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CORROSION BEHAVIOUR OF SINTERED AISI 316L STAINLESS STEEL MODIFIED WITH BORON-RICH MASTER ALLOY IN 0.5M NaCl WATER SOLUTION

ODPORNOŚĆ KOROZYJNA W ŚRODOWISKU 0.5M ROZTWORU WODNEGO NaCl SPIEKÓW AUSTENITYCZNEJ STALI NIERDZEWNEJ 316L MODYFIKOWANYCH BOREM

Present study describes results of research conducted on sinters manufactured from a powdered AISI 316L austenitic stainless steel modified with an addition of boron-rich master alloy. The main aim was to study impact of the master alloy addition on a corrosion resistance of sinters in 0.5M water solution of NaCl. In order to achieve it, a potentiodynamic method was used. Corrosion tests results were also supplemented with a microstructures of near-surface areas. Scanning electron microscope pictures of a corroded surfaces previously exposed to the corrosive environment were taken and compared. It was successful to increase the corrosion resistance of AISI 316L sinters modified with master alloy. It was also successful in particular samples to obtain a densified superficial layer not only on the sinters sintered in the hydrogen but also on sinters sintered in the vacuum. No linear correlation between presence of the densified superficial layer and the enhanced corrosion resistance was noticed.

Keywords: AISI 316L, boron, sintering, corrosion, layer.

W poniższym artykule zaprezentowano wyniki badań, jakie zostały przeprowadzone na spiekach wykonanych z proszku austenitycznej stali nierdzewnej AISI 316L z dodatkiem mikroproszku bogatej w bor zaprawy. Głównym celem było poznanie wpływu, jaki ma dodatek zaprawy na odporność korozyjną badanych spieków w środowisku 0.5M roztworu wodnego NaCl. W tym celu użyto metody potencjo- dynamicznej. Wyniki badań korozyjnych zostały uzupełnione o mikrostruktury strefy przypowierzchniowej. Wykonano również zdjęcia powierzchni poddanych testom korozyjnym przy użyciu metody SEM. Powodzeniem zakończyły się próby podwyższenia odporności korozyjnej spieków AISI 316L poprzez wprowadzenie bogatej w bor zaprawy zarówno dla spieków spieczonych w atmosferze wodoru, jak i tych spieczonych w próżni. W wybranych spiekach uzyskana została zagęszczona warstwa przypowierzchniowa nie tylko na spiekach spiekanych w atmosferze wodoru, ale również na spiekach spiekanych w próżni, .

1. Introduction

A classical sintering process is mainly utilized for manufacturing objects of a relatively simple shape and of small dimensions. It's main advantage lays in production of massive volumes of repetitive parts in an economical way. Usually, parts manufactured in this way characterize with some residual porosity which lowers mechanical properties and what is more important, in some cases allows the corrosive environment to penetrate a bulk material [1-2]. An increasing demand for sintered stainless steel parts results in growing interest of researchers into a phenomena of sinters corrosion [3, 4]. This penetration intensifies pitting corrosion which is the most popular and the most dangerous kind of corrosion observed in stainless steels. Infiltration, double pressing or forging are just examples of methods utilized for closing up the porosity and for increasing the sinters density. Alternative way to achieve this

goal is to modify a powder blend with an element or a master alloy addition effecting in a liquid phase occurrence during sintering process [5-14]. A proper liquid phase may intensify sintering process in a way resulting in an almost full density of the sinter. According to the research results of German [15], boron is one of the most efficient sintering activator of ferrous alloys and enhances sintering by occurrence of a low melting eutectic phase: E(Fe₂B+Fe). The sintering enhancement occurs by means of: lowering friction between neighboring particles allowing for their rearrangement, particles fragmentation, grains rearrangement, dissolving and precipitating as well as pores filling. All those effects increase transport of matter during sintering process [15-17]. Attractiveness of boron-induced liquid phase sintering lays also in fact that in some particular process conditions a dense superficial layer may be obtained. Such layer which almost do not contain borides, consist of one phase and do not have any opened pores what greatly increases

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the corrosion resistance by minimizing reaction surface. These features classifies sintering of AISI 316L modified with boron as an attractive technological goal to master due to promise of manufacturing ready-to-use parts in one-step process. In this investigation elemental boron was replaced by the boron-rich and patent protected low-melting master alloy in order to intensify the boron reaction with the base powder.

2. Experimental procedure

Test samples were prepared by one-side cold compaction process under pressure of 600MPa. In order to avoid the material polluting, only die walls and punches were layered with lubricant – zinc stearate. Manufactured samples were of cylindrical shape of dimensions: radius equal to 10 [mm] and height equal to 5 [mm]. The powdered AISI 316L austenitic stainless steel provided by Hoganas AB was used as a base powder. Introduction of the boron-rich master alloy was implemented by 24 hour mixing in Turbula® mixer. The utilized master alloy was a patent protected boron-rich master alloy based on following elements: Fe, Si, Mn and Cr. Proper master alloy amounts were added to introduce precisely 0.0 [wt.%], 0.1 [wt.%], 0.2 [wt.%], 0.3 [wt.%], 0.4 [wt.%] of boron element into the base powder. Sintering was performed in Nabertherm P330 tubular furnace. Vacuum and hydrogen were used as sintering atmospheres. temperature profile utilized for sintering was as follows:

- temperature stabilization to 30 [°C] for 30 [min]
- heating up to 1240 [°C] with heating rate of 10 [°C/min]
- isothermal sintering at the temperature of 1240 [°C] for 30 [min]
- cooling down to 30 [°C] with cooling rate of 20 [°C/min]
- temperature stabilization to 30 [°C] for 60 [min]

For sintering in atmosphere of hydrogen, gas flow was strictly controlled and was equal to 1.6 [m/h] over the sample. For sintering atmospheres hydrogen of 99.9999% purity was used while for sintering in vacuum an atmospheric air of 10^{-2} Pa pressure was used.

Corrosion resistance tests were performed on an ATLAS 0531 EU&IA device manufactured by Atlas-Solich. The tests were performed at room temperature in a stationary liquid (without flow). 0.5M water solution of NaCl was utilized for estimation tendency of the sinters for corrosion. The samples were used as electrodes. As reference electrode a saturated calomel electrode was used. The calomel electrode was characterized with a potential of +240 [mV] in comparison to the hydrogen electrode. Platinum electrode was used as an auxiliary electrode. Total tested area on each sample was equal to 1.33 [cm²].

Changes of open circuit potential (OPC) were measured in time. Polarization curves were made using potentiodynamic method. Changes rate of a potential of tested samples was equal to 1 [mV/s]. The polarization of samples was realized from potential equal to value lower than stationary potential of approx. 250 [mV] up to value of 1500 [mV]. Tafel curves were plotted in a range of –250 [mV] ÷ +250 [mV] around the corrosion potential. Polarization resistance for tested samples was calculated using current measurements in a potential range from –20 [mV] ÷ +20 [mV] around the corrosive potential.

Basing on obtained data following values were calculated: corrosion potential, corrosion current, polarization resistance, critical potential, critical current, passivation potential, passive current and pitting potential.

3. Results and discussion

Fig. 1 presents changes of open circuit potential as a function of time for 316L powdered steel sintered in dry hydrogen and vacuum. The tested steels were modified with boron-containing master alloy in proper amount to introduce from 0.0 [wt.%] up to 0.4 [wt.%].
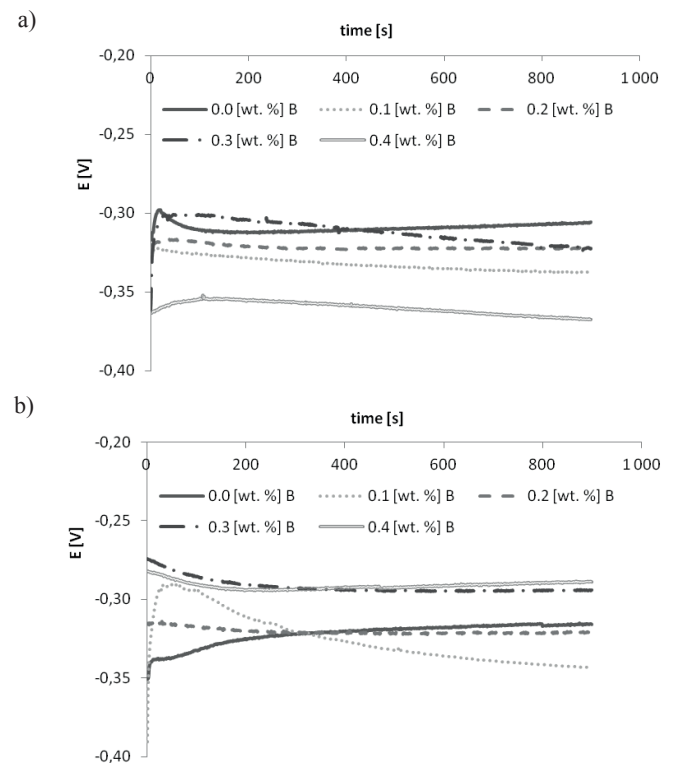

Fig. 1. OCP potential as a function of time for 316L stainless steel modified with boron in 0.0 [wt.%] - 0.4 [wt.%]. sintered in a) dry hydrogen, b) vacuum.

In case of both utilized sintering atmospheres, a non-modified sintered 316L steel showed consequent increase of OCP potential according to time lapse which indicated tendency of those sinters for passivation. Although for 316L sintered in vacuum a depassivation may be noticed after the initial stage of the passivation what was highlighted by a peak in recorded data. After that, consequent passivation took place. Introduction of boron strongly influenced the corrosion behavior of tested 316L stainless steel sinters. In case of 316L modified with 0.1 wt% of boron (especially sintered in hydrogen) at the beginning of the curve a significant increase of OCP value might be noticed. This OCP value increase informed about creation of a passive layer, however following constant decrease of the potential along with time lapse indicated a considerable intensity of corrosive phenomena. For the samples sintered in vacuum and modified with higher amounts of boron than 0.1 [wt. %] (Fig. 1b) the recorded curves had similar progress.

For samples modified with boron amount from 0.2 [wt.%] up to 0.4 [wt. %] and sintered in hydrogen, OCP potential decreased slightly along with time lapse and then became stabilized.

For samples sintered in hydrogen atmosphere according to boron amount increase from 0.1 [wt. %] up to 0.4 [wt. %] the OCP potential shifted into direction of more positive values. While for their counterparts sintered in vacuum similar dependence was not noticed. The reason was an OCP potential shift into negative values for samples modified with boron from 0.2 [wt. %] up to 0.4 [wt. %]. Potentiodynamic polarization curves presenting a current density dependence from potential (in half logarithmic scale) in a range from -0.65 up to + 1.5 [V] for samples sintered in hydrogen and vacuum were plotted in Fig. 2. Registration of polarization curves in a wide anodic range opened a possibility of determining potential ranges responsible for e.g. passivation.

Indifferently on the sintering atmosphere or boron addition introduced into powdered AISI 316L steel, all presented curves (Fig. 2) showed consequent active, active-passive, passive and trans-passive regions. Passive region was wide and clear but the current in this range changed slightly what is characteristic phenomenon for sintered metallic materials. Current densities in cathodic and anodic ranges were comparable.

Introducing boron to the AISI 316L stainless steel did not have a significant impact on a character of the current density dependence on the potential. However it decreased the potential and density of the passivation current which was clearly visible for sinters sintered in vacuum. Polarization curves recorded during potentiodynamic tests for all tested samples were similar. One could see that for samples modified with 0.3 [wt.%] and 0.4 [wt. %] they overlap almost in the whole range.
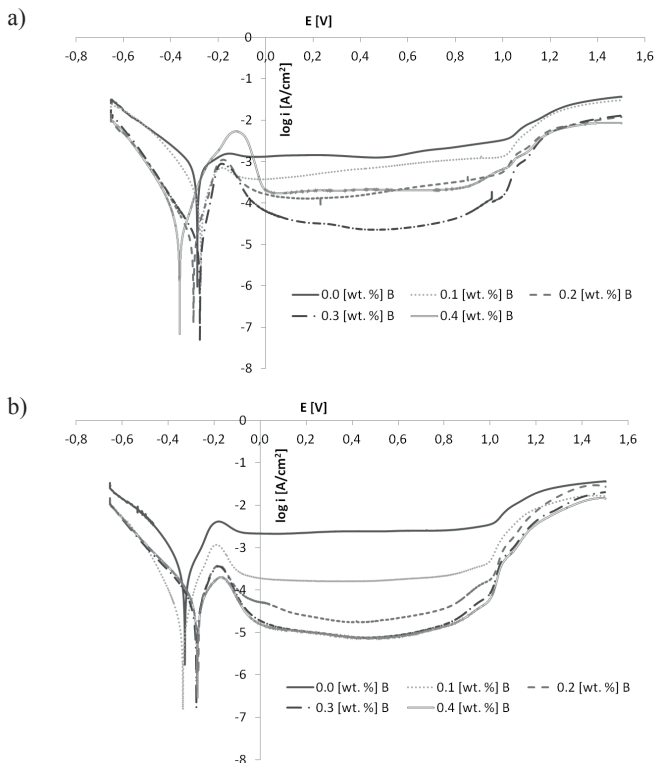
a)

b)

Fig. 2. Polarization curves for austenitic stainless steel AISI 316L modified with boron in amount from 0.0 [wt. %] up to 0.4 [wt. %] and sintered in a) hydrogen, b) vacuum.

An analysis of obtained polarization curves allowed for a determination of following electrochemical parameters: corrosion potential (E_{corr}), corrosion current (i_{corr}), polarization resistance (R_{pol}), anodic and cathodic Tafel coefficient (b_a, b_c), critical potential (E_{crit}), critical current (i_{crit}), passivation potential (E_{pass}), passive current (i_{pass}) and pitting potential (E_{pitt}). All these parameters describing the corrosion resistance and corrosive behavior were presented in Tab.1

The least value of the polarization resistance among all tested samples was recorded for the non-modified steel. This observation is true for samples sintered in hydrogen as well as in vacuum. Along with boron content increase in tested sinters polarization resistance increased and reached maximum for sinter modified with 0.2 [wt. %] of boron. Exceeding the amount of 0.2 [wt. %] of boron led to a slight decrease of polarization resistance but its value was still much higher than for the non modified sinter. This dependence is true for both of tested atmospheres. Another worth noticing fact was that for samples sintered in vacuum and modified with 0.0 [wt.%] or 0.1 [wt.%] the polarization resistance was much higher than for corresponding samples sintered in hydrogen. Reaching or exceeding boron amount equal to 0.2 [wt.%] reverted this status. Samples with higher amount than 0.2 [wt.%] of boron and sintered in hydrogen characterized with higher polarization resistance than those sintered in vacuum. According to data presented in Tab.1 it might be concluded that the highest corrosion resistance in 0.5M NaCl showed AISI 316L steel modified with boron in 0,2 – 0.3 [wt. %] after sintering in hydrogen.

An inverse correlation exist between polarization resistance and corrosion current. The sintered non-modified AISI 316L steel which showed the least value of polarization resistance was also characterized with the highest value of corrosion current. Worth noticing is also a fact that along with boron content increase, a value of icorr was increasing slightly, especially in case of samples sintered in vacuum. The least density of the corrosion current equal to $-0.11 \cdot 10^{-4}$ [A/cm^2] was recorded for the sample modified with 0.2 [wt.%] of boron and sintered in hydrogen. After comparison of corrosion current values it may be concluded, that the highest corrosion resistance was showed by samples modified with boron addition equal to 0.2 [wt. %] and 0.3 [wt. %] while the least corrosive resistance was obtained for the non-modified samples.

An influence of sintering atmosphere as well as the boron amount introduced to powdered steel on corrosion potential is similar to their impact on the polarization resistance.

For non-modified AISI 316L and the one with 0.1 [wt.%] of boron and sintered in vacuum the corrosion potential value was much more negative than for corresponding samples sintered in hydrogen. In a case of sample modified with 0.1 [wt.%] this relation was opposite. Comparing obtained results with the corrosion potential of samples without boron addition, the increase of corrosion resistance can be seen.

Introducing boron into AISI 316L steel powder and utilization of vacuum as the sintering atmosphere lowered the density of critical current. Along with boron content increase from 0.1 [wt.%] up to 0.4 [wt.%] a value of the icorr tended to decrease from $1.15 \cdot 10^{-3}$ down to $0.20 \cdot 10^{-3}$ [A/cm^2] respectively. The same coincidence occurred in case of

TABLE 1

Parameters characterizing corrosion resistance of 316L steel modified with boron in amounts from 0.0 [wt.%] up to 0.4 [wt.%]. after sintering in hydrogen and in vacuum

	Boron addition [wt. %]				
	0	0.1	0.2	0.3	0.4
	Hydrogen				
R_{pol} [$\Omega \cdot cm^2$]	35	175	1009	1001	816
E_{corr} [V]	-0.288	- 0.272	- 0.272	- 0.277	- 0.362
i_{corr} [A/cm^2]	- $6.15 \cdot 10^{-4}$	- $0.63 \cdot 10^{-4}$	- $0.11 \cdot 10^{-4}$	- $0.13 \cdot 10^{-4}$	- $0.34 \cdot 10^{-4}$
b_a [V]	0.262	0.048	0.050	0.035	0.080
b_c [V]	- 0.197	- 0.074	- 0.086	- 0.115	- 0.097
E_{crit} [V]	- 0.152	- 0.183	- 0.179	- 0.185	- 0.124
i_{crit} [A/cm^2]	$1.57 \cdot 10^{-3}$	$0.71 \cdot 10^{-3}$	$1.11 \cdot 10^{-3}$	$0.89 \cdot 10^{-3}$	$5.35 \cdot 10^{-3}$
E_{pass} [V]	- 0.022	- 0.091	- 0.102	- 0.102	- 0.048
i_{pass} [A/cm^2]	$1.35 \cdot 10^{-3}$	$0.41 \cdot 10^{-3}$	$0.33 \cdot 10^{-3}$	$0.26 \cdot 10^{-3}$	$1.01 \cdot 10^{-3}$
E_{pitt} [V]	1.017	1.018	1.019	1.019	0.807
$i_{E=0,5\ V}$ [A/cm^2]	$1.28 \cdot 10^{-3}$	$0.75 \cdot 10^{-3}$	$0.19 \cdot 10^{-3}$	$0.02 \cdot 10^{-3}$	$0.21 \cdot 10^{-3}$
	Vacuum				
R_{pol} [$\Omega \cdot cm^2$]	92	378	602	498	461
E_{corr} [V]	-0.327	- 0.333	- 0.268	- 0.276	- 0.272
i_{corr} [A/cm^2]	- $1.72 \cdot 10^{-4}$	- $0.16 \cdot 10^{-4}$	- $0.33 \cdot 10^{-4}$	- $0.30 \cdot 10^{-4}$	- $0.60 \cdot 10^{-4}$
b_a [V]	0.093	0.081	0.062	0.113	0.175
b_c [V]	- 0.079	- 0.055	- 0.124	- 0.068	- 0.159
E_{crit} [V]	- 0.180	- 0.189	- 0.179	- 0.180	- 0.168
i_{crit} [A/cm^2]	$4.09 \cdot 10^{-3}$	$1.15 \cdot 10^{-3}$	$0.39 \cdot 10^{-3}$	$0.36 \cdot 10^{-3}$	$0.20 \cdot 10^{-3}$
E_{pass} [V]	- 0.104	- 0.102	- 0.090	- 0.063	- 0.058
i_{pass} [A/cm^2]	$2.49 \cdot 10^{-3}$	$0.29 \cdot 10^{-3}$	$0.91 \cdot 10^{-3}$	$0.36 \cdot 10^{-3}$	$0.28 \cdot 10^{-3}$
E_{pitt} [V]	1.018	1.019	1.021	1.018	0.986
$i_{E=0,5\ V}$ [A/cm^2]	$2.40 \cdot 10^{-3}$	$1.62 \cdot 10^{-3}$	$1.86 \cdot 10^{-5}$	$7.52 \cdot 10^{-6}$	$7.37 \cdot 10^{-6}$

utilizing hydrogen as a sintering atmosphere. An exception was the sample with 0.4 [wt.%] of boron sintered in hydrogen – in this case the critical current was over three times higher than the value recorded for non-modified sample.

On the other hand, the critical potential of samples modified with boron tended to shift in direction of more positive values along with boron addition increase for each of utilized atmospheres. An exception was the sample containing 0.4 [wt.%] of boron. For this sample Ecrit reached a value more positive comparing to AISI 316L sinter non-modified with boron.

Stability of formed passive layer on boron modified AISI 316L sinter was significant what was confirmed by a high value of passivation potential as well as by low values of current density in the passive region. The increasing boron content starting from 0.1 [wt.%] up to 0.4 [wt.%] resulted in a decrease of average value of passive current and in increase of passivation potential. A deviation from this dependence was observed for sinter modified with 0.4 [wt.%] sintered in hydrogen. Also the value of i_E=0,5 [V] decreased along with boron content increase in a range of boron addition up to 0.3

[wt.%]. In case of sinter modified with 0.4 [wt.%] of boron this effect was not observed. Boron addition to sinters made an impact also on the passive region. Sintering process performed in hydrogen atmosphere effected with an extending passive region along with boron content increase. A reverse effect was observed when the sintering process was performed in vacuum – the passive region was consequently shortened with increasing boron addition.

Assessing the boron influence on pitting potential, one could say that its addition caused a slight increase of E_{pitt} value. By taking into consideration obtained results one could say that the highest corrosive resistance had the sample modified with boron both in 0.2 [wt.%] sintered in hydrogen.

Before the corrosion tests were performed, a metallographic cross-sections were prepared in direction perpendicular to investigated surface. A comparison of cross-sections of all samples in hydrogen were plotted in Table. 2.

The more boron was introduced the less pores were left in the microstructure. Reaching boron addition in samples equal to 0.2 [wt.%] or more resulted in almost complete elimination of opened porosity, and, what is more, in cases of 0.3 [wt.%]

and 0.4 [wt.%] of boron addition there was almost no opened porosity. The superficial densified layer without precipitations observed by other authors was obtained not only in case of 0.4 [wt.%] of boron addition, but also in case of 0.3 [wt.%] of boron addition [18]. Probable reason was master alloy particles low melting point (approx. 967 [°C])[19]. Though the liquid phase was formed at much lower temperature than in case of using the elemental boron where the liquid is formed at temperature of an eutectic reaction – approx. 1177 [°C].

TABLE 2

Superficial layer of samples sintered in hydrogen

Boron addition [wt..%]				
0.0	0.1	0.2	0.3	0.4
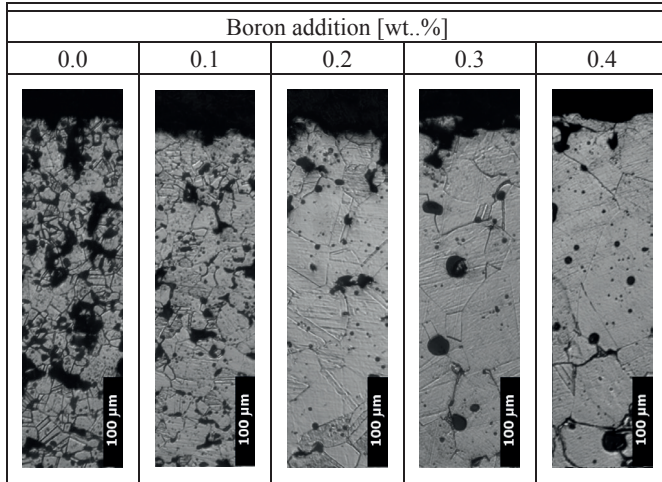
An effect of the superficial layer creation was observed also in case of sintering compacts in vacuum (Table 3). In fact, till now the creation of the superficial layer was observed only in samples sintered in hydrogen.

TABLE 3

Superficial layer of samples sintered in vacuum

Boron addition [wt..%]				
0.0	0.1	0.2	0.3	0.4
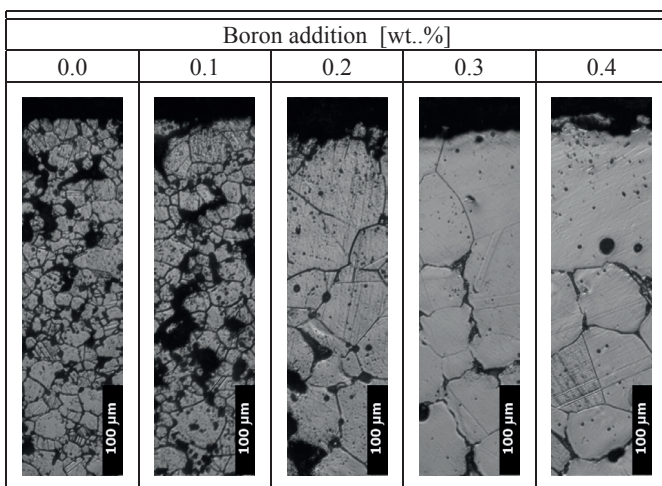
The observed superficial dense layer is almost deprived of opened porosity (Fig. 3) This superficial densification was the main reason of increase in corrosion resistance of sinters with amount of boron equal to 0.2 [wt.%] and higher. In the investigated material there were two different phenomena influencing the corrosion resistance oppositely. The first one was a densification process of superficial layer while the second one was the creation of borides on grain boundaries. Densification of superficial layer increased the corrosion resistance while the presence of borides decreased it by creation of electrochemical circuits. When a high amount of borides is present simultaneously with high opened porosity, the corrosion resistance drops – especially noticeable for samples sintered in vacuum. For samples sintered in hydrogen the lesser was opened porosity the higher was corrosion resistance. Figure 3 presents impact of those phenomena on resistance of polarization.
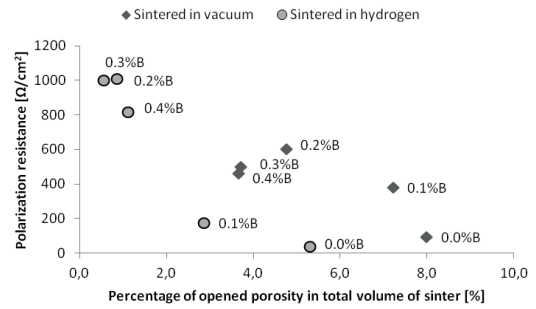

Fig. 3 Polarization resistance as a function of opened porosity percentage.

Increase of boron addition resulted in increase of borides volume [18] thus a probability of a corrosive liquid contact with some residual borides present on sinters surfaces is also increased. A drop of polarization resistance observed for samples with high addition of boron in form of master alloy were caused by increasing amount of borides placed on the grain boundaries.

After the corrosion tests, samples surfaces exposed to corrosion were investigated under a scanning electron microscope. Data placed in a Table 4 presents the comparison of surfaces after corrosion tests of samples with 0.0 and 0.4 [wt. %] of boron sintered in vacuum and hydrogen as well. Multiple pits found on the surfaces of non-modified samples after corrosion tests originated from multiple opened pores which were present before the test started. On the other hand, almost no corrosive pits were observed on samples modified with 0.4 [wt.%] of boron. Superficial dense layer stopped the corrosive liquid from penetrating into the sample, significantly reducing an area of contact. An analysis of samples surface modified with 0.4 [wt.%] of the boron and sintered in the vacuum showed, that most probably corrosion started with a formation of electrochemical circuits between the residual borides and matrix. Intensified corrosion started in their joints, so the less precipitations were present on the surface the more resistant to corrosion the material was.

Summarizing, introducing boron in a form of master alloy into austenitic stainless steel AISI 316L had impact on its corrosion resistance in the environment of 0.5 M NaCl. The main effects were:
- increase of polarization resistance value,
- decrease of the value of anodic and increase of cathodic Tafel's coefficients,

- decrease of: corrosion current, critical current (except the sample modified with 0.4 [wt.%] of boron), passive current and density of the current in the passive region.
- shift of passivation potential as well as critical potential (except the sample modified with 0.4 [wt.%] of boron) into negative values.
- shift of corrosion potential and pitting potential into positive values (except the sample modified with 0.4 [wt.%] of boron)

However, when sintering process was performed in vacuum, boron addition in base steel powder took its impact as:

- increased value of polarization resistance
- decreased corrosion current and critical current values.
- decreased value of passive current and current density in the passive range.
- shift of critical potential (an exception is sample modified with 0.4 [wt.%] of boron) in a direction of negative values.
- shift of corrosion potential, passivation potential and pitting potential in a direction of positive values.

TABLE 4

Comparison of surfaces after corrosion test

	Sintered in hydrogen	Sintered in vacuum
0.0 [wt. %] B		
0.4 [wt. %] B		
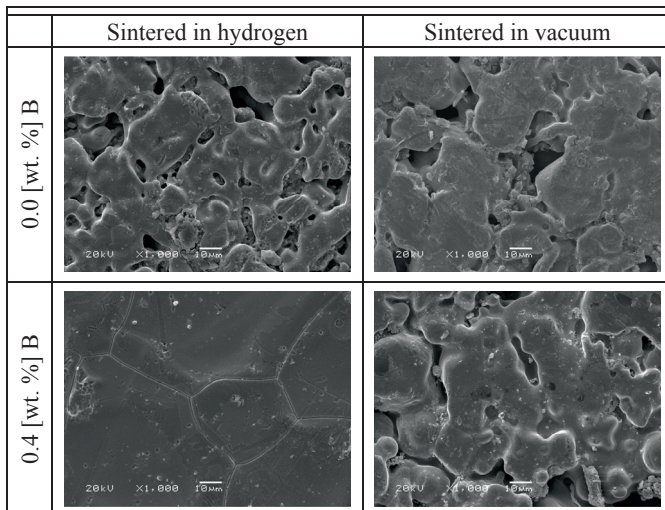
4. Conclusions

Basing on results of performed corrosion tests one could say, that usage of master alloy significantly increase corrosion resistance of samples sintered both in hydrogen and in vacuum. In most cases utilization of hydrogen atmosphere results in higher corrosion resistance than using the vacuum does. Observations of superficial layer on samples sintered in vacuum suggest that the molten master alloy facilitates a reduction of oxides layer placed on powder particles which is necessary for a successful sintering process. Utilization of master alloy increases a potential application area of AISI 316L sinters by allowing for obtaining densified layer which

was not observed in this sintering conditions previously at all.

Obtained data suggest that the highest corrosion resistance in environment of 0.5 M water solution of NaCl may be assured by introducing 0.2 [wt.%] or 0.3 [wt.%] of boron combined with the sintering process performed in hydrogen. Results also suggest that further increase of boron amount will not increase the corrosive resistance of a sinter.

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