changing of the lattice parameter a of γ - Fe (without B and with adding 0.005 wt. % by B) in temperature interval 925... 1200°C showed (Figure 1, data of (Mirkin, 1979)), that by all temperatures of this interval the lattice parameter a of the boron – alloyed γ - Fe had a lower value, than such in γ - Fe without B. As the case stands it means, that only a sub-interstitial solid solution in γ - Fe could be generated by boron.

Table 1. Size's parameters of Fe -, N -, C -, B - atoms, of octahedral and tetrahedral voids of α - and γ -Fe – lattices (Lyakishev, Pliner & Lappo, 1986)

Atomic radius, nm				Void's radius, nm			
Fe	N	C	B	γ -Fe		α -Fe	
				octahedral	tetrahedral	octahedral	tetrahedral
0.127	0.072	0.077	0.091	0.053	0.029	0.019	0.036

Table 2. A boron solubility in γ - and α -Fe (McBride, Spretnak & Speiser, 1954)

Mo- difi- cation	Temperature, °C / Boron contain, wt. %							
	1131	1049	949	915	906	837	794	710
γ -Fe	0.0182	0.0061	0.0034	0.0024	0.0020	-	-	-
α -Fe	-	-	-	-	0.0082	0.0061	0.0011	0.0002

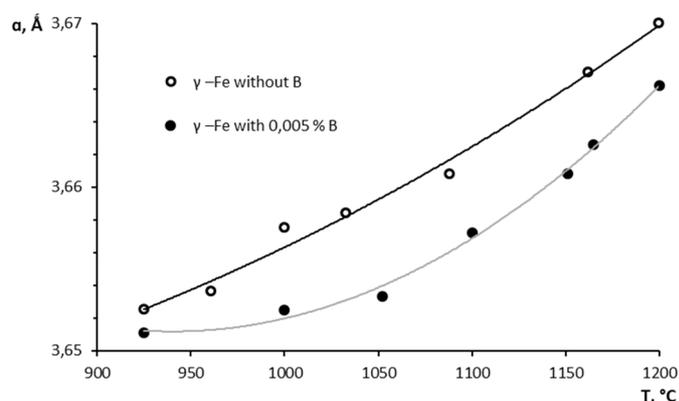


Figure 1. Changing of crystalline lattice parameter a of γ -Fe (without B and with 0.005 wt. % by B) in temperature interval 925...1200°C (Mirkin, 1979)

Strain aging is commonly referred to as a change in properties of metals after cold or hot (warm) working (Babich, Gul & Dolzhenkov, 1972). Its key effects are hardening and embrittlement of steels and alloys. They can be observed in deformed steels during its soaking at ambient temperatures or thermal activation temperatures (normally, at 100–500 °C) – static strain aging (SSA), and during deformation of steels – dynamic strain aging (DSA). The mechanism of strain aging is based on effects of dislocation pinning by impurity atoms (primarily carbon, C, and nitrogen, N).

Aging of steels, *on the one part*, leads to adverse implications, such as loss of ductility and toughness and certain instability of properties of steels over time (Babich, Gul, & Dolzhenkov, 1972). To mitigate these effects technological solutions and methods are being studied with the aim to inhibit the development of strain aging processes in steels. *On the other part*, aging is employed as a type of hardening treatment. For instance, recently in automotive manufacturing bake hardening (BH) process is used (Calcagnotto, Adfachi, Ponge, & Raabe, 2014). BH process is in active during automotive paint baking. Consequently, it is factually the implementation of SSA in cold deformed (after press forming) automotive parts. Authors (Calcagnotto, Adfachi, Ponge & Raabe, 2014) simulated BH process in dual-phase (DP – ferrite – martensite) sheets by steel of composition (in wt. %): 0.17C; 1.49Mn; 0.22Si; 0.0031S; 0.0011P; 0.033Al; 0.0033N. To simulate BH process the specimens of DP sheets with ultra fine ferrite grains (UFG) (ferrite grain size $d_{FG} = 1.2\mu\text{m}$) were aged at 170 °C for 20 min in a vacuum furnace. Aging at 170 °C (bake hardening) of the UFG DP steel led to a strong increase in YS and small decrease in TS (Table 3). Plasticity was enhanced in term of total elongation EL . Reduction in area RA was improved by 22% (from 15.3% to 37.5% - Table 3). These effects were attributed to dislocation's locking, relaxation of internal stress, reduction of supersaturated interstitial carbon and nitrogen and tempering effects in martensite (Calcagnotto, Adfachi, Ponge, & Raabe, 2014).

A development of new combinations of steels composition and conditions of their deformation and thermal treatment is particularly important for steel products that are further subjected to cold deformation, such as wire

rod of carbon-alloyed and low-alloyed steels for commercial wire drawing. Even though numerous studies have been conducted in this field, many issues connected with the influence of B - micro-additions, in particular, on mechanical properties of low-alloyed steel wire rod during strain aging has remained uninvestigated at all thus making this research very important.

2. Materials and Research Techniques

The steel's object employed in the investigation was C – Mn – Si - steel wire rod 5.5 mm in diameter without B (heat 1) and with 0.005% B additions (heat 2) from Moldova Steel Works. The Table 4 below shows chemical compositions of the C – Mn – Si - steel heats under study. Optical microscopy method (light microscope “Axiovert 200 M MAT”) was used by study structures of wire rod's specimens. The parameters of fine crystalline structure in these specimens - levels of the broadening of X-ray interferences, micro-deformation $\Delta a/a$ in α - Fe – crystalline lattice and dislocation's destiny ρ in α - Fe (ferrite) were determined by X-ray diffraction method of approximations (Gorelik, Skakov & Rastoguev, 1994). X-ray diffraction analysis of wire rod's specimens was realized by computerized diffractometer DRON – UM1.

Table 3. Volume of martensite and mechanical properties of UFG DP steel (Calcagnotto, Adfachi, Ponge & Raabe, 2014)

Type of DP-steel	Volume of martensite, %	Yield strength <i>YS</i> , MPa	Tensile strength <i>TS</i> , MPa	Total elongation <i>EL</i> , %	Reduction in area <i>RA</i> , %
UFG	29.8	525±8	1037±16	7.3±4	15.3±4.0
UFG BH	28.2	619±6	1005±5	11.4±1.5	37.5±0.9

Table 4. Chemical composition of tested heats

Heat number	Fraction of elements, wt. %										
	C	Mn	Si	S	P	Cr	Ni	Cu	Al	N	B
1	0.07	1.87	0.78	0.003	0.011	0.04	0.09	0.15	0.0045	0.008	-
2	0.07	1.79	0.75	0.002	0.012	0.05	0.10	0.19	0.0010	0.008	0.005

The experiment for study SSA in C – Mn – Si - steel wire rod 5.5 mm in diameter without B and with 0.005% B additions B (heats 1 and 2, respectively, Table 4) included a preliminary deformation with a residual elongation 6 % and an aging at temperature 200°C in due 1 hour.

In the experiment for study an effect of DSA specimens by investigated steel's wire rods were subjected to tensile tests at room temperature (to determine the original level of mechanical properties of specimens) and at temperatures from 100 °C to 500 °C on a tensile testing machine fitted with a high temperature accessory. The specimens were held at the test temperature for 15 minutes and then subjected to tensioning at the rate of 2 mm/min.

3. Results and Discussions

As shown by the microstructure analysis, hot-rolled C – Mn – Si - steel wire rod with and without B additions originally had a multi-phase microstructure consisting of a polygonal ferrite matrix (ferrite grain size $d_{FG} = 15 - 17 \mu\text{m}$, № 10 (ASTM)) with evenly distributed pearlite and martensite (bainite) islands (Figure 1).

The results of experiment for study SSA were showed, that SSA in C – Mn – Si - steel wire rod with 0.005% B developed less intensive, than in such wire rod by steel without B. The decrement of strengthening properties (tensile strength *TS* and yield strength *YS*), induced by SSA in C – Mn – Si - steel wire rod with 0.005% B was more significantly, than in such wire rod without B. A difference between the decrement of strengthening properties was more expressed for *TS*: $\Delta TS = - 52 \text{ MPa}$ because for *YS*: $\Delta YS = - 11 \text{ MPa}$. These results showed a weakened influence of the boron – micro-alloying on strengthening properties of C – Mn – Si - steel wire rod by SSA. Less development of SSA in steel with B was explained by realizing “de - nitrogenous” and “de - carbonaceous” mechanisms, when boron atoms from the α – Fe sub-interstitial solid solution by producing wire rod (the first step of binding effect) and by its strain aging thermal treatment (the second step of binding effect) bind nitrogen and carbon atoms in boron- nitrogenous and boron - carbonaceous – nitrogenous precipitations. Most part of these precipitations wouldn't be so small for the effect of dispersion hardening.

Special chemical analysis showed that in α - Fe solid solution of hot-rolled C - Mn - Si - steel wire rod with 0.005% B (heat 2, Table 4) content of free (don't bind) nitrogen atoms was 0.004%, but in hot-rolled C - Mn - Si - steel wire rod without B (heat 1, Table 4) - 0.008%. It means that by producing in C - Mn - Si - steel wire rod with 0.005% B approximately 50% of nitrogen atoms were bound by boron - nitrogenous and boron - carbonaceous - nitrogenous precipitations. This fact and above mentioned "binding effect" by aging (the second step of binding effect) inhibit developing of SSA in C - Mn - Si - steel wire rod with B.

The results of X-ray diffraction study showed that levels of the broadening of X-ray interferences, micro-deformations $\Delta a/a$ in α - Fe - crystalline lattice and dislocation's density ρ in α - Fe (ferrite) of hot-rolled C - Mn - Si - steel wire rod with B were lower than such in hot-rolled C - Mn - Si - steel wire rod without B (Table 5). These results could be explained in the first place by realizing "de - nitrogenous" and "de - carbonaceous" mechanisms in hot-rolled C - Mn - Si - steel wire rod with B (above mentioned the first step of "binding effect" - by producing).

In the experiment for study an effect of DSA in specimens of C - Mn - Si - steel wire rod without B and with B additions (heats 1 and 2, respectively, Table 4) a steady reduction in tensile strength properties is virtually observed for all test temperatures (Figure 3) rather than an increase, which is typical for carbon steels with ferrite - pearlite structures (Babich, Gul & Dolzhenkov, 1972). The only exclusion is the positive change of TS at 350 °C compared to the initial values of the wire rod without B additions (Figure 3 a).

Apparently, steady loss of TS and YS at 100-120 °C (Figure 3) is associated with decreasing a strained state's level in steels that are stipulated by the strain's relocation between the structure fractures of C - Mn - Si - steel wire rod - ferrite, pearlite and martensite (bainite) (Figure 2), with reduction of supersaturated interstitial carbon and nitrogen and tempering effects in martensite (bainite). Notably the effects similar to such in UFG DP steel sheets at 170 °C aging (bake hardening) (Calcagnotto, Adfachi, Ponge & Raabe, 2014) were implemented. Small increasing of strengthening properties by temperatures 150-450 °C (Figure 3) was caused by break-up's effect in martensite (bainite), increasing dislocation's locking and generating a lot of new sub-micro- precipitations. One part of these precipitations had very small size and could be inhibit the dislocation's flow in steels.

The trend of ductility is of interest. The variation of the total elongation EL_{100} with temperature slightly differs between C - Mn - Si - steel wire rod with B and without B, but level of EL_{100} permanently was higher for wire rod with B (Figure 4 a). Values of the reduction in area RA observed in the wire rod with B at a consistent high level of 77-80 % at 200-450 °C (Figure 4 b), which is the most dangerous range of temperatures affecting plasticity and drawability of the steel. Apparently, it is the key factor that accounts for the high deformability of this wire rod, which can be observed under the conditions of commercial production. As can be seen in the same Figure 4 b C - Mn - Si - steel wire rod without B has a lower level of RA .

In the current literary sources there are the controversial points on the subject of changing of dual- phase (DP - ferrite - martensite (bainite)) steels mechanical properties by DSA - process. So, in (Shahriari, Koohbor, Ahadi, Ekrami, Khakian-Qomi & Izadyar, 2012) was researched specimens of AISI 4340 steel bars with DP - microstructure imposed a 3% pre-strain at the temperature range of 150-450 °C and its the room temperature tensile tests were carried out, afterwards. Results indicated that the maximum values for both YS and TS would exist for the pre-strained samples at the temperature range of 250-300°C. Also, a sudden drop of the ductility was observed at the mentioned temperature range. The observed properties' behavior might be attributed to the DSA process that took place at this temperature range. Queiroz et al. (Queiroz, Cunha & Gonzalez, 2012) revealed too a significant strengthening and loss in ductility of DP - steel - specimens between 200 - 400 °C by its tensile tests at the temperature range of 150-450 °C. Queiroz et al. Authors (Queiroz, Cunha & Gonzalez, 2012) linked this properties' behavior to the DSA phenomena. On the other hand, authors (Bayramin, Şimşir & Efe, 2017) determined, that mechanical testing of DP590 and DP800 steels between room temperature - 400°C revealed two types of strain aging behavior for given temperature range. Both grades of the steels demonstrated a slight softening of TS and minimal loss in total ductility up to 100°C. At 400°C testing softening both grades of the steels was significant, notably TS decreased considerably. At 200 - 300°C - testing the increasing of TS took place, what caused a significant drop in ductility. Special authors' research proved (Bayramin, Şimşir, & Efe, 2017), that both DP590 and DP800 steels strain aging behavior for given temperature range of 150-450°C linked to the DSA - process.

Above mentioned data testified, that C - Mn - Si - steel with B - adding and MP - microstructures has been shown the different mechanical properties' behavior by DSA because of realizing boron - "de - nitrogenous" and "de - carbonaceous" both mechanisms then such AISI 4340 (with DP - microstructure), DP590 and DP800 steels (all without B - adding).

Table 5. The parameters of fine crystalline structure in the investigated specimens

Kind of C-Mn-Si-steel's specimens	Broadening of X-ray interferences, radian $\times 10^{-3}$ *		$\Delta a/a \times 10^{-3}$	$\rho \times 10^{10} \text{cm}^{-2}$
	(110)	(220)		
Without B	1.990	12.313	0.98	1.96
With 0.005% B	1.799	11.725	0.93	1.78

*((110) and (220) interferences' of steel's α - Fe solid solution (ferrite) were gained by using Fe - X-ray radiation.

4. Conclusions

The specimens of C - Mn - Si - steel wire rod 5.5 mm in diameter with 0.005% B and without B additions from Moldova Steel Works has been studied. It was been established by crystallographic-geometrical analysis, that the boron atoms could be allocate in B - alloyed α - Fe only in the positions of a sub-interstitial solid solution. The higher characteristics of ductility (EL_{100} , RA) and more expressed decreasing of strengthening properties in micro-alloyed by boron C - Mn - Si - steel wire rod versus such wire rod by C - Mn - Si - steel without boron (both with MP - microstructures) could be explained by "de - nitrogenous" and "de - carbonaceous" mechanisms, when boron atoms from the α - Fe sub-interstitial solid solution by producing wire rod and by its strain aging thermal treatment at temperatures 150 - 450 °C generate boron- nitrogenous and boron - carbonaceous - nitrogenous precipitations. By realizing these mechanisms nitrogen and carbon atoms are partly excluded from the dislocation pinning's process. This, in fact, explains to inhibit the development of strain aging (SSA and DSA) in boron - micro-alloyed C - Mn - Si - steel.

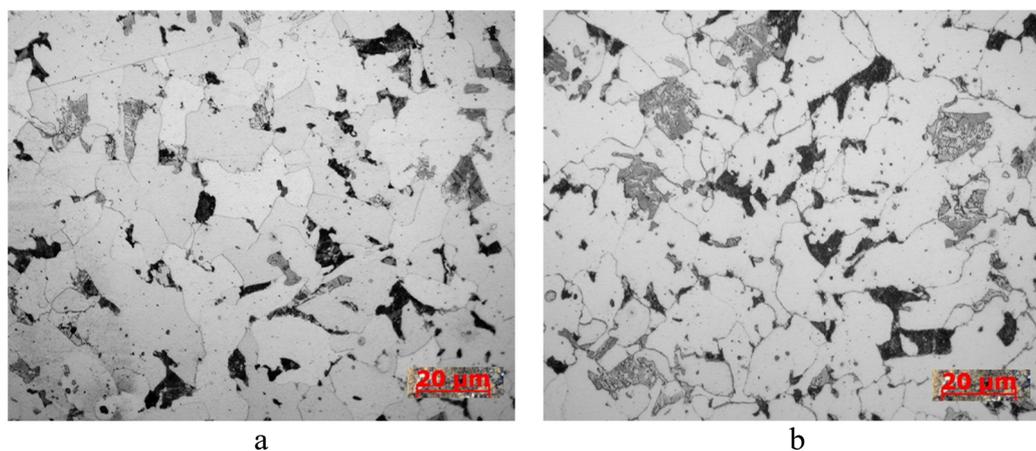


Figure 2. Microstructure of wire rod by C - Mn - Si - steel without B (a) and with B - adding (b)

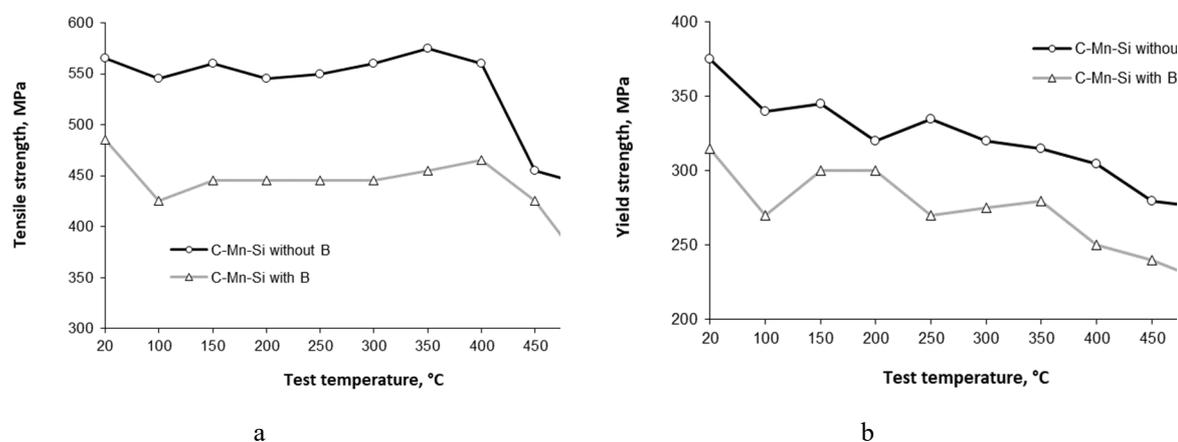


Figure 3. The relationship between tensile test temperature and both TS (a) and YS (b) for 5.5-mm-diameter wire rod by C - Mn - Si - steel with B and without B

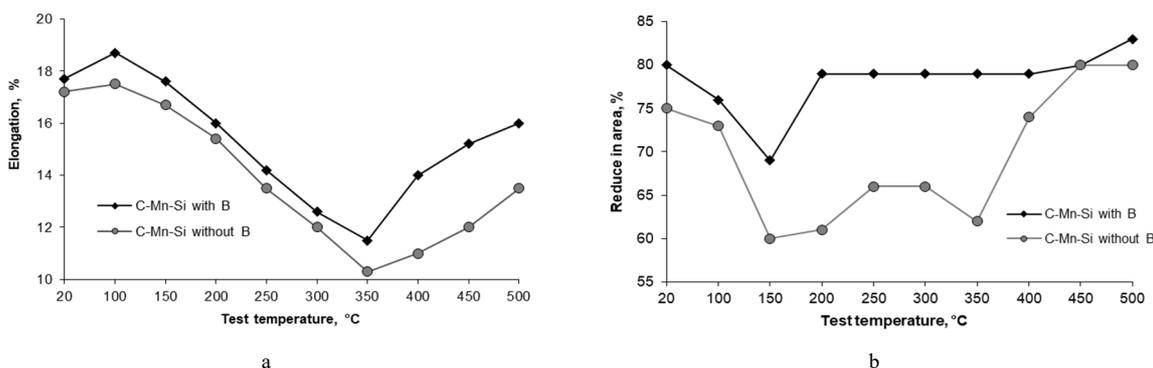


Figure 4. An influence of tensile test temperatures on characteristics of ductility EL_{100} (a) and RA (b) in 5.5-mm-diameter wire rod by C – Mn – Si – steel with B – adding and without B

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