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MODERN METHODS OF RESEARCH THROUGH OPTICAL AND ELECTRONIC MICROSCOPY OF SPECIAL METALIC MATERIALS STRUCTURE

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Abstract: *In this paper we present the results of experimental research performed using a Philips XL 30 ESEM TMP scanning electronic microscope, owned by UPB-CEMS, on a number of 6 samples taken from an iron-nickel-chromium special material, used for making high voltage overhead power lines, which were disc-shaped, with a diameter of 7mm and a height of 0,5 – 1,2 mm, electrically eroded.[1],[2]. Inside a vacuum environment, using a 30 kV voltage for accelerating the electron beam, a spot dimension on the sample surface equal to 3 and a distance between the polar piece of the microscope and the surface of the sample of 10 mm, there have been obtained images of secondary electrons, which have helped morphologically characterize all the samples, at magnifications between 100x and 25.000x.*

Keywords: *electronic microscope, vacuum environment, secondary electrons, morphologically characterize*

INTRODUCTION

Results of the compositional analyzes have been obtained using a energy dispersion EDAX – Sapphire spectrometer, at acceleration voltages of 30 kV, dimension of electron beam spot equal to 5,5, distance between the polar piece and the surface of the sample of 10 mm, at an angle of 35° between the sample surface and the X-ray detector [3]. Compositional analyzes have been achieved at magnifications of 100x, on five fields, the results presented in this report being an average of the individual quantitative results. It must be taken into account during data interpretation that the microanalysis is a compositional characterization method of micro volumes, determining chemical composition of nonhomogeneous substance volumes requires performing atomic spectrometric or mass determinations.[4].

METHODOLOGY

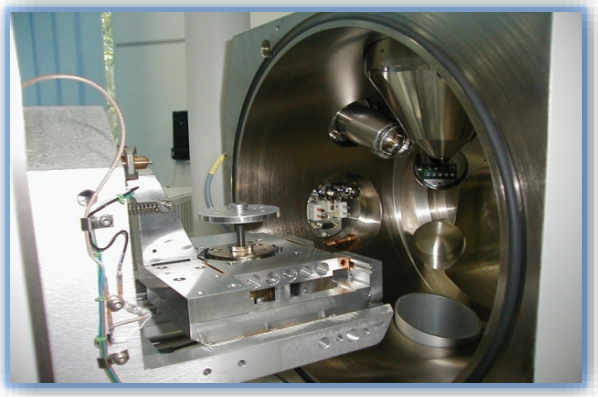
Within this study six samples (Sample 1 – Sample 6) have been examined through SEM / EDS method using a XL 30 ESEM (3,5 nm resolution) electronic scanning microscope, coupled with an energy dispersion EDAX Sapphire spectrometer (128eV resolution). (Figure 1a and 1b).

Table 1. Quantitative compositional results corresponding to sample 1.

Element	Wt %	At %
AlK	1.49	3.18
SiK	1.81	3.72
MoL	8.14	4.89
CaK	0.52	0.75
NdL	4.23	1.69
CrK	3.75	4.16
MnK	2.05	2.15
FeK	55.39	57.21
NiK	22.64	22.24
Total	100.000	100.000

Results are objectified by using morpho-compositional images (secondary and re-dispersed electrons) and EDS spectra with adjacent quantitative results.

a)



b)

Figure 1. EDAX Sapphire spectrometer

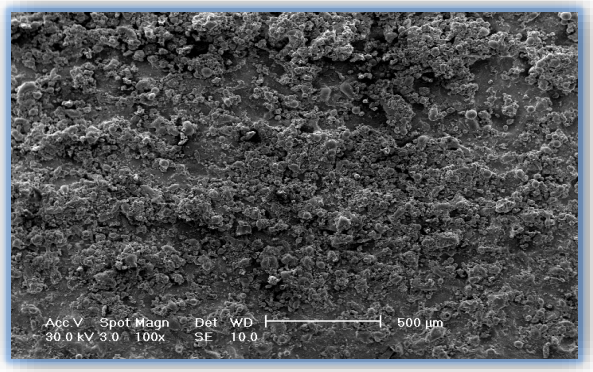


Figure 1.1 Secondary electrons image – surface morphology at 100x magnification.

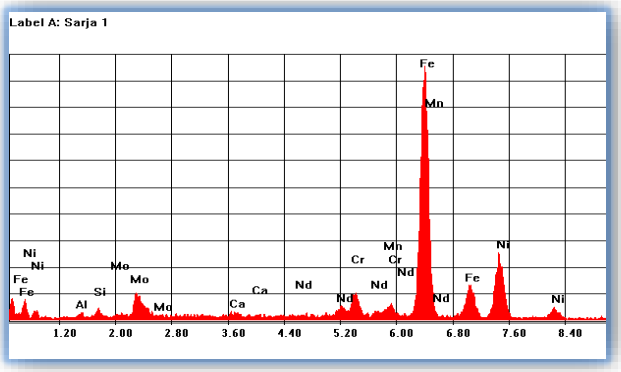


Figure 1.5. Characteristic X-ray emission specter, corresponding to sample 1 compositional analysis.

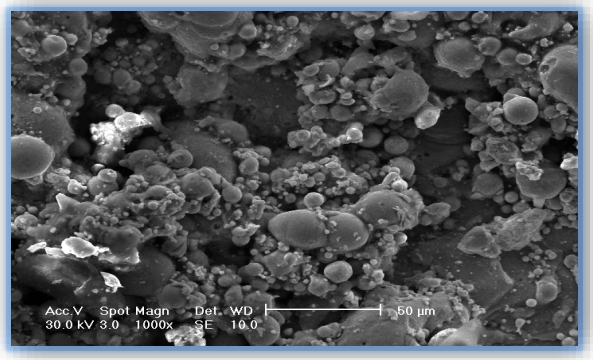


Figure 1.2. Secondary electrons image – surface morphology at 1.000x magnification.

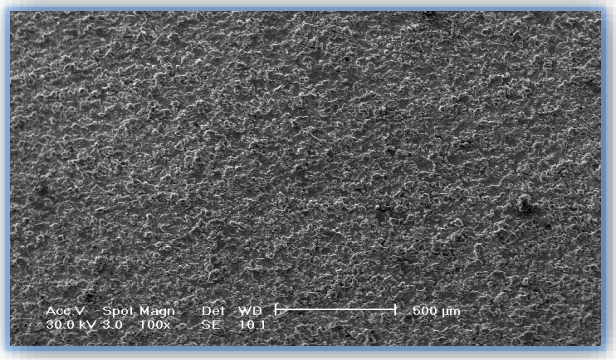


Figure 2.1. Secondary electrons image – surface morphology at 100x magnification.

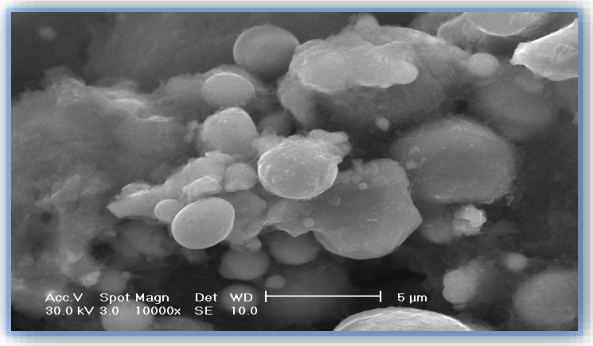


Figure 1.3. Secondary electrons image – surface morphology at 10.000x magnification.

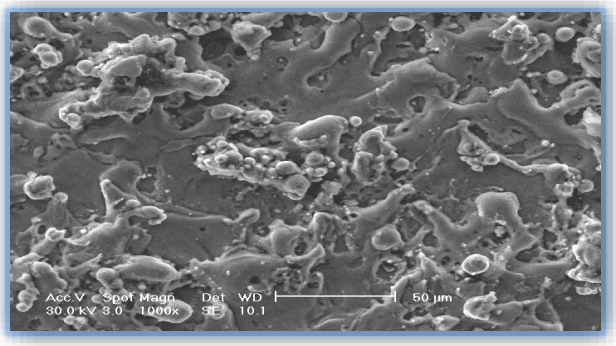


Figure 2.2. Secondary electrons image – surface morphology at 1.000x magnification.

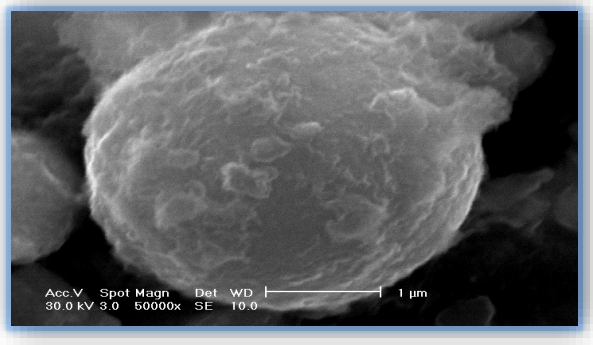


Figure 1.4. Secondary electrons image – surface morphology at 50.000x magnification.

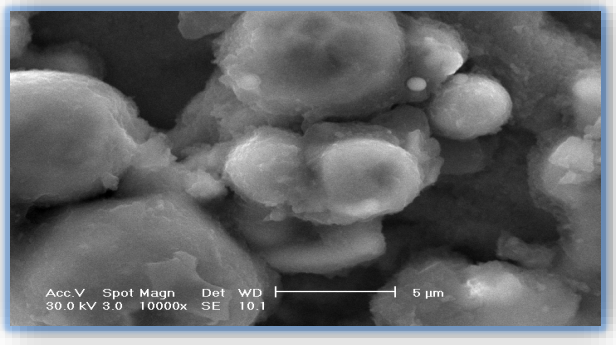


Figure 2.3. Secondary electrons image – surface morphology at 10.000x magnification.

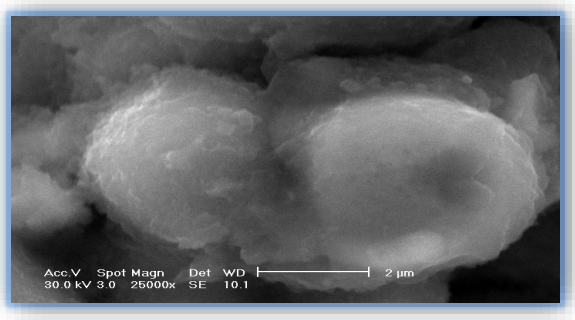


Figure 2.4. Secondary electrons image – surface morphology at 50.000x magnification.

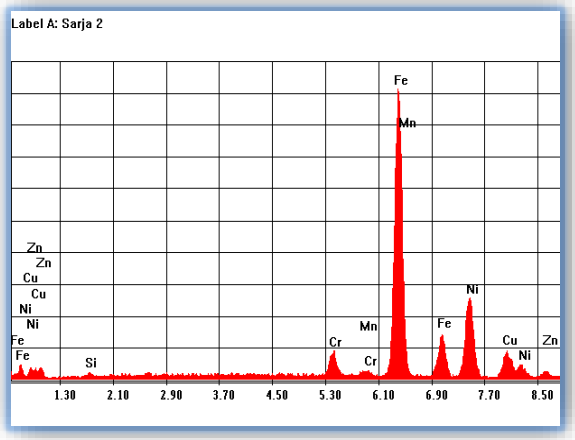


Figure 2.5. Characteristic X-ray emission specter, corresponding to sample 2 compositional analysis.

Table 2. Quantitative compositional results corresponding to sample 2.

Element	Wt %	At %
SiK	1.02	2.07
CrK	3.31	3.61
MnK	0.68	0.70
FeK	55.02	55.97
NiK	27.00	26.12
CuK	10.20	9.12
ZnK	2.77	2.40
Total	100.000	100.000

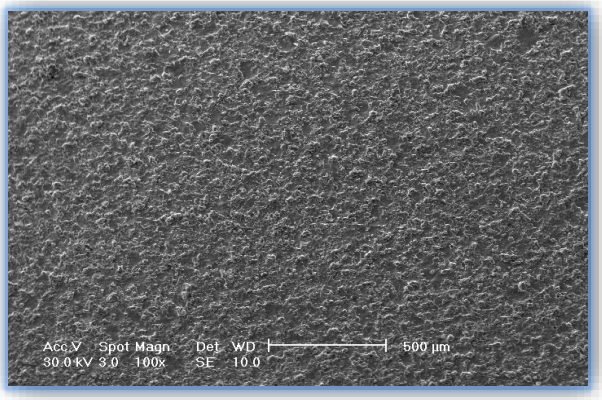


Figure 3.1. Secondary electrons image – surface morphology at 100x magnification.

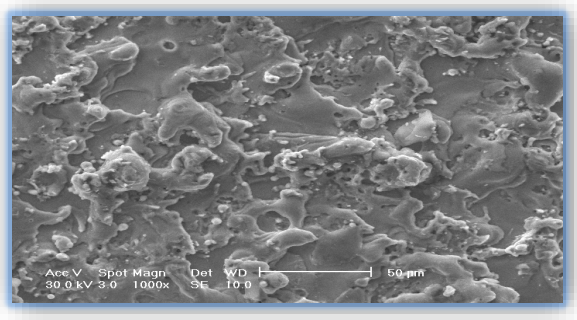


Figure 3.2. Secondary electrons image – surface morphology at 1.000x magnification.

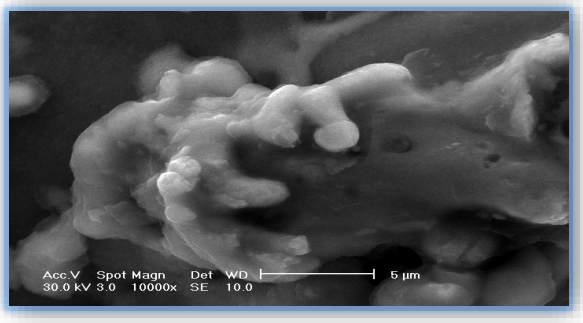


Figure 3.3. Secondary electrons image – surface morphology at 10.000x magnification.

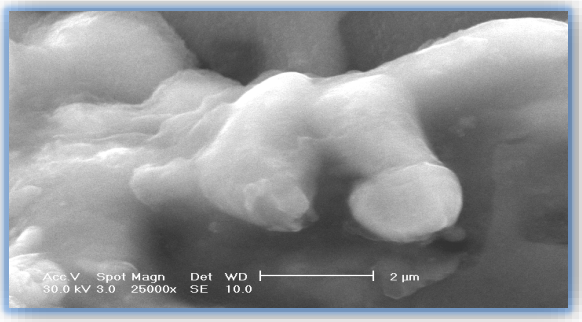


Figure 3.4. Secondary electrons image – surface morphology at 25.000x magnification.

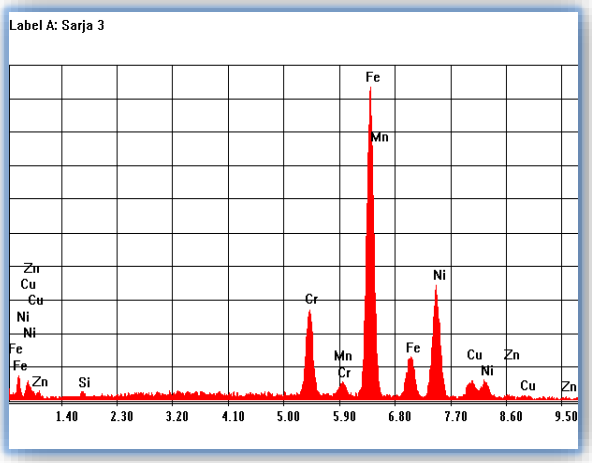


Figure 3.5. Characteristic X-ray emission specter, corresponding to sample 3 compositional analysis.

Table 3. Quantitative compositional results corresponding to sample 3.

Element	Wt %	At %
SiK	1.05	2.09
CrK	10.51	11.35
MnK	0.51	0.52
FeK	50.78	51.07
NiK	29.68	28.39
CuK	5.88	5.20
ZnK	1.59	1.37
Total	100.000	100.000

Table 4. Quantitative compositional results corresponding to sample 4.

Element	Wt %	At %
SiK	1.14	2.28
CrK	10.67	11.53
MnK	3.10	3.17
FeK	47.42	47.70
NiK	28.71	27.47
CuK	6.48	5.73
ZnK	2.47	2.12
Total	100.000	100.000

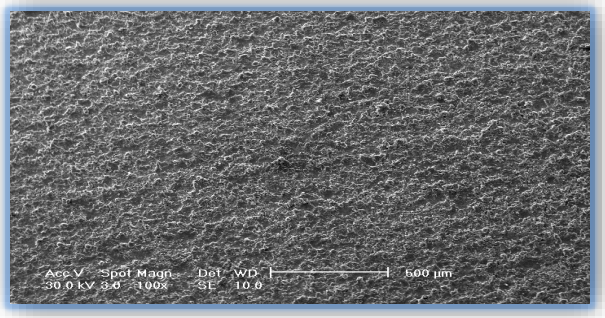


Figure 4.1. Secondary electrons image – surface morphology at 100x magnification.

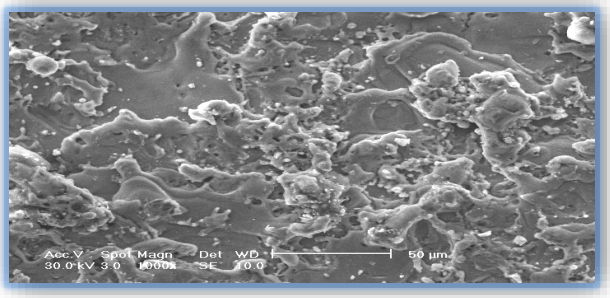


Figure 4.2. Secondary electrons image – surface morphology at 1.000x magnification.

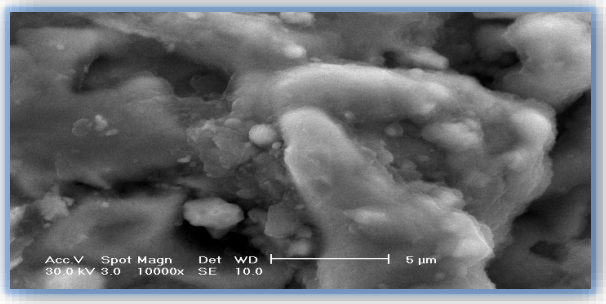


Figure 4.3. Secondary electrons image – surface morphology at 10.000x magnification.

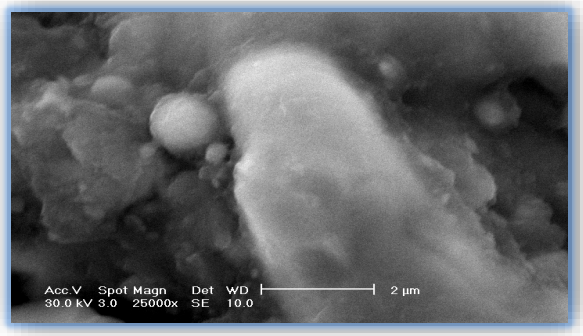


Figure 4.4. Secondary electrons image – surface morphology at 25.000x magnification.

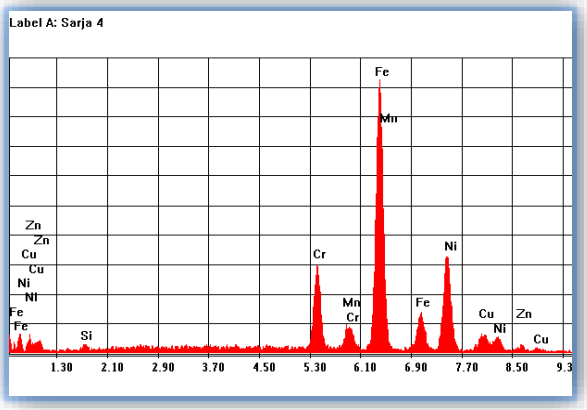


Figure 4.5. Characteristic X-ray emission specter, corresponding to sample 4 compositional analysis.

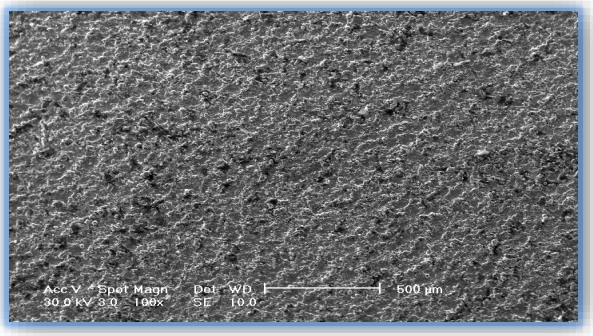


Figure 5.1. Secondary electrons image – surface morphology at 100x magnification.

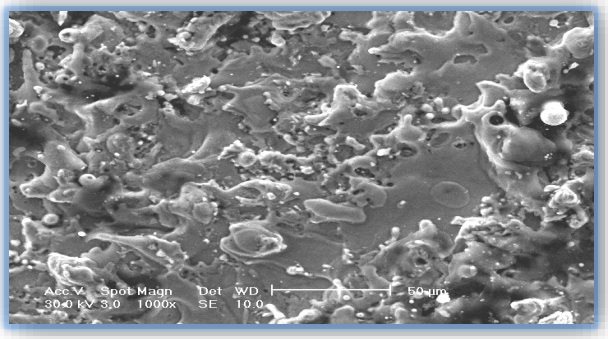


Figure 5.2. Secondary electrons image – surface morphology at 1.000x magnification.

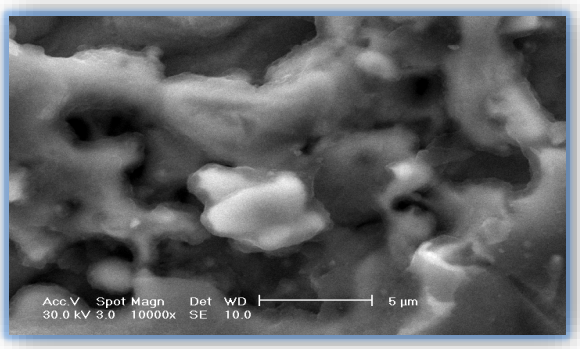


Figure 5.3. Secondary electrons image – surface morphology at 10.000x magnification.

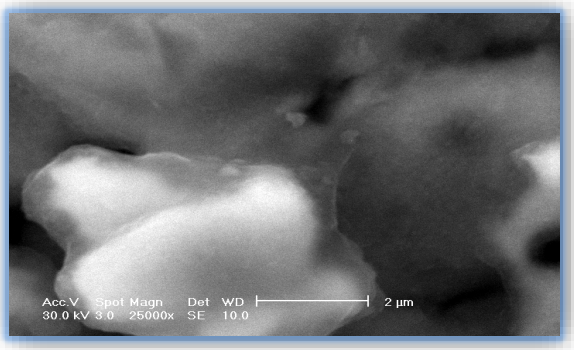


Figure 5.4. Secondary electrons image – surface morphology at 25.000x magnification.

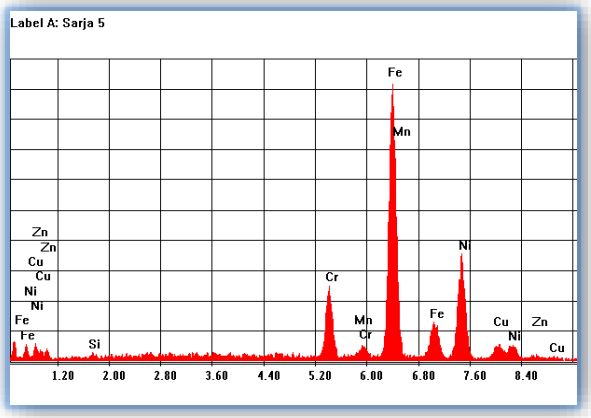


Figure 5.5. Characteristic X-ray emission specter, corresponding to sample 5 compositional analysis.

Table 5. Quantitative compositional results corresponding to sample 5.

Element	Wt %	At %
SiK	0.58	1.17
CrK	9.42	10.24
MnK	0.92	0.94
FeK	50.68	51.29
NiK	30.63	29.49
CuK	5.91	5.25
ZnK	1.86	1.61
Total	100.000	100.000

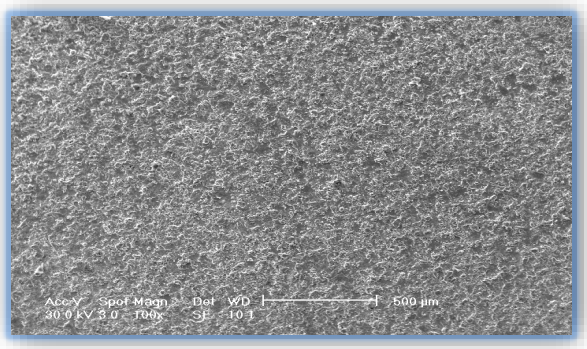


Figure 6.1. Secondary electrons image – surface morphology at 100x magnification.

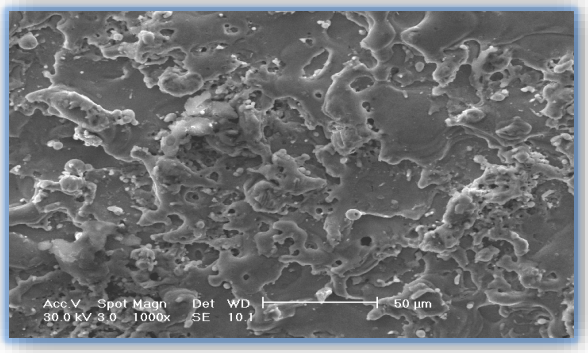


Figure 6.2. Secondary electrons image – surface morphology at 1.000x magnification.

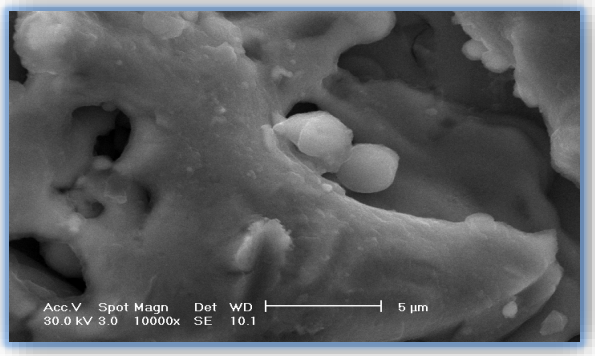


Figure 6.3. Secondary electrons image – surface morphology at 10.000x magnification.

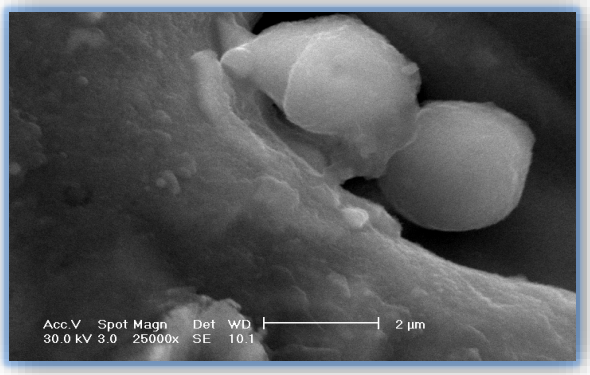


Figure 6.4. Secondary electrons image – surface morphology at 25.000x magnification.

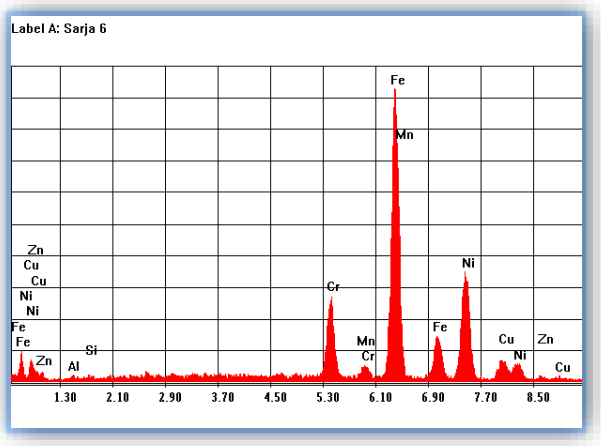


Figure 6.5. Characteristic X-ray emission specter, corresponding to sample 6 compositional analysis.

Table 6. Quantitative compositional results corresponding to sample 6.

Element	Wt %	At %
AlK	0.91	1.88
SiK	0.65	1.29
CrK	9.68	10.42
MnK	0.52	0.53
FeK	49.57	49.66
NiK	30.55	29.11
CuK	6.69	5.89
ZnK	1.43	1.22
Total	100.000	100.000

CONCLUSIONS

This report highlights the possibilities offered by this investigation method and allows drawing important conclusions over the structure of the investigated materials from analyzing the results.

Examining the samples using this method allows collecting information over:

- *The morphology of analyzed surfaces – characteristics of object surface or, also known as, details regarding their textures, the link between material characteristics and properties (ductility, resistance, reactivity, etc.);*
- *Chemical composition of the samples – data regarding elements and compounds they are made of, but also quantitative ratio*
- *Crystalline structure – atom distribution in the crystal; the direct correlation between atom arrangement in the crystal network and material properties (conductivity, electric properties, resistance, etc.).*

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