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THE EFFECT OF THE ADDITION OF BORON ON THE DENSIFICATION, MICROSTRUCTURE AND PROPERTIES OF SINTERED 17-4 PH STAINLESS STEEL

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Abstract

It is generally known that boron is an effective activator of the sintering process of iron as well as stainless steels. During sintering, boron contributes to the formation of a liquid phase wetting the surfaces of powder particles. As a consequence, a reduction of porosity, the rounding of pores and an increase in density is obtained. It is necessary to improve mechanical properties and corrosion resistance. The aim of this study is to investigate the effect of the addition of boron in the amounts of 0.2, 0.4 and 0.6% wt. on the density, microstructure and selected properties of sintered 17-4 PH stainless steel.

Keywords: 17-4 PH steel, boron, microstructure, sintering, dilatometry

Streszczenie

Bor jest skutecznym aktywatorem procesu spiekania żelaza, ale także stali nierdzewnych. Podczas spiekania bor aktywuje proces spiekania w wyniku pojawienia się cieczy zwilżającej powierzchnie cząstek proszków. W konsekwencji przyczynia się do zmniejszenia porowatości, zaokrąglenia porów i wzrostu gęstości niezbędnego do poprawy właściwości mechanicznych i odporności na korozję. Celem przeprowadzonych badań było wyjaśnienie wpływu dodatku boru w ilości 0,2, 0,4 i 0,6 % ciężaru w postaci elementarnego proszku na kształtowanie się mikrostruktury i właściwości utwardzanych wydzieleniowo stali nierdzewnych gatunku 17-4 PH.

Słowa kluczowe: stal 17-4 PH, bor, mikrostruktura, spiekanie, dylatometria.

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1. Introduction

PM stainless steels were developed in order to improve on the mechanical properties offered by their wrought counterparts. Precipitation hardening (PH) stainless steels are defined by the strengthening mechanism [1]. The strengthening of these alloys is developed by aging. Several elements are commonly used for the precipitation reaction, including aluminum, copper and titanium. Because aluminum and titanium have high affinities for nitrogen and oxygen, it is difficult to sinter without the formation of oxides or nitrides in PM stainless steels. For this reason, it is necessary to strictly control the atmosphere during sintering. Therefore, copper is the most commonly element used in precipitation hardening steels. It is known that excessive amounts of copper can lead to lower densities of sintered steels, this is due to its negative effect on compaction. Consequently, their mechanical properties are inferior due to their limited final density [1–5]. It is known that the combination of a relatively high temperature (usually above 1350°C) and an extended sintering time should be used in order to obtain a high density of sintered 17-4 PH steel [6–8].

It should be pointed out that one of the most common PH stainless steel grades is 17-4 PH [1–7, 9, 10]. This is a martensitic stainless steel containing approximately 3% (by mass) of Cu and is strengthened by the precipitation of highly dispersed copper particles in the martensite matrix. 17-4 PH exhibits high strength, high apparent hardness and superior corrosion resistance compared with martensitic stainless steels [1–3, 8]. The ductility and toughness of this steel are generally higher than in the carbon-containing martensitic grades [3]. 17-4 PH combines high strength and good corrosion resistance at a reasonable cost [6–12]. In addition, the mechanical properties of sintered 17-4 PH steel can be improved by heat treatment [1–3, 5, 11]. Due to these properties, 17-4 PH has widespread applications in the automotive, aerospace, military, chemical and food processing industries [2, 6–7, 9–11]. 17-4 PH stainless steel can also be used in medicine as biomaterial [10–11].

If we compare wrought and powder metallurgy materials, a disadvantage of sintered stainless steels is the deleterious effect of porosity on mechanical properties such as tensile strength, ductility and impact toughness [5]. It is known that a high density of sintered materials is necessary for improved mechanical properties and corrosion resistance. It is not surprising that residual pores decrease the above mentioned properties in sintered stainless steel [6, 7]. It is worth mentioning that the properties of interfaces (for example, the presence of hard and brittle phases) are also important for mechanical properties and the corrosion resistance of sintered stainless steels.

Analysis of the literature leads to the conclusion that the introduction of boron as a sintering additive to iron-based material may lead to a noticeable improvement in the total density and the mechanical properties. Several pieces of research indicate that boron is an excellent activator for the sintering of ferrous alloys [6, 7, 9–11, 13–27].

The analysis of a binary Fe-B diagram phase leads to the conclusion that boron is the ideal sintering additive. Namely the eutectic reaction ($\text{Fe}_2\text{B} + \text{Fe} \rightarrow \text{liquid}$) occurs at 1174°C [11, 18–22]. The solubility of boron in iron is very low, whereas the solubility of iron in the eutectic melt is high. The liquid phase has a very low solubility in iron and provides a continuous network between the solid grains [20]. The eutectic phase has a low melting point, and therefore even small amounts of boron should be sufficient to generate a fast mass transport after the liquid phase formation [22]. It is not surprising that the higher the

boron content, the higher the amount of eutectic [18]. In addition, boron has a high affinity to oxygen. Therefore, during the sintering process, boron reacts with oxygen which is chemically bounded on the surface of powder particles. This simultaneously activates the sintering process [7, 25, 26]. As a consequence, this results in higher densities, rounded pores, and improved mechanical properties [18]. According to [6, 7, 9], the addition of boron increases the hardenability of steels, improves grain boundary and cohesive strength, and enhances corrosion resistance.

The earliest studies were related to the addition of boron to iron, wherein boron was used in various forms – elemental boron powder (crystalline or amorphous) or borides, such as FeB, Fe₂B. These studies focused on the effect of: powder characteristics, form and amount of boron addition, and also sintering atmosphere, sintering temperature, sintering time on microstructure, densification and mechanical properties of sintered. The conclusions of these studies are that the addition of boron to iron resulted in greater densification and a noticeable improvement in the mechanical properties compared to sintered iron [6].

Many investigators were involved in studies concerning the addition of boron to iron-based materials such as: Astaloy Mo [14, 22]; Astaloy CrL [22]; Astaloy CrM [16]; Distaloy AE [20]; Distaloy SA [13, 23]; austenitic stainless steel (mainly 316L) [19, 21, 25, 27, 28]; ferritic stainless steel [18, 23, 24]; martensitic stainless steel [17, 29]; 17-4 PH steel [6, 7 9–11]. Boron was introduced in different forms and different mass percentages. Thus, the following forms of boron were used – an elemental boron powder [10, 11, 22–26] or compounds such as: FeB [17, 18, 20]; h-BN [21, 22]; B₄C [16, 22]; CrB₂ [28]; NiB [7] and boron containing master alloy [16, 19, 27]. Their studies showed that the addition of boron increased density (at a lower sintering temperature), mechanical properties and corrosion resistance. Thus, boron can be successfully applied to enhance the sintering process and obtain high-density sintered iron-based alloys [19].

There are only a few studies regarding the 17-4 PH steel with boron or different borides in the literature [6, 7, 9]. The sintering behaviour and properties of an injection molded 17-4 PH stainless steel with additions of elemental boron [9–11, 22, 24, 29], FeB [6] and nickel boride [7] were studied. The addition of elemental boron as well as FeB and NiB increased the sintered density, the ultimate tensile strength, elongation, impact energy and hardness of 17-4 PH steel. Moreover, it decreased the time and temperature of sintering process [6, 7, 9].

In accordance with the presented results, the full density and the highest mechanical properties were obtained with the addition of 0.5% (by mass) of boron at 1250°C for 30 min [9] and 1% (by mass) of NiB at 1280°C for 45 min [7]. The heat treatment (a solution treatment in argon for 1 hour at 1050°C, and then aging treatment in argon for 4 hours at 480°C) after sintering resulted in a further increase of mechanical properties. For example, the properties of heat treated 17-4 PH steel containing 0.5% (by mass) of B were an ultimate tensile strength of 1520 MPa and a hardness of 55.1 HRC [9]. Whereas in the case of heat treated 17-4 PH steel containing 1% (by mass) of NiB the following properties were obtained: ultimate tensile strength of 1402 MPa; elongation of 4.8%; impact energy of 23 J; hardness of 52.3 HRC [7].

Based on the review of literature, it can be concluded that there is virtually no information about the effect of boron addition on 17-4 PH stainless steel produced by conventional powder metallurgy.

It is known that the combination of a relatively high temperature (usually above 1350°C) and an extended sintering time should be used in order to obtain a high density of sintered 17-4 PH steel [6, 7]. To facilitate the sintering process of 17-4 PH stainless steel, boron was added to create a liquid eutectic phase. Thus, the aim of this investigation was to study the effect of the elemental boron powder on the densification, microstructure and sintering behaviour of 17-4 PH steel.

2. Materials for research

In this research, water atomized 17-4 PH stainless steel powder provided by AMETEK was used. The chemical composition of 17-4 PH powder (corresponding with standards: ASTM-A564 grade 630; UNS S17400) was given in Fig. 1. It should be noted that the studied powder contained Acrawax lubricant at an amount of 0.75% wt. The apparent density of 17-4 PH powder was 2.54 [g/cm³] while its flow was 31 [s/50 g]. Boron in the form of elemental powder (product of Goodfellow, average particle size of 2 μm, purity of 99.8 %) was used.

The following powder mixtures were prepared:

- 17-4 PH + 0.2% wt. B,
- 17-4 PH + 0.4% wt. B,
- 17-4 PH + 0.6% wt. B.

In order to compare the results, pure 17-4 PH steel powder was also used for the studies.

Table 1

Chemical composition of 17-4 PH stainless steels powder (% wt.)

C	S	Si	Cr	Ni	Cu	Nb	Mn	P	Fe
0.027	0.011	0.73	16.28	4.28	4.04	0.32	0.05	0.015	Bal.

3. Experimental procedure

All investigated mixtures were prepared by mixing in Turbula® mixer. The mixing time was 6 hours. Then mixtures of powders and also powder of 17-4 PH were uniaxial pressed in a rigid matrix at 600 MPa. In this manner, the following samples were obtained: cuboidal samples with dimensions of 4 × 4 × 15 [mm] for dilatometric studies; cylindrical samples of size Ø20 × 5 [mm]. After pressing, the cuboidal samples were sintered in the horizontal NETZSCH 402E dilatometer, while the sintering process of cylindrical samples was carried out in a Nabertherm furnace. All green compacts were sintered in a pure (99.9992%) and dry (dew point below -60°C) hydrogen atmosphere. The flow rate of the gas was 100 ml/min. The temperature of isothermal sintering was 1260°C. The sintering time was 45 minutes. The samples were slowly heated to the isothermal sintering temperature at a rate of 10°C/min. The same rate was applied during the cooling of the samples from sintering temperature to

ambient temperature. In order to remove the lubricant, the samples were held at a temperature of 400°C for 60 minutes during heating.

The density measurements of green compacts were carried out by the geometrical method. The density and porosity of the sintered cylindrical samples were measured by the water-displacement method (according to the demands of the PN-EN ISO 2738:2001 norm).

Before and after sintering, samples were measured to estimate dimensional changes caused by densification. Due to possibility of vaporization of boron and chromium, samples were weighted to estimate mass loss.

The hardness by Vickers method was determined with the computer-aided hardness tester INNOVATEST CV-600.

Metallographic cross-sections were prepared. The microstructural study of the sintered steels was done with Nikon Eclipse ME 600P Light Optical Microscopy and Scanning Electron Microscopy (SEM).

Dilatometric investigations were carried out in the horizontal NETZSCH 402E dilatometer.

4. Results and discussion

Figure 1 shows the results of green and sintered density measurements of unalloyed and boron alloyed 17-4 PH steel. Results obtained through the measuring of the open and closed porosity of the studied steels are presented in Figure 2 by the amount of boron added.

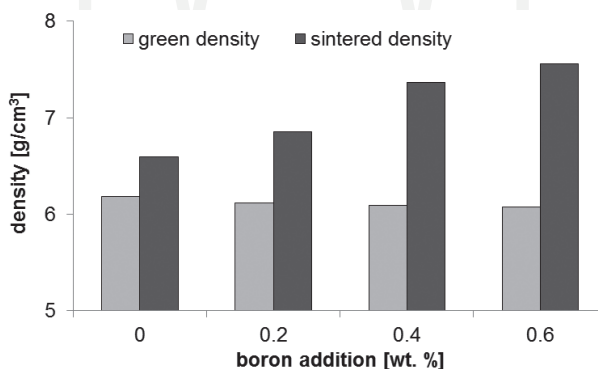


Fig. 1. Green and sintered density of 17-4 PH stainless steel depending on the amount of the addition of boron

The introduction of boron to steel powder caused a slight deterioration of compressibility. The higher the quantity of boron the lower the green density of the studied compacts.

The density of sintered 17-4 PH steel was 6.60 [g/cm³]. It can be observed that the density of boron alloyed steel was higher than the density of sintered 17-4PH steel. It is clear that boron promotes densification of 17-4 PH steel. The degree of densification depends on the amount of boron introduced to the powder mixture. Specifically, the higher the amount of boron introduced to 17-4 PH steel, the higher the sintered density, while an open and closed

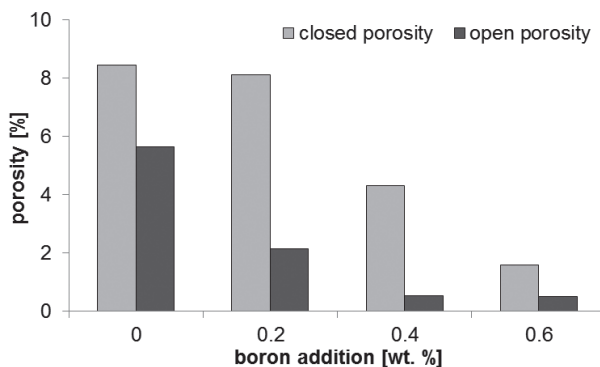


Fig. 2. Open and closed porosity of boron alloyed 17-4 PH stainless steel after sintering in hydrogen at 1260°C

porosity decreases with increasing the amount of boron. It is not surprising that the highest density of boron-alloyed steel was obtained for boron at an amount of 0.6 % wt. This steel had the lowest open and closed porosity.

Measurement of sample heights before and after sintering was carried out in order to designate dimensional changes. The obtained results are presented in Figure 3. The steel samples shrank during sintering. The amount of shrinkage (determined by means of Δh) was primarily dependent upon chemical composition. In the case of pure 17-4 PH steel, dimensional changes of height were approximately 1.5%. It can be observed that the presence of boron contributed to a larger shrinkage of steel samples. Specifically, dimensional changes of samples height increased with increasing of the boron content in powder mixtures. When the boron content was 0.6% wt., Δh reached a level of almost 10%. These results confirm that the addition of boron improves the densification of the 17-4PH stainless steel.

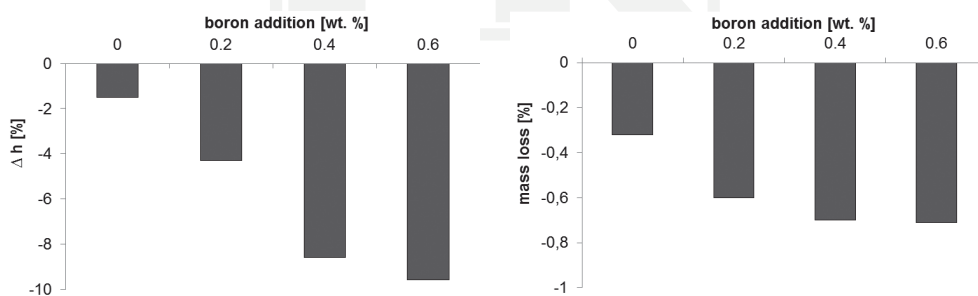


Fig. 3. Dimensional changes of samples height and mass loss of sintered 17-4 PH stainless steel depending on the amount of the addition of boron

The mass loss of samples occurred during sintering for all of the studied steels. The final values obtained after sintering are presented in Figure 3. In the case of sintered 17-4 PH steel, loss of sample mass was associated with the removal of the lubricant. It can be seen that the loss of mass of boron-alloyed steels was much higher in comparison to the value obtained for boron-free 17-4 PH steel and it cannot be exclusively caused by lubricant removal.

The addition of boron improves the densification and also the hardness of the sintered stainless steel 17-4PH. The results of the hardness measurement of studied materials (by the Vickers method) are presented in Figure 4. As can be seen from this figure, the presence of boron effected a significant increase in the hardness of the sintered steel 17-4 PH. The hardness of boron-alloyed steel increases with an increasing proportion of boron. As can be seen in a later section of this article, the introduction of boron changed the microstructure of the investigated steels, and the presence of a hard eutectic phase on the grain boundary has an influence on hardness. The 17-4 PH steel which was modified boron in an amount of 0.6 wt. % obtained two times higher value of hardness in compared with hardness value of boron-free steel.

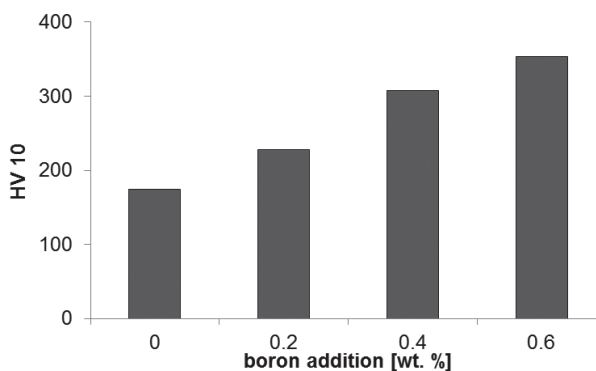


Fig. 4. Hardness of sintered 17-4 PH steel depending on the amount of the addition of boron

The addition of boron to 17-4 PH steel caused a distinct change in the microstructure. It can be observed that the morphology of porosity changed. Specifically, as the boron amount increases, the number of pores reduces. Also the shape of the pore is changed from an irregular shape (existing in the microstructure of sintered stainless 17-4 PH) to a spheroidal shape (for 0.4 and 0.6% wt. of boron added).

The microstructure obtained for stainless steels with boron addition are presented in Fig. 5 and 6. The microstructure of sintered 17-4 PH steel consists of the martensite matrix and δ ferrite. Besides it in the microstructure of boron-alloyed steel appeared the solidified eutectic at the grain boundaries. It is particularly observed for sintered 17-4 PH steel with higher amount of boron.

The SEM microstructure of sintered 17-4 PH steel modified 0.4% wt. of boron is presented in Figure 7. An EDS analysis was performed in order to distinguish the differences in chemical composition of observed constituents: eutectic and matrix. Therefore, the point analyses was performed at points 1, 2 and 3. The results of chemical composition microanalysis are shown in the table below the photograph. Point number 1 was designated in the matrix, which is martensite, while points numbers 2 and 3 were on the grain boundary where the presence of eutectic was previously observed.

Microanalysis of chemical composition indicated that the main elements in point number 1 are Fe, Cr, Ni, Cu – traces of Si also appear. It should be noted that the chromium content in martensite grain is lower than the chromium content in the chemical composition of 17-4 PH

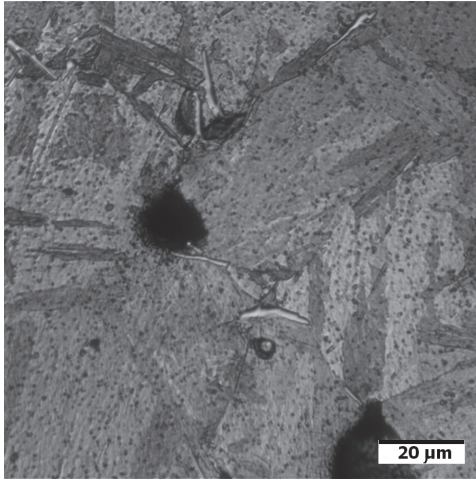


Fig. 5. Microstructure of sintered 17-4 PH steel modified 0.2% wt. of boron

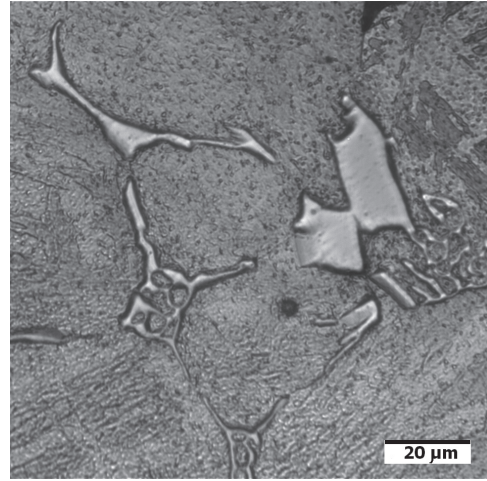
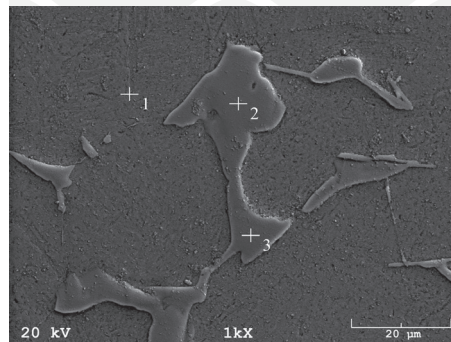


Fig. 6. Microstructure of sintered 17-4 PH steel modified 0.6% wt. of boron



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc.	Units
Point 1					
Si	Ka	7.70	0.292	0.865	wt.%
Cr	Ka	93.00	0.543	14.714	wt.%
Fe	Ka	280.16	1.465	76.040	wt.%
Ni	Ka	10.22	0.619	3.980	wt.%
Cu	Ka	9.23	0.712	4.401	wt.%
Point 2					
Cr	Ka	286.29	1.237	58.865	wt.%
Fe	Ka	129.40	1.315	41.135	wt.%
Point 3					
Cr	Ka	285.71	1.263	58.408	wt.%
Fe	Ka	130.24	1.362	41.592	wt.%

Fig. 7. The SEM microstructure and microanalysis of chemical composition of sintered 17-4 PH – 0.4% wt. of boron

steel (table 1). The main elements in point number 2 and 3 are Fe and Cr. The eutectic occurs on the martensite grain boundaries. This is associated with the Fe – B equilibrium phase diagram. Based on the results of the EDS analysis, it could be concluded that complex iron and chromium borides were formed during heating up to sintering temperature. Subsequently, a liquid phase was formed on grain boundaries as a result of eutectic reaction between the alloy matrix and the borides.

The results of the EDS analysis clearly shows that the boron amount slightly influences the chemical composition of both the matrix and eutectic. Generally, it can be concluded that as boron content increases, the chromium and iron content decrease in the matrix.

Figure 8 presents the sintering behaviour of the investigated steels. The presented dilatometric curves show the dimensional changes which occur during sintering in hydrogen for boron-free as well as boron modified 17-4 PH stainless steels. In the case of boron-free 17-4 PH, three distinct peaks can be distinguished during heating to the temperature of isothermal sintering. The first peak is at a temperature of approximately 800°C. It is associated with the typical for iron-based alloys phase transformation involving tetragonal-to-cubic crystal lattice reconstruction. Water atomized powder of 17-4PH stainless steel has a martensitic microstructure. Thus, martensite (α) – austenite (γ) transformation occurs during heating to the temperature of isothermal sintering. This is accompanied by a reduction in the specific volume and it results in momentary shrinkage. The second peak on the presented curve is at a temperature above 1000°C. This is related to the start of densification, specifically the material transport mechanisms, which lead to shrinkage, start to dominate over the phenomena associated with the thermal expansion. In turn, the third peak is at a temperature above 1200°C. This is associated with the beginning of the δ ferrite formation in the microstructure. δ ferrite specifically enhances the densification process of a sintered sample. The beginning of the δ ferrite formation results in a rapid shrinkage prior to reaching the isothermal sintering temperature.

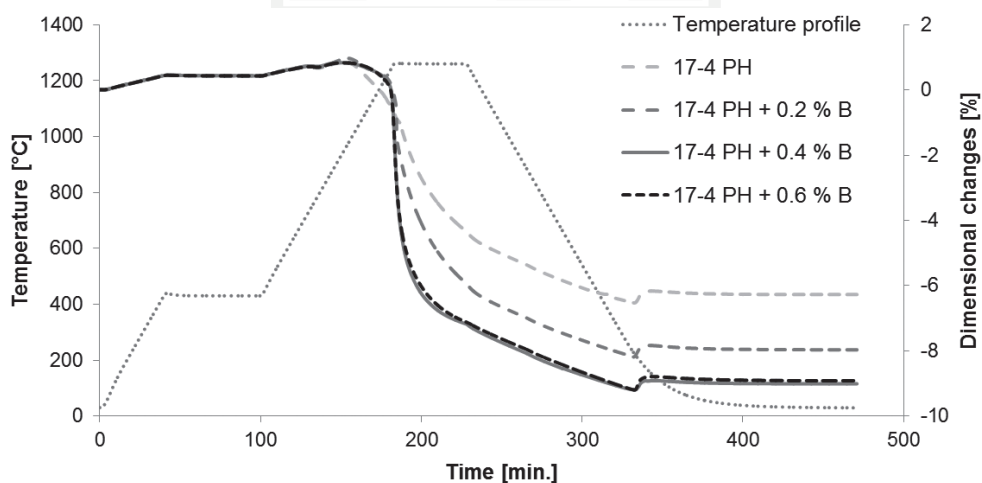


Fig. 8. Dimensional changes during sintering in hydrogen boron modified 17-4 PH steel

The addition of boron to 17-4 PH does not influence dimensional in the range from ambient temperature to about 800°C. However, above this temperature, the presence of boron has an effect on the sintering behaviour of 17-4 PH stainless steel. Specifically, boron slightly raises the transformation temperature, contributes to significant shrinkage during both heating and isothermal sintering compared to boron-free steel. This is directly due to the formation of a boron-rich liquid on the grain boundaries during heating. This liquid improves mass transport phenomena, promotes rearrangement and the fragmentation of particles. The temperature of the creation of a eutectic phase is dependent upon the amount of boron, specifically it ranged of 1160°C up to 1185°C with increasing quantities of boron. The total shrinkage of boron-alloyed 17-4 PH is higher than the total shrinkage of boron-free 17-4 PH. It can be concluded that even a small boron addition (such as 0.2% wt.) is enough to intensify the sintering process of 17-4 PH steel.

Thus, the addition of boron produces a permanent liquid phase that enhances the densification of the compacts during sintering (especially for steel modified by 0.4 and 0.6% wt. addition of boron). This was confirmed in the results of density presented in Fig. 1.

5. Conclusions

17-4 PH stainless steel was modified by boron powder in amounts of 0% wt., 0.2% wt., 0.4% wt. and 0.6% wt. and manufactured using powder metallurgy technology. Boron was added to activate the sintering process.

Experimental results showed that a persistent liquid phase appeared during sintering at 1260°C. There was a formation of borides and an occurrence of eutectic reactions between borides and 17-4 PH stainless steel during sintering. The liquid phase remained as an almost continuous network between solid grains. As a consequence, the classical phenomena of the liquid phase sintering, densification, porosity and microstructure of 17-4 PH steel have been changed. The number of pores reduces with an increasing content of boron. Furthermore, the pore shape is changed from irregular to spherical.

The use of boron has contributed to the successful obtaining of high density sintered stainless steel with improved hardness. However, density and other properties are determined by the amount of boron. It seems that the optimum amount of boron to add to 17-4 PH is 0.6% wt.

It should be underlined that boron has contributed to achieving a reduction of porosity and increase in density, both of which are necessary to improve corrosion resistance of 17-4 PH stainless steel.

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