CHARACTERIZATION TECHNIQUES OF ELECTRODES DEPOSITION MATERIALS FOR IONIZATION CHAMBER DETECTORS

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ABSTRACT

The paper focuses on evaluating the depositions and support materials used in radiation detectors, by microstructure characterization techniques. Boron depositions on aluminium surface and colloidal graphite deposition on polyethylene support were experimentally obtained. Advanced examination techniques, such as Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectrometry (EDS) were used for investigations. Because boron cannot be detected by EDS, its deposition was investigated using backscattered electron signals in SEM. The adherence, compactness, chemical composition and content of impurities are the main characteristics of graphite deposition used in radiation detectors. In order to obtain an accurate detection for all deposition types, the impurities content from the depositions has to be small. As result, the examined boron deposition, which was not dense, presented un-connected, non-adherent crystals with different shapes and sizes. The graphite deposition has proven to be adherent, continuous, compact, but not uniform in terms of thickness.

Key words: radiation detectors, boron, graphite, deposition

Introduction

Development of nuclear power imposed the development and improvement of detection systems and the Instrumentation and Control field had also to be adapted to the new generations of reactors. To improve the effectiveness of charges collection, which is one of the main tasks in designing an ionization chamber detector, theoretical studies were conducted, with the aim to determine the formula of the current collected by an ionization chamber, having a cylindrical-hemispherical geometry [1], [3].

Radiation detectors

The reactor instrumentation can be divided into two categories: systems within the core and systems outside the core [1], [2], [3].

The first category includes small detectors that provide detailed information on the neutron spectrum; they can be placed on a mobile system or at the centre of the core. This type of detectors can provide information continuously or at regular time intervals. From this category, the paper uses detectors having components with depositions in their composition and working in intense neutron fields - the ”Boron Detector" (with boron deposition) and the "Fission chamber detector" (with uranium deposition).

In the second category, where the environmental conditions are less severe, detectors are larger and they answer to the neutron flux properties integrated throughout the core. From this category, the "Ionization chamber detector from intelligent detection assemblies used for surrounding monitoring" (with graphite deposition) is used.
Materials such as aluminium and polyethylene were used as support material for boron and, respectively, for graphite deposition.

Examination of boron deposition on aluminium layer

To examine the boron depositions, both physical phenomena and problems induced by neutron irradiation of boron depositions on different layers need to be taken into account. One of the critical issues for the deposited boron in the case of neutron detectors is the minimization of the amount of impurities which interfere with the neutron detection by spreading the resultant reaction products of the nuclear reaction, produced at the neutron irradiation of boron depositions inside the detector [4], [6].

To make an accurate detection, the deposited boron amount must be maximized and the amount of impurities in the deposition must be as small as possible. Therefore the boron depositions will be examined in order to determine the impurity type chemical elements and their distribution in the deposition. Also, the submitted films must have a good adherence to the layer, although the design characteristics have not set special conditions for the films crystallography [5], [6].

The results consisted in obtaining an experimental colloidal graphite deposition on polyethylene support and in analyzing, together with the support materials, of the boron depositions on aluminium and graphite polyethylene support. Advanced analysis techniques, as Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) were used [5], [6].

Because boron is not detectable by the EDS method, the electron backscatter signal (instead of using secondary electrons) was used for analysis. The chemical composition and the level of impurities from depositions and from support material were determined; an evaluation of the chemical element distributions on the deposition surface, using cartography maps, was performed.

The boron depositions were visually examined to observe the deposition color. The electronic microscopy examination were conducted in order to observe both the deposition and particles adherence and an energy dispersive X-ray spectroscopy was conducted to observe the impurities distribution on the surface with deposition.

Even if EDS analysis cannot reveal the boron, it can be used for observing the locations where boron is located. Because these regions are covered and do not provide a strong signal, boron may be detected in regions where other elements have minimum signal. After an EDS analysis a correlation can be made between the regions with minimal EDS signal and the regions with low gray level from the backscattered electron images.

The backscattered electron (BSE) images can achieve an atomic number contrast, so that elements with small atomic number, such as boron, will produce a very low gray level in the BSE image. Secondary electron images will provide information about adherence, shape and distribution of the crystallites present on the surface with boron deposition.

Figure 1 shows the aluminum cylinder used in the neutron detector. On the inner surface, the matted, dull, gray color boron deposition, can be observed.

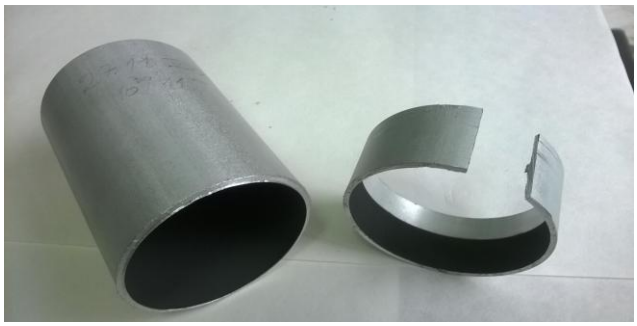

Fig. 1. *Boron deposition on a cylindrical aluminium support [6]*

Figure 2 presents a topographic SEM image of the deposition surface, showing that the deposition is not dense. The un-connected crystals can be observed, and the depostion consists of crystals which are probably non-adherent, with different shapes and sizes. This suggests that these crystals are composed by different chemical elements, which wouldmean a high impurities content.

Therefore, the visual field in Figure 3 was examined by X-ray spectroscopy, to determine their possible chemical nature. Since boron cannot be detected, all others chemical elements presented in the investigated field were identified.

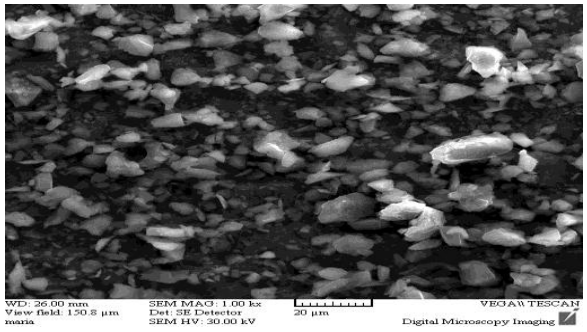

Fig.2. *Topographic SEM image showing the boron deposition surface [6]*

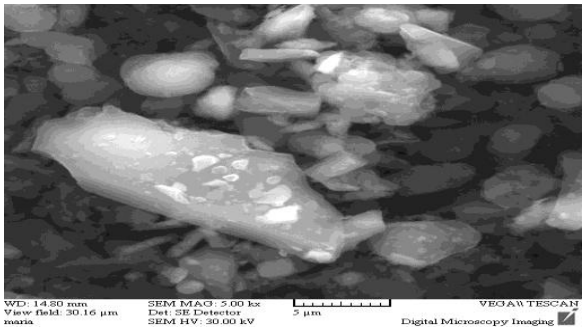

Fig. 3. *SEM field of view from the boron deposition surface [6]*

Figure 4 shows the EDS spectrum obtained after the field investigation illustrated in Figure 3.

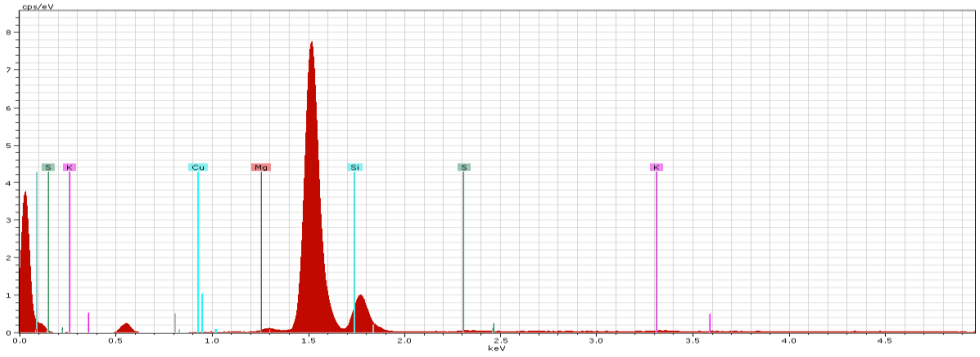

Fig. 4*. EDS spectrum of the field illustrated in Figure 3 [6]*

Table 1 shows the chemical elements which were present on the mentioned surface, except boron.

Table 1. *Impurities present in the deposition [6]*

No.	Chemical element	Atomic no.	Spectral line	Relative quant. (% mass)
1	Aluminium	13	K series	54.8
2	Silicium	14	K series	17.7
3	Copper	29	K series	0.4
4	Sulphur	16	K series	0.3
5	Magnezium	12	K series	0.1
6	Oxygen	8	K series	26.7

As seen from the spectrum in Figure 4 and Table 1, the following chemical elements were present on the surface: Aluminium, Oxygen, Silicon, Copper. The presence of aluminum in high volume shows that not all surface is covered with boron deposition, and there are regions not covered by the deposition. Aluminium can also occur from the sub-layer in the EDS analysis. The analysis shows that boron was not identified on certain areas, and silicon, in percentage of 17.7 %, is in the form of impurity deposit. Therefore, a mapping of the chemical elements present on the surface was performed.

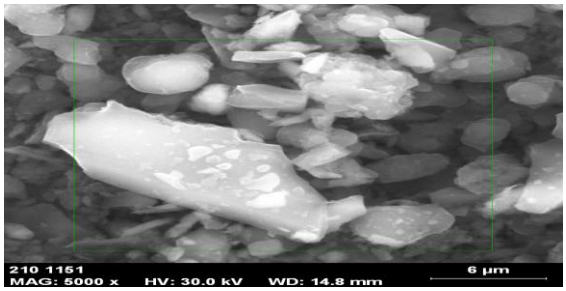

Fig. 5. *SEM topographic image from the boron deposition surface [6]*

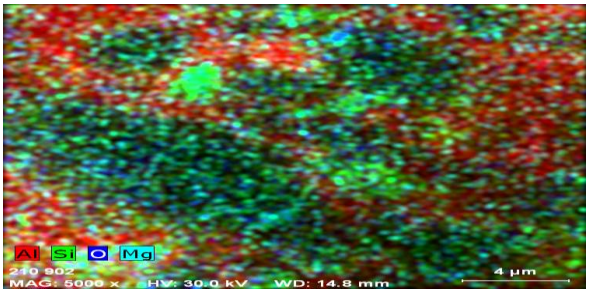

Fig. 6. *Global EDS mapping for Al, Si, O and Mg [6]*

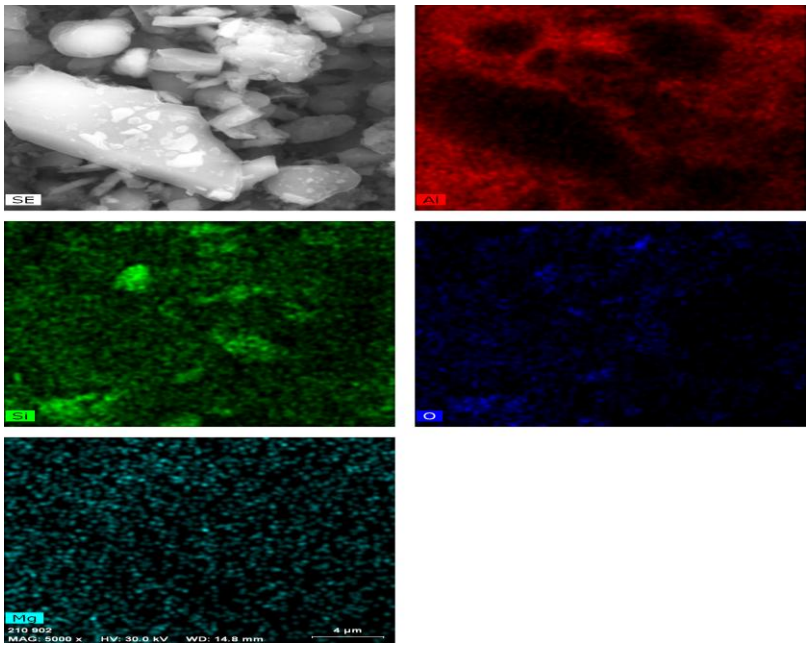

Fig.7. *EDS mapping for the following chemical elements: Al, Si, O and Mg [6]*

Figures 5, 6 and 7 show: secondary electron image of the investigated field, global EDS mapping for Al, Si, O and Mg, respectively, the mapping of these chemical elements.

Examination of graphite deposition on nonconductive polyethylene layer

The important characteristics of the boron depositions are the following: adherence, compactness, chemical composition and impurities content. In order to obtain an accurate detection, the impurities level for all depositions must be as small as possible. Therefore, the deposited graphite will be examined to determine the impurity type chemical elements of and their distribution in the deposition.[5], [6].

Figure 8 shows the graphite deposition on polyethylene layer. The polyethylene tubing was cut into two components on which the adherent, black, uniform color graphite deposition can be observed. The film adherence and compactness will be highlighted by electron microscopy.

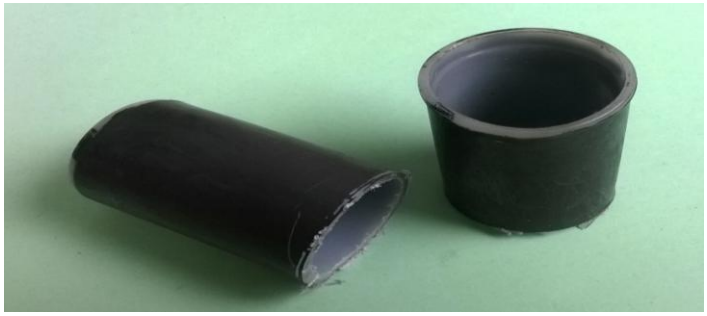

Fig. 8. *Graphite deposition on polyethylene layer [6]*

Figure 9 shows the secondary electron image, at a relatively low magnification rate (x200), of the cylinder outer surface with graphite deposition. Note that on surface are present ditches in two parallel lines, which were originally produced during the manufacture of polyethylene support. The fact that they are visible shows that the deposition is not very thick and the ditches can help at a better adherence of the deposited graphite film.

Figure 10 shows the image of surface with high magnification rate: regions where the deposition thickness is high can be seen and rarely in some regions pores may be present in the deposition, in the initial ditches on the polyethylene. Still, they do not have any effect on the detection, due to their small number and size. High magnification images confirm that the layer is adherent, continuous and compact.

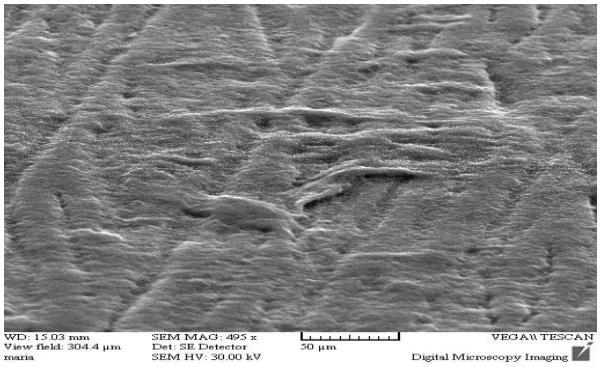

Fig.9. *Details from the graphite depostion [6]*

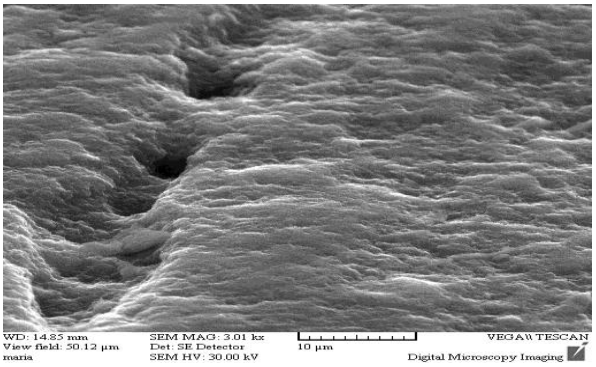

Fig. 10. *Pores from the graphite deposition [6]*

The image in Figure 11 shows the graphite layer thickness measurement, the measured value being 50.73 microns. The deposition was measured in four different points, and the measured thickness value had a variability between 8 and 100 microns (8.6µm, 10.56µm, 71.75µm, 50.73µm).

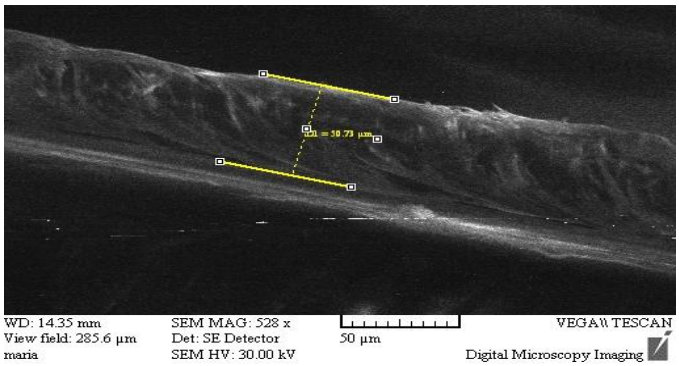

Fig. 11. *Measured thickness of the graphite layer [6]*

After SEM examination, the chemical composition of the deposition was determined and a mapping of the present chemical elements was made, the aim being to find out the nature and distribution of impurities in the graphite deposition.

Figure 12 shows the EDS spectrum obtained after investigating the analysis field,with a magnification rate of 500.

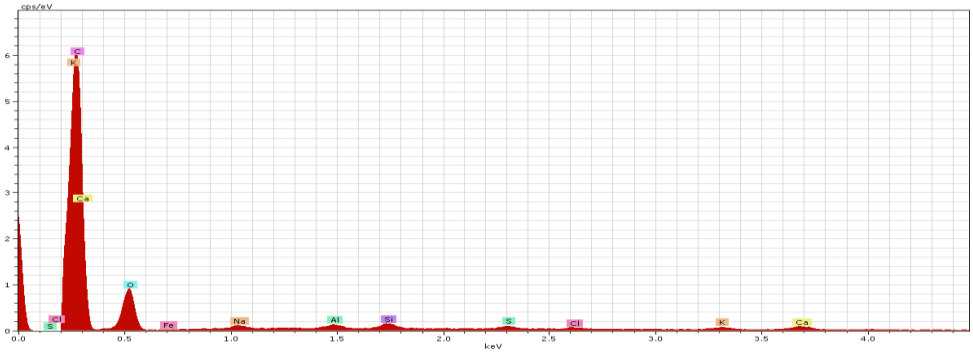

Fig. 12. *EDS spectrum obtained by investigating the analysis field [6]*

Table 2 shows the chemical elements which were present on the scanned surface.

Table 2. *The impurity type chemical elements present on the deposition [6]*

No.	Chemical element	Atomic no.	Spectral line	Rel. quantity (% mass)
1	Carbon	6	K series	25.1
2	Calcium	20	K series	0.3
3	Aluminium	13	K series	0.3
4	Silicium	14	K series	0.3
5	Iron	26	K series	0.2
6	Sulphur	16	K series	0.2
7	Sodium	12	K series	0.4
8	Oxygen	8	K series	73.2

Figure 13 shows the SEM image of the marked area and the mapping for the following chemical elements: C, Si, O, Ca, Na and Al.

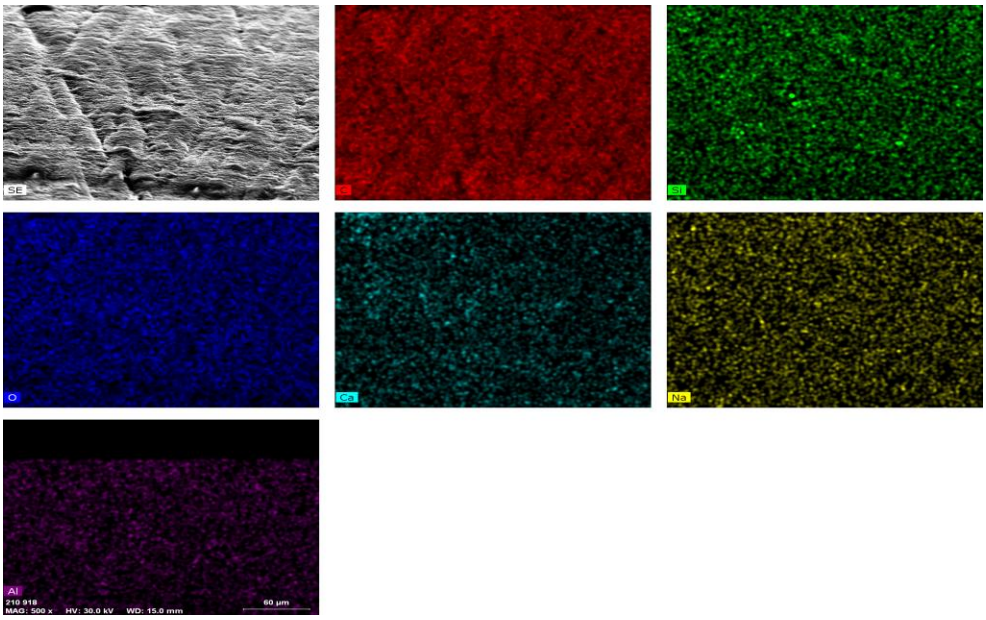

Fig. 13. *Chemical elements mappings [6]*

From the two types of mappings, it can be seen that the chemical elements presented as impurities in small amounts are evenly distributed in the deposition and very likely they do not have a significant influence on the detection properties of the detector.

5. Conclusions

The paper has made an assessment of the capability to use potential microstructural characterization techniques for the materials or depositions on various support materials which are present in the composition of radiation detectors. These techniques have shown a good caracterization of materials or depositions specific to radiation detectors.

The requirements of radiation detectors manufacturers specify that the support materials must have a high level of purity and their impurities must present a small neutron capture section, in order to maximize the Signal to Noise Ratio.

- ❖ It was found that boron deposition is not dense, is formed of non-adherent crystals which are not interconnected, having different shapes and sizes.
- ❖ The deposition contains large amounts of impurities of Silicon, Copper, Magnesium.
- ❖ Although the polyethylene could not be examined by electron microscopy because it is not conductive, there were no examination problems for the graphite deposition on polyethylene support.
- ❖ The graphite deposition was found to be adherent, continuous and compact, but with variable thickness.
- ❖ Very few pores were detected in the initial polyethylene ditches; they do not have any effect on the detection due to their small numbers and sizes.
- ❖ The deposition has a very small amount of impurities: Calcium, Aluminium, Silicon, Iron and Sodium.

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