Examination of boronized surface layers on steel and nickel alloys using glow discharge optical emission spectrometry - recent application of GD OES technique in Instytut Metalurgii Żelaza

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Introduction

The steel surface is subjected to boronizing in order to obtain a high surface hardness of up to 2100 HV, persisting at temperatures as high as 900^{0}C. Boronized layers show high corrosion resistance in many aggressive environments. For nickel alloys having good corrosion resistance, boronizing surface improves protection against wear in a mechanical way. Boronized layers are formed with the use the reactive diffusion mechanism. These layers consist of iron boride: Fe_2B for single-phase structure, whereas for the two phase structure, the FeB phase is located in the outer layer and Fe_2B is located below. The development of boronizing technology requires studies which need analysis of the chemical composition of the layers, including the distribution of boron within the multi-zone of the surface layers as well as distribution of other light elements. The aim of the work was to extend the research capabilities of the Department of Analytical Chemistry Laboratory in the area of the GD OES for depth profile analysis of boronized layers on steel and nickel alloys The developed method has been used as a complementary test method for X-ray microanalysis during the development of the method of formation those layers.

Method

Two separate analytical programs for the GD OES spectrometer LECO 850A for analyzing boronized layers on steel and nickel alloys were used.
For calibration the following excitation conditions of dc source were applied:

- preintegration: U = 1000 V = const, i = 30 mA = const, 40 seconds;
- integration: U = 700 V = const, i = 20 mA = const, 5 seconds (steels)
  U = 1000 V = const, i = 30 mA = const, 5 seconds (nickel alloys)

For calibration of boron the following CRMs and in-house samples were used:

- certified reference materials of steel with boron up to 0,065 % B
- chrome steel castings containing up to 1,43 % B (determined by AES ICP technique, spectrometer Agilent 5100),
- two samples of Fe-B layers, containing 14 and 20 % of B, (semi-quantitative determination by microanalysis XRF technique)
- the set of CRMs of steels, nickel alloys and also some Ti- and Al- alloys.

The following elements were included in the analytical programs B, Fe, C, S, Mn, Si, Cr, Cu, Al, Ni, Mo, V, Ti, Nb, W, Co, W and also Ta, Hf and Zr for nickel alloys.

Test samples

- The series of boronized AMS 5504 steel samples - before (Fig.1) and after heat treatment (Fig. 2) AMS 5504 steel;
- Inconel 718 alloy samples with boronized layers, at the different time of heat treatment (1.5 and 7 hours) (Figs 3 and 4).

The results and conclusions

The GD OES depth profiles for steel samples compared with the results of the EDS analysis are shown in Fig. 1 and 2. Similar comparison for the two In718 alloy samples is presented in Fig. 3 and 4. The results of the analysis with both techniques seem to be not only comparable but also complementary. GD OES depth profiles allow to observe the concentration and distribution of B, Si, Cr, Mn, Ni, Fe, Mo and Ti in both the boronized layers and in the diffusion zone as well. For silicon, two peaks are observed: the first in the diffusion zone and the second - near the surface.
The GD OES technique has been proven to be an excellent analytical tool for the determination of diffusion processes during deposition of protective coatings. It was possible to assess the variation in chemical composition of particular elements in different zones formed on the surface layer of boronized steel and nickel superalloy and to determine the total thickness of boron diffusion inwards the studied materials. The obtained results provided significant insight into the differences between various coating deposition parameters, such as temperature, time and process activity, which determined the chemical composition of the boronized layers. Moreover, the influence of the heat treatment on the depth of boron diffusion inward the surface layer of the studied materials was evaluated.

Literature

1. Hasan Dinc, Amir Motellabzadeh, Huseyin Cimenoglu; Thermochemical boriding of Inconel 718 superallloy, Academic Journal of Science, CD-ROM. ISSN: 2165-6282 : 2(2):385–389 (2013)
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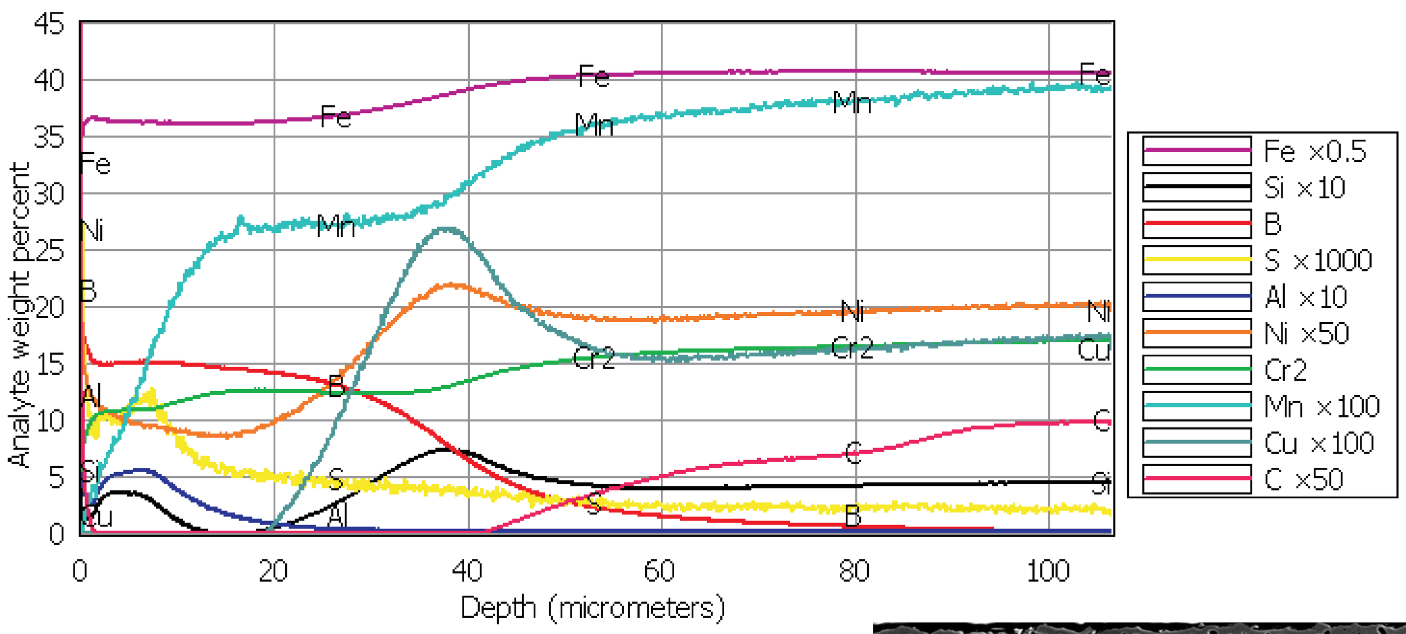
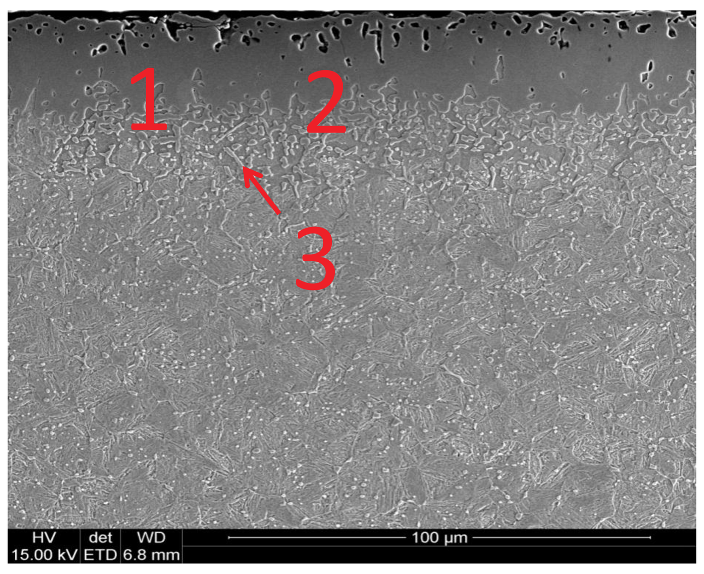


Fig.1. Boronized AMS5504 steel sample –before heat treatment: GD OES depth profile (above) and the results of WDS analysis on the cross-section (right and below)



Point no.	B	Cr	Mn	Fe
	Wt. %			
1	11.1	13.0		75.9
2	11.5	14.4		74.2
3	8.3	45.2	1.3	45.3

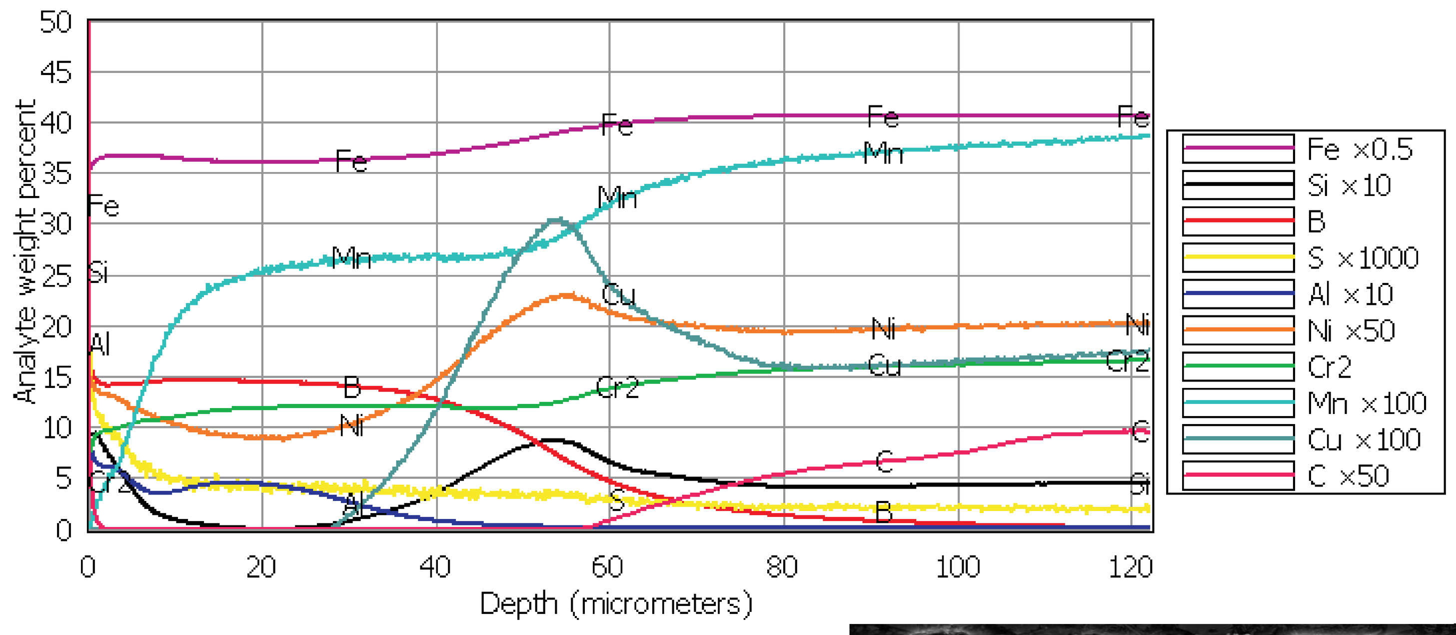
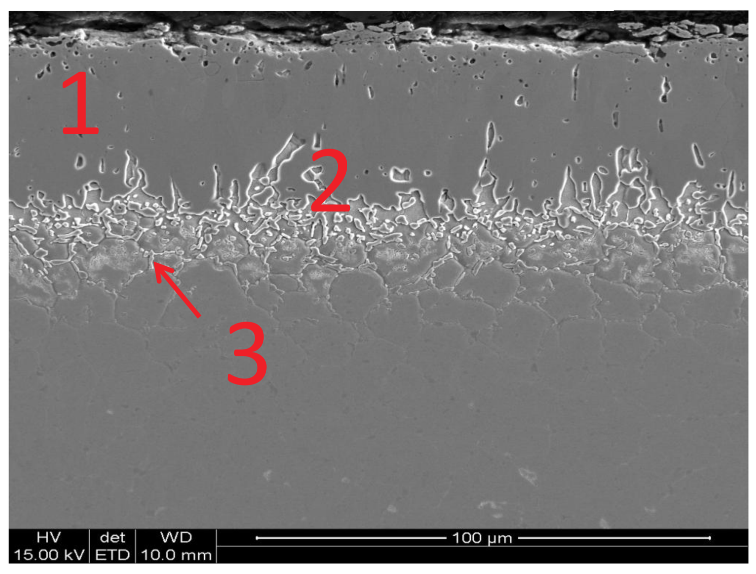


Fig.2 . Boronized AMS5504 steel sample after heat treatment:GD OES depth profile (above) and the results of WDS analysis on the cross-section (right and below)



Point no.	B	Cr	Mn	Fe
	Wt. %			
1	13.8	14	0.5	71.7
2	13.7	13.6	0.4	72.3
3	13.6	26.7	0.4	59.4

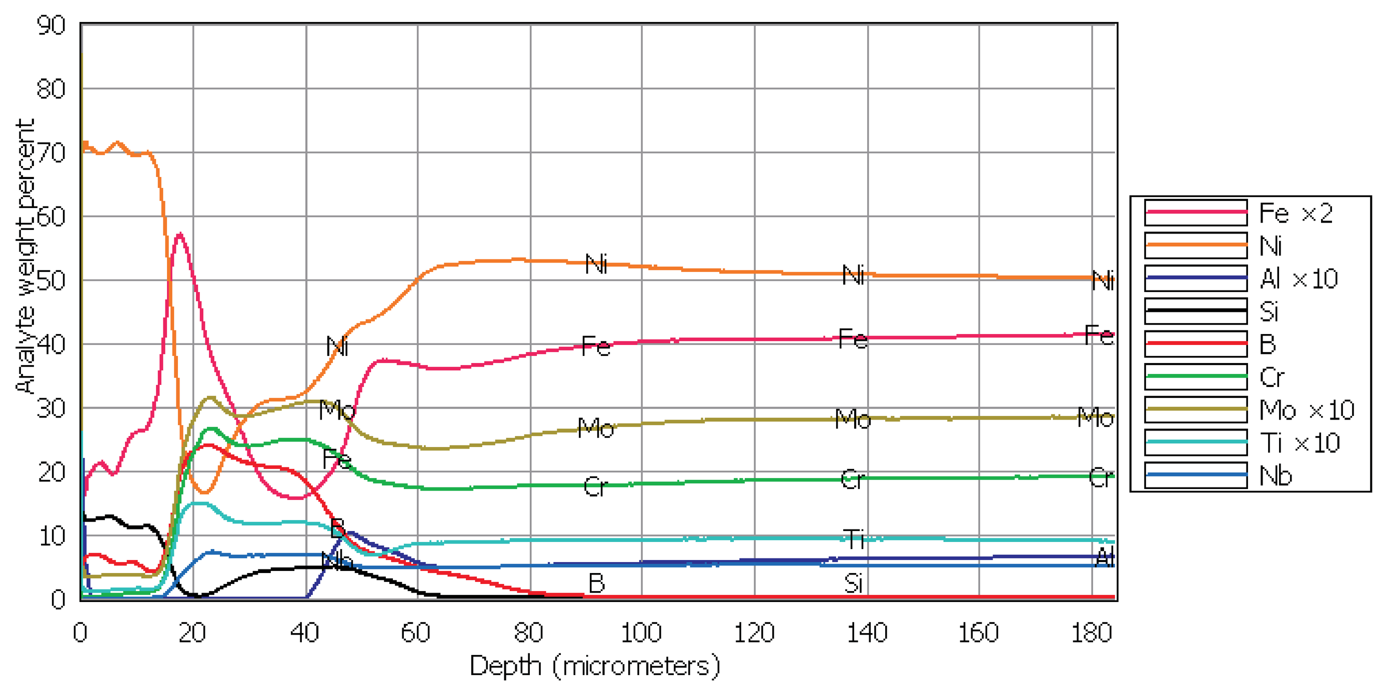
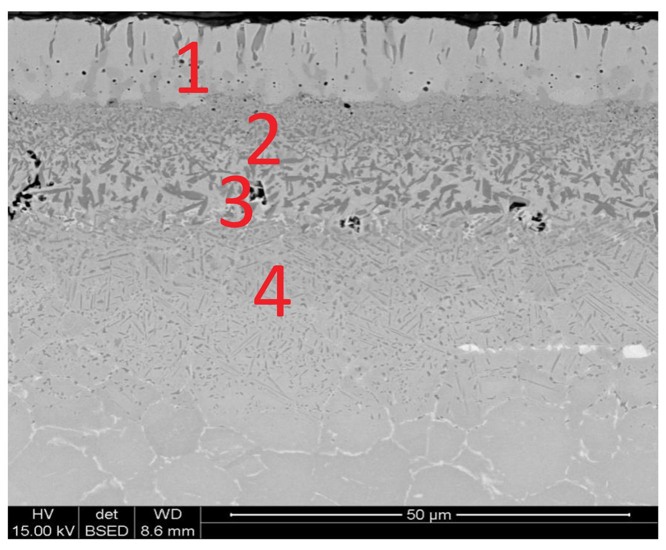


Fig.3 . Boronized In718 sample after 1.5h of heat treatment (950^{0}C): GD OES depth profile (above) and the results of WDS analysis on the cross-section (right and below)



Point no.	B	Si	Fe	Ni	Nb	Mo	Ti	Cr
	Wt. %							
1	3.4	13.4	6.3	76.9				
2	23.4		22.7	15.0	6.7	5.2	0.3	26.7
3	23.0	2.8	8.6	32.6	17.8	3.7	0.6	11.0
4	23.0		6.1	9.4	14.2	8.0		39.3

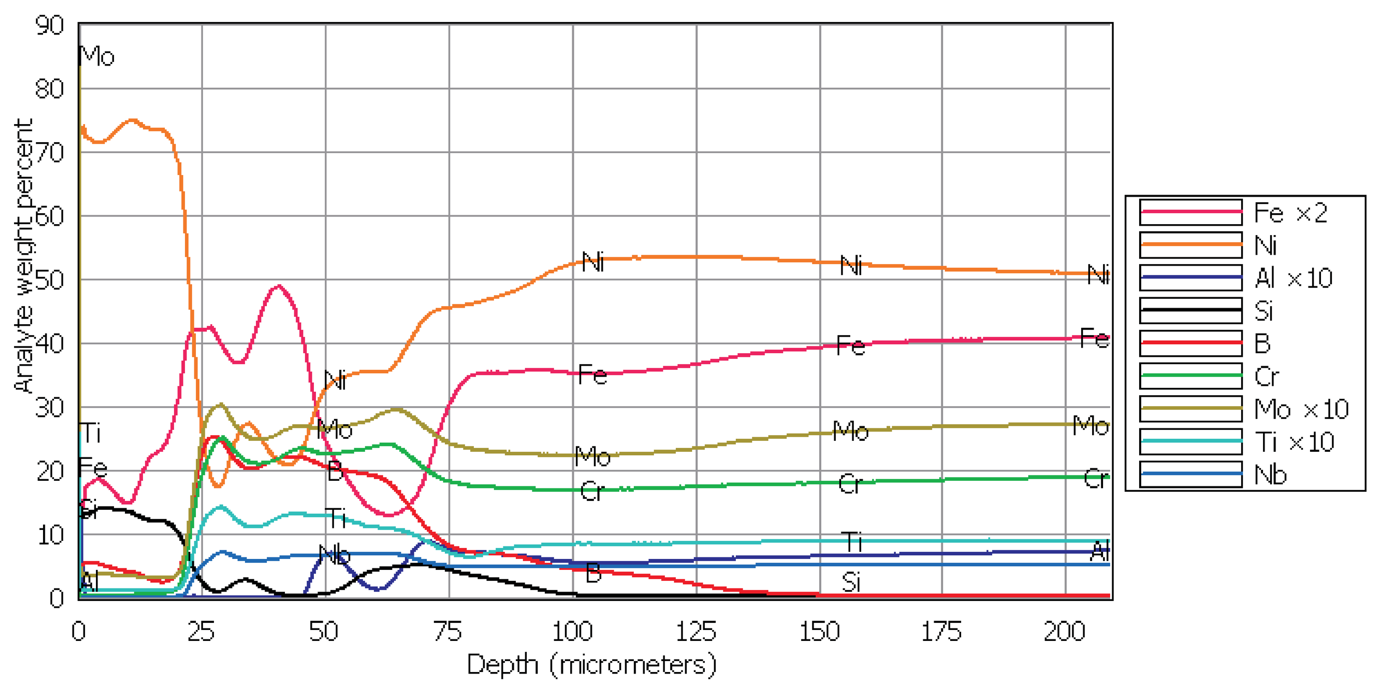
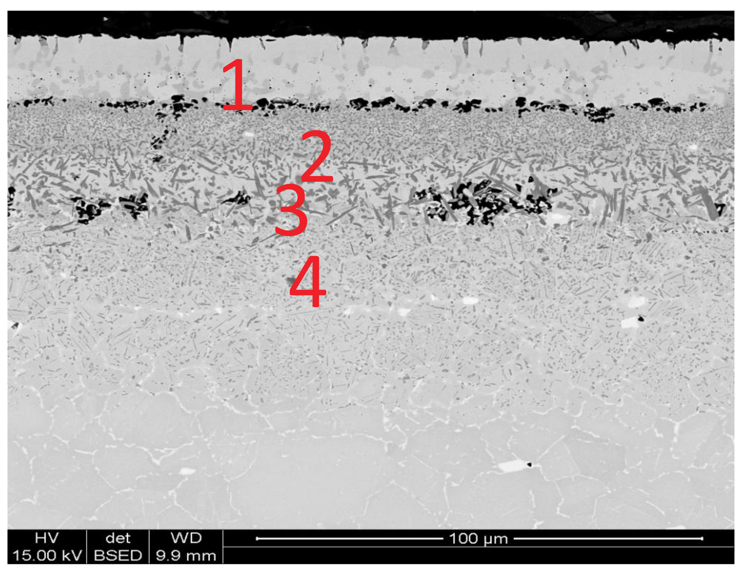


Fig.4 . Boronized In718 sample after 7h of heat treatment (950^{0}C): GD OES depth profile (above) and the results of WDS analysis on the cross-section (right and below)



Point no.	B	Si	Fe	Ni	Nb	Mo	Ti	Cr
	Wt. %							
1	3.0	14.0	4.7	78.3				
2	19.4	0.2	24.2	15.3	8.8	6.0	0.4	25.7
3	20.9	0.2	11.8	12.5	2.0	5.7	0.1	46.9
4	20.7		4.1	4.1	18.0	10.9	0.2	42.1