Study on powdered samples and particle size determinations by scanning eletronic microscopy (SEM), diffraction of X-rays

Cristiana – Zizi Rizescu, Elena Valentina Stoian, Dan Nicolae Ungureanu, Zorica Bacinschi, Cristi Petre Fluieraru

Abstract—Study has been analyzed qualitatively and particle size determinations powders from blast furnace by scanning eletronic microscopy (sem**),** diffraction **of** x-rays
X-ray spectrum shows that the sample contains the following elements: Fe, Ca, Mn, Ti, K, S, Al, Si and Mg. It is mentioned that using EDX spectrometer cannot detect light elements (*e.g.* oxygen).
Sample morphology is revealed by scanning electron microscopy images, secondary electron images (SEI).
To investigate the scanning electron microscope, small amounts of samples were spread on a foil tape, conductive, carbon.
For a qualitative analysis by X-ray diffraction, a monochromatized CuK_{α} X-ray beam with a Ni filter have been used.
The particle size determinations were made by means of ANALYSETTE FRITSCH particle size analyzer**.**

Keywords— Energy Dispersive X-ray Spectrometer (EDX), particle size determinations, qualitative analysis, secondary electron images (SEI), X-ray spectrum

I. INTRODUCTION

IT has been analyzed qualitatively and particle size determinations powders from blast furnace.

II. EXPERIMENTAL TECHNIQUE

Study of powdered samples was performed with scanning electron microscope HITACHI S-2600N model equipped with Energy Dispersive X-ray Spectrometer (EDX) and X-ray diffractometer Shimadzu type (2θ Bragg-Brentano geometry).
To investigate the scanning electron microscope, small amounts of samples were spread on a foil tape, conductive, carbon.
For a qualitative analysis by X-ray diffraction, a monochromatized CuK_{α} X-ray beam with a Ni filter have been used.
The particle size determinations were made by means of ANALYSETTE FRITSCH particle size analyzer.[1,6]

III. RESULTS OBTAINED

SAMPLE P1

Sample 1 morphology is revealed by scanning electron microscopy images, secondary electron images (SEI) from Figure 1, 3 and 5.
The sample consists of polyhedral shaped particles with a wide distribution by size but also submicron particles slightly rounded.

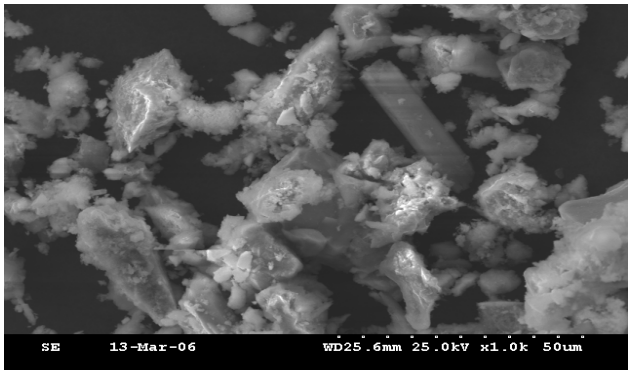
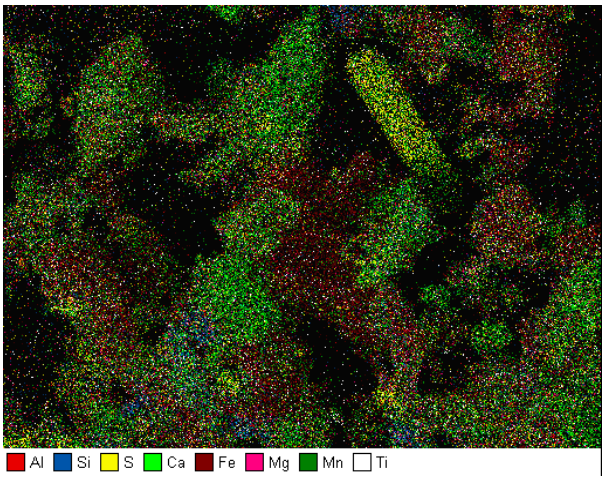
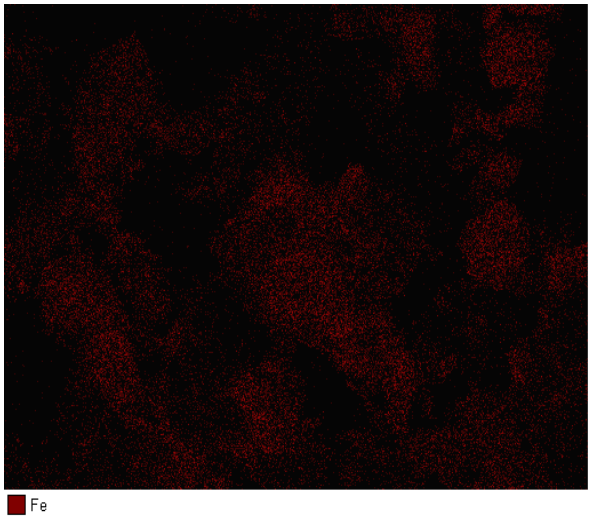


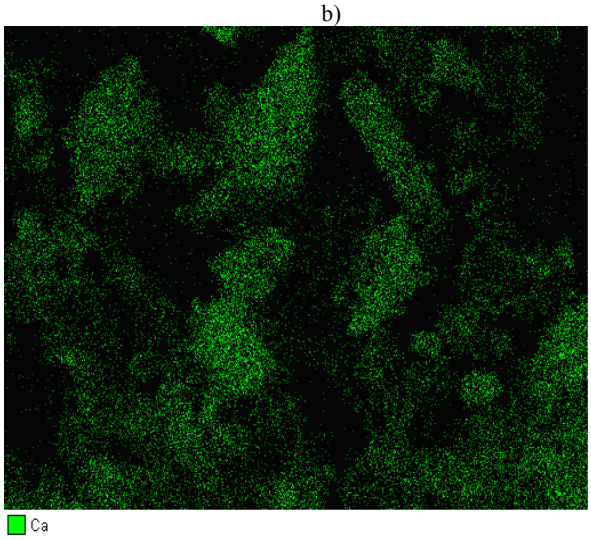
Figure 1 Scanning electron microscopy images (SEI) - sample P1



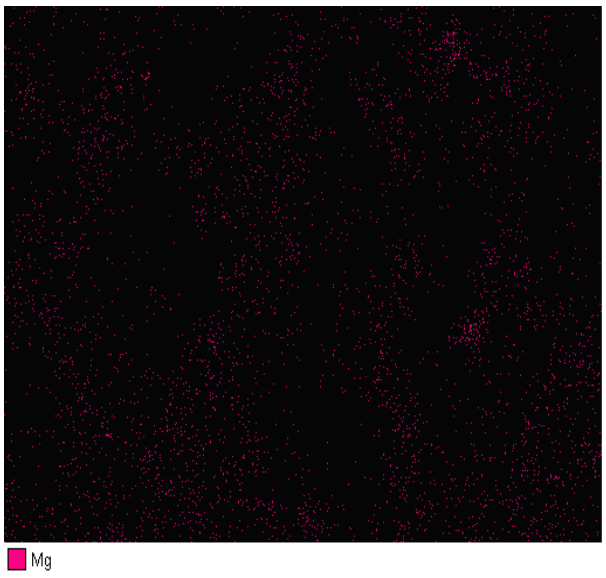
a)



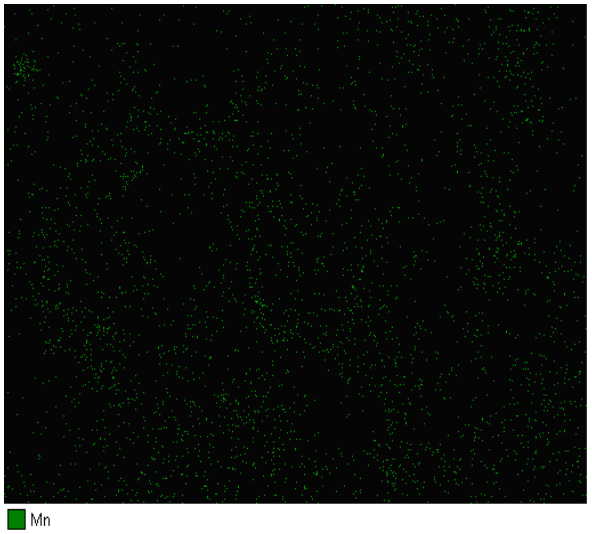
b)



c)



d)



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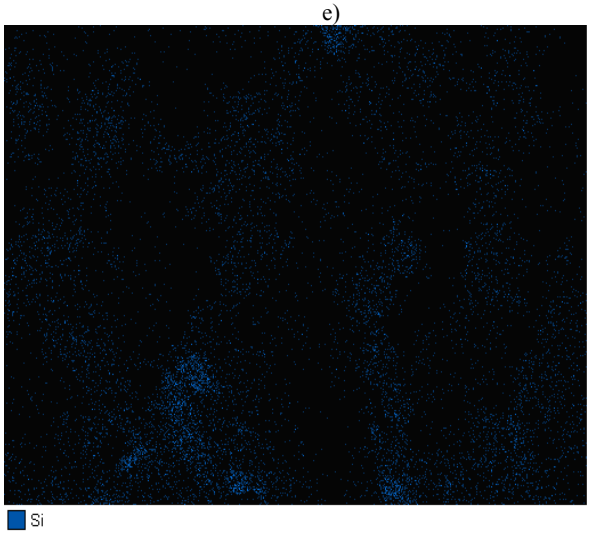

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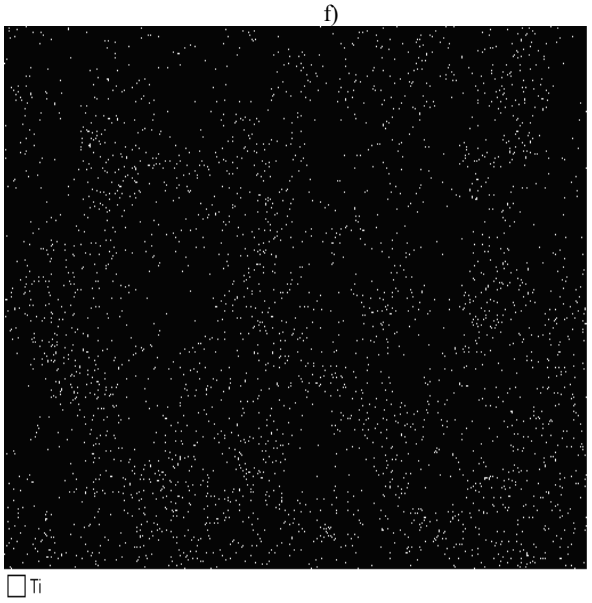
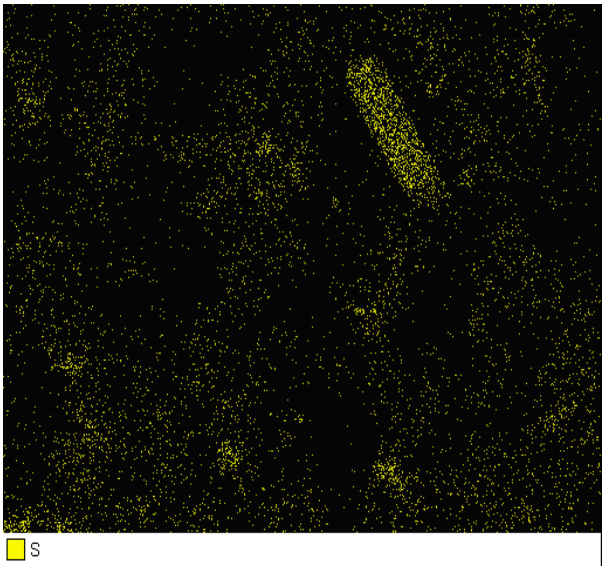


g)



h)

Figure 2 X-ray distribution images suggesting the location of elements Fe, Ca, Mg, Mn, Si, Ti, S in microzone in Figure1

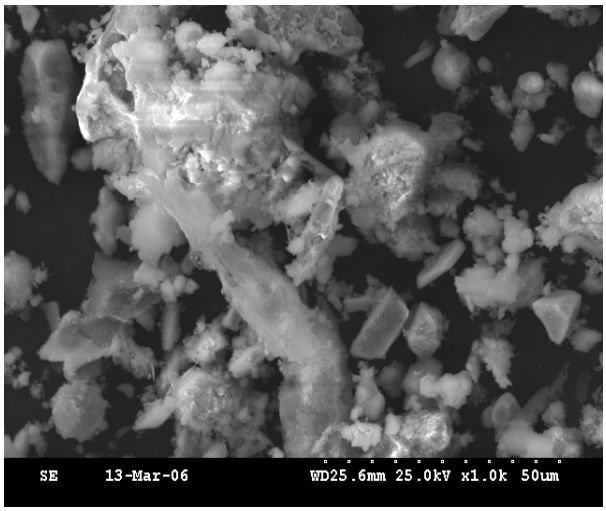
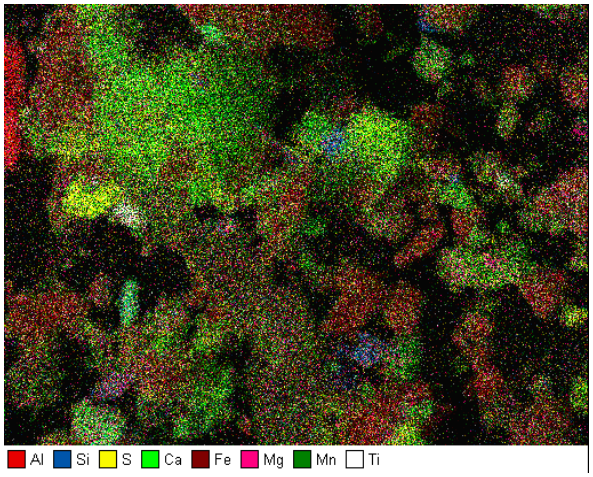
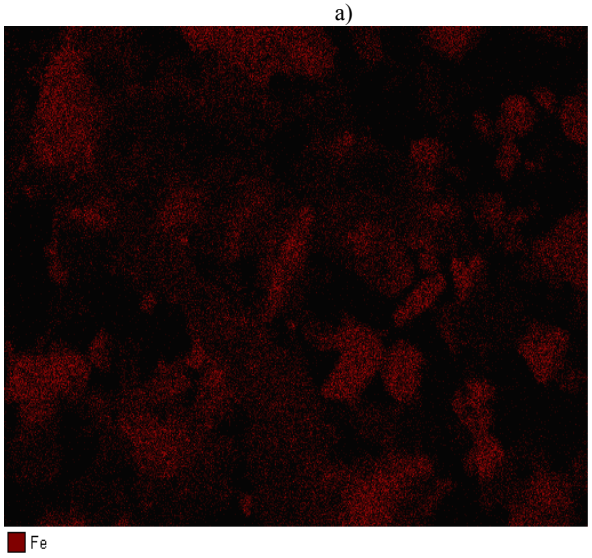


Figure 3 Scanning electron microscopy images (SEI), another microzone – sample P1



a)



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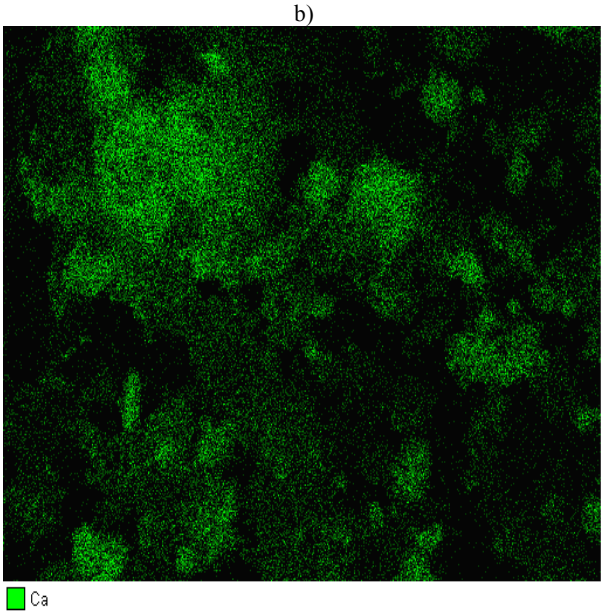


c)

Figure 4 X-ray distribution images suggesting the location of elements Fe, Ca, in the microzone in Figure.3

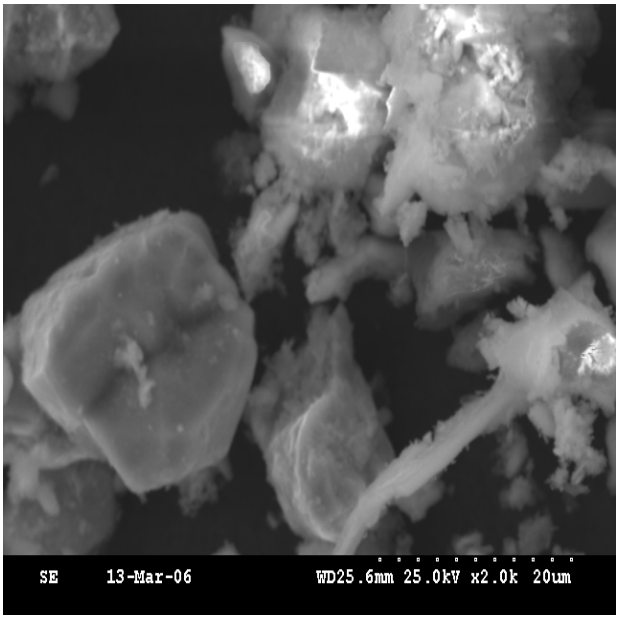
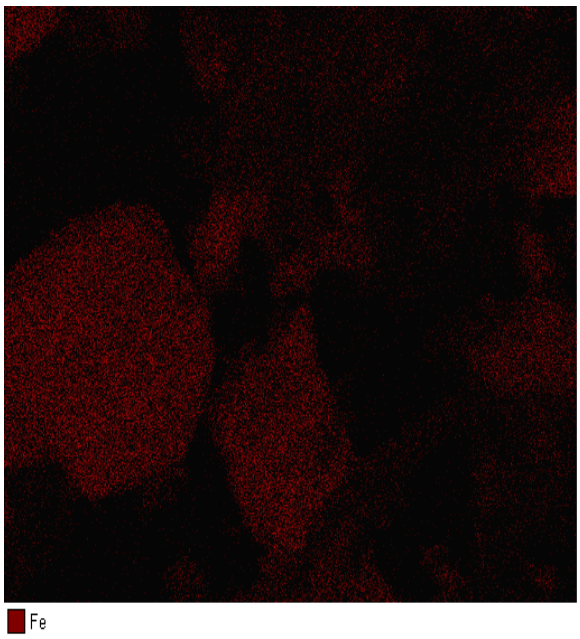


Figure 5 Scanning electron microscopy images (SEI), sample P1



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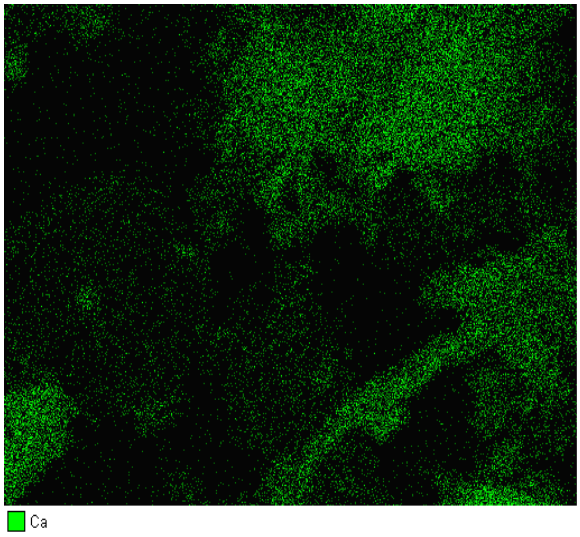
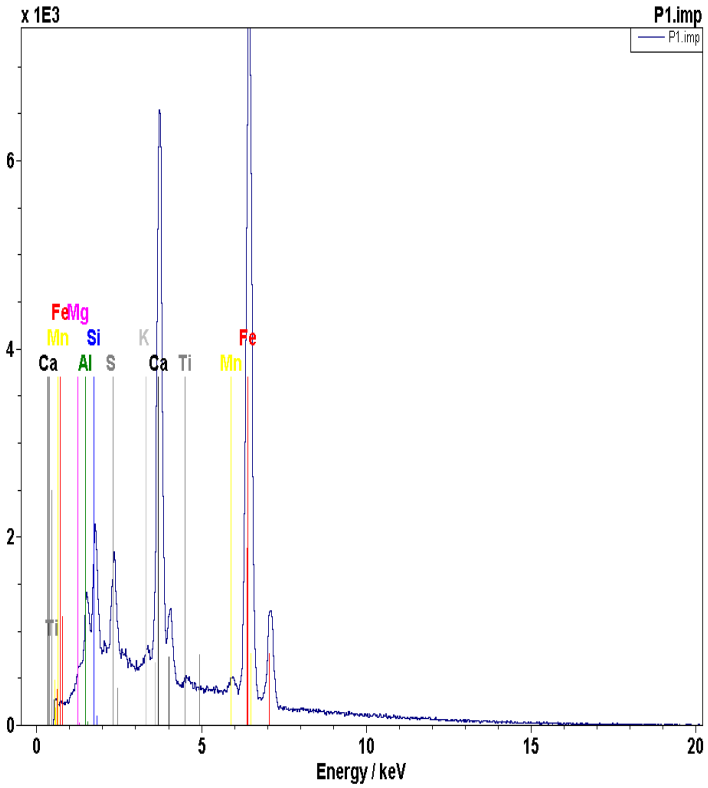


b)

Figure 6 X-ray distribution images suggesting the location of elements Fe, Ca, in the microzone in Figure 5



Figu

Figure 7 Energy Dispersive X-Ray Spectrum (EDS or EDX) associated to microzone in Figure 1

IV. CONCLUSIONS

X-ray spectrum from Figure 7 shows that the sample contains the following elements: Fe, Ca, Mn, Ti, K, S, Al, Si and Mg. It is mentioned that using EDX spectrometer cannot detect light elements (*e.g.* oxygen).[4,5]

Indexing X-ray diffractometry (attached to this report) corresponding to sample P1 comes out the presence of the following phases in sample:

- majority phase Fe_2O_3 (hematite) – indexed F on diffractometry attached;

– minority phases-SiO_2 (Quartz), indexed Q

- CaCO3 (calcite),indexed C1

- $K_2Ca(CO_3)_2$, indexed C2

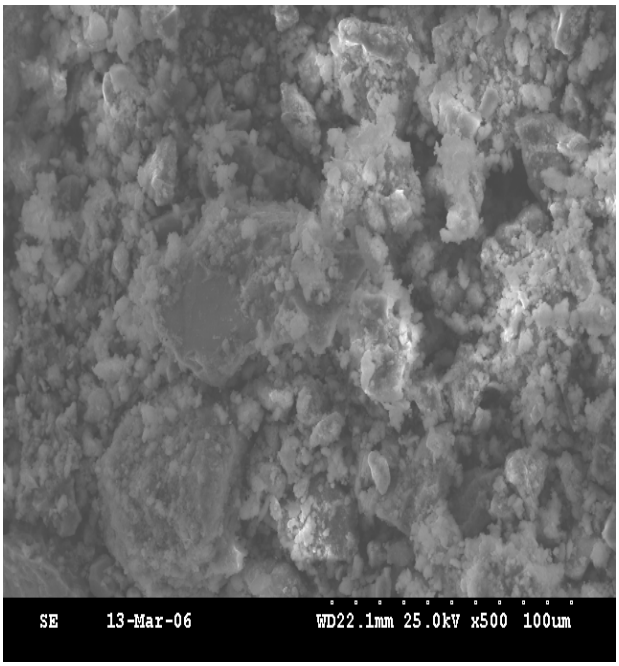
It is possibly that also exists the following minority phases in: FeS_2, $CaSO_4$

Results of particle size determinations are attached to this report.

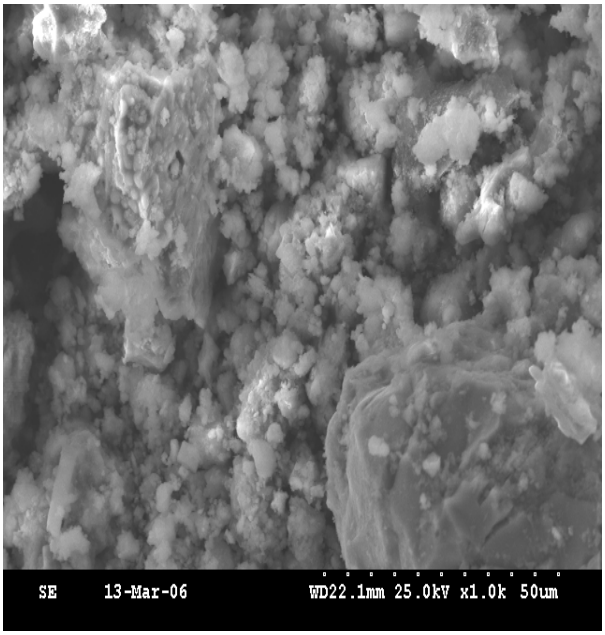
Sample 2

P2 sample morphology is revealed by scanning electron microscopy images, secondary electron images (SEI) in Figure.8 a, b, c and 10.

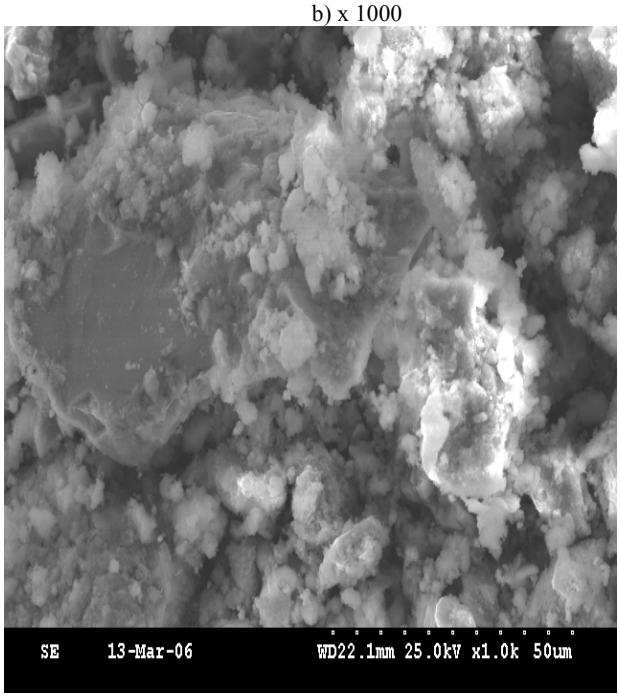
The sample consists of clusters of rounded submicron particles and polyhedral-shaped particles of different sizes.



a) x 500

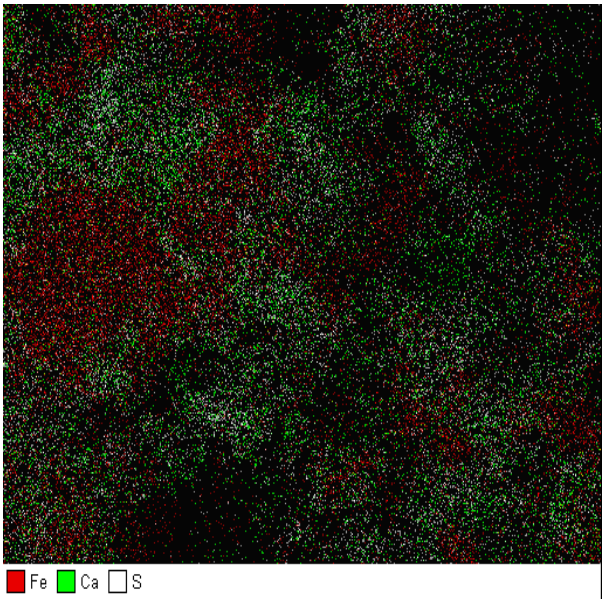


b) x 1000

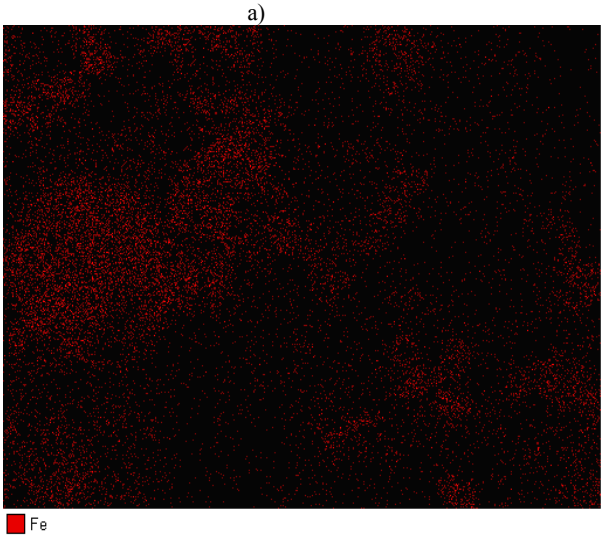


c) x 1000

Figure 8 Scanning electron microscopy images (SEI), sample P2



a)



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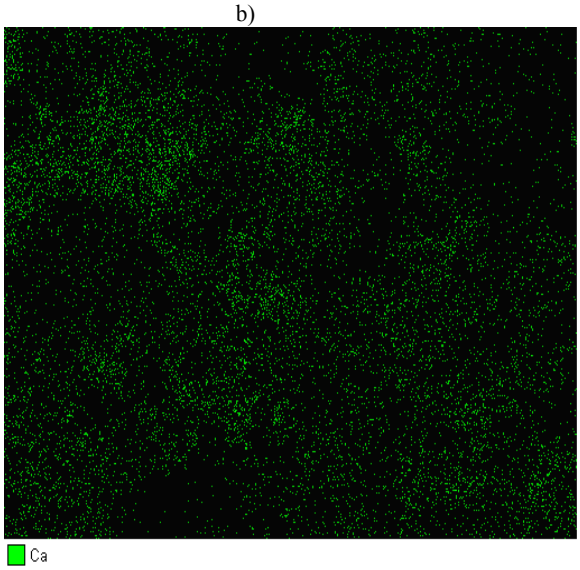
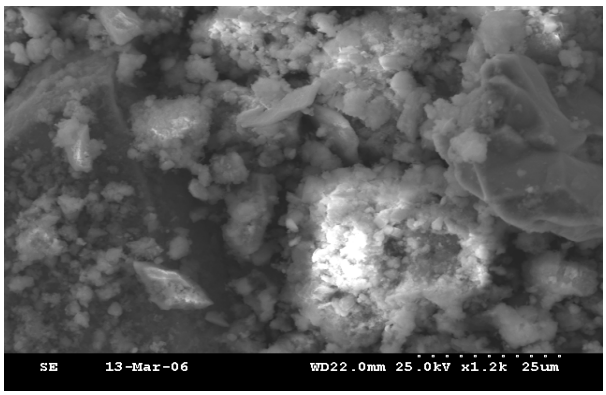


c)

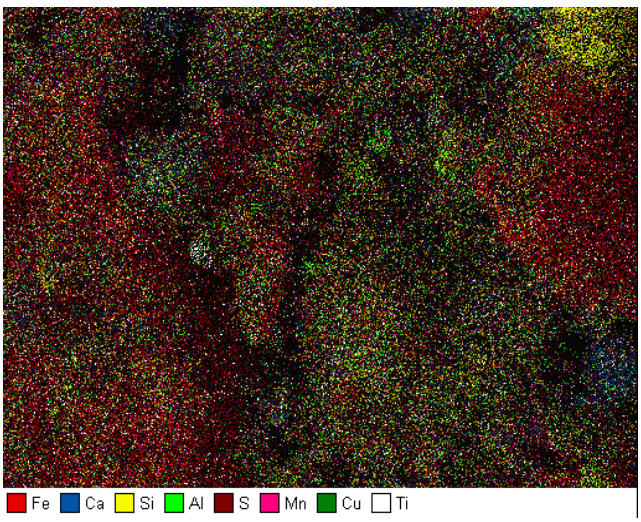
d)

Figure 9 X-ray distribution images suggesting the location of elements Fe, Ca, S in microzone in Figure 8c

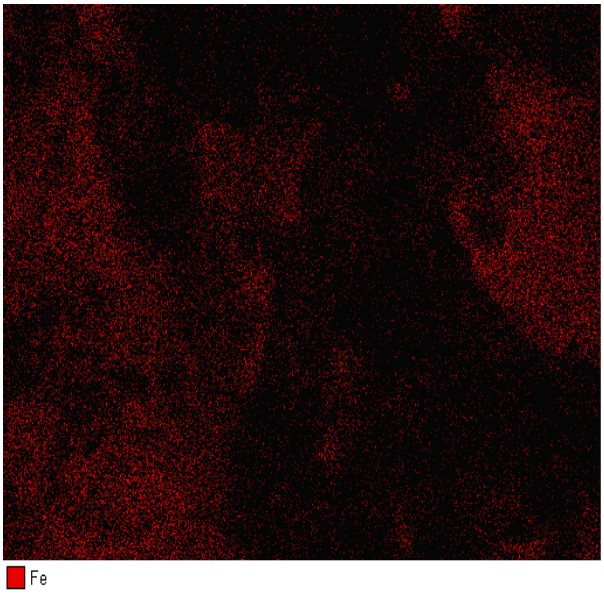


X 1200

Figure 10 Scanning microscopy images (SEI) on another microzone



a)



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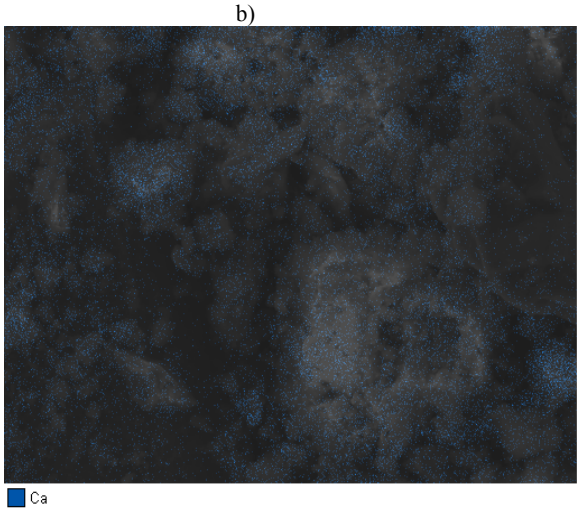
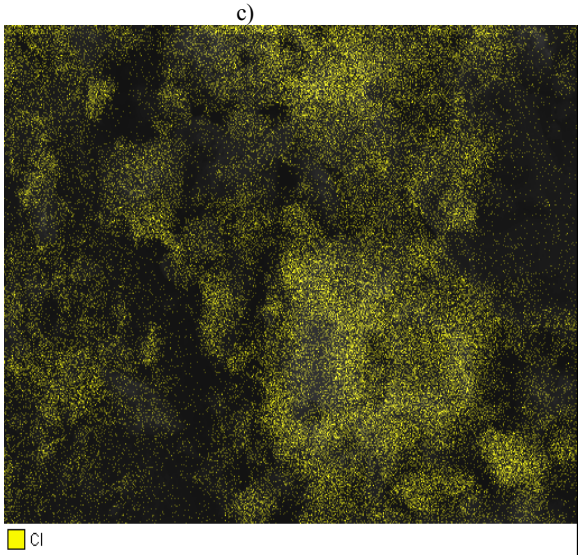
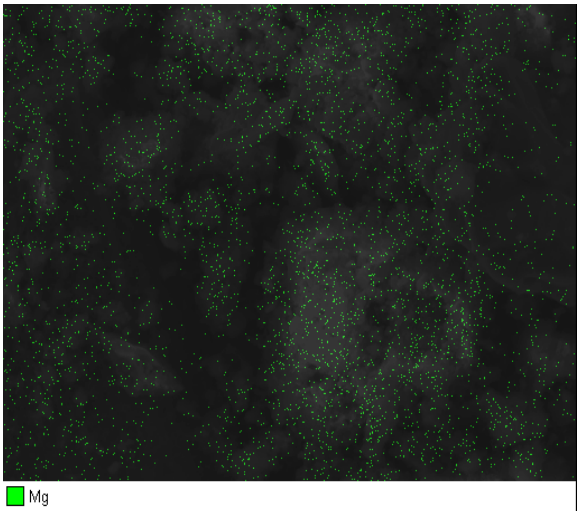


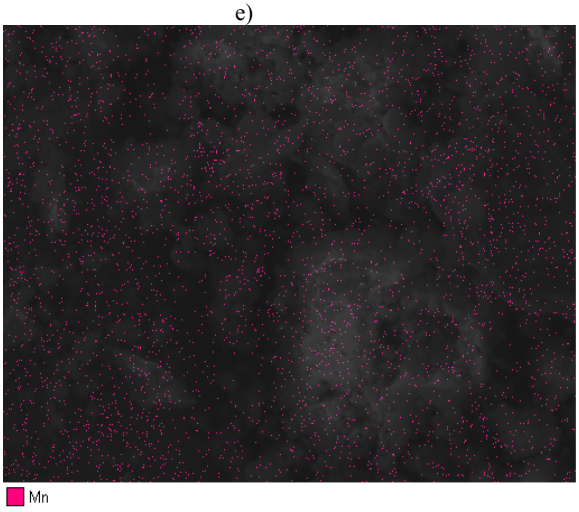
c)



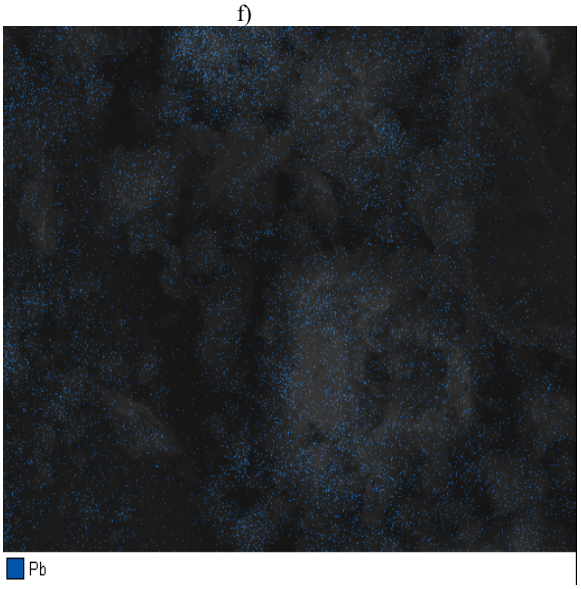
d)



e)



f)



g)

Figure 9 X-ray distribution images suggesting the location of elements Fe Ca, Cl, Mg, Mn, Pb in microzone in Fig.10

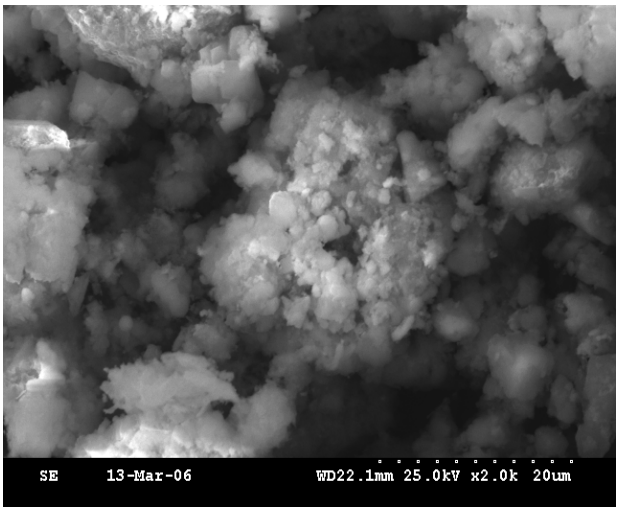


Figure 10 Scanning electron microscopy images (SEI), sample P2- another microzone

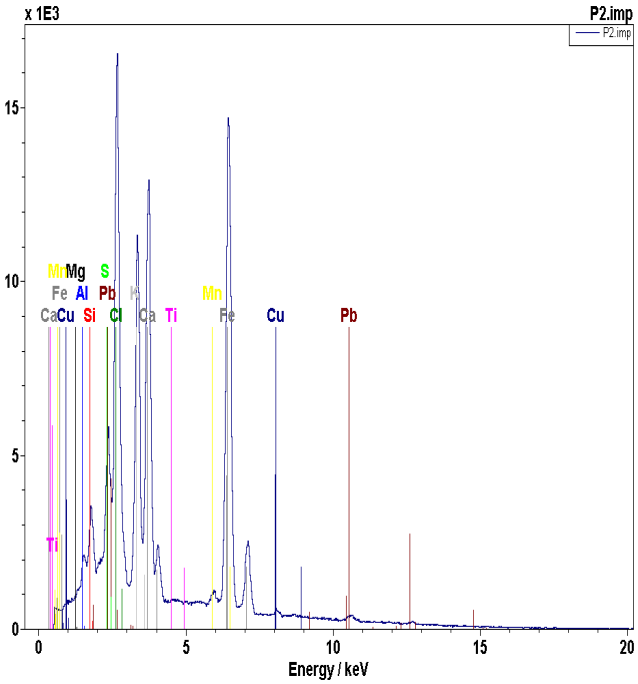


Figure 11 Energy Dispersive X-Ray Spectrum (EDS or EDX) associated to microzone in Figure 8a

REFERENCES

[1] Fahlman, B. D. (2007). *Materials Chemistry*. pp. 282–283. ISBN 1402061196.
[2] Goldstein, J. I. *et al.* (2003). Scanning Electron Microscopy and X-Ray Microanalysis. ISBN 0306472929 .
[3]. Study of EDS vs. WDS accuracy in common labs.
[4]. Tomáš Navrátil, Ivana Šestáková, and Vladimír Mareček, Supported phospholipid bilayers and transport of heavy metals across them, DEVELOPMENT, ENERGY, ENVIRONMENT, ECONOMICS International Conference on Development, Energy, Environment, Economics (DEEE '10) Puerto De La Cruz, Tenerife November 30-December 2, 2010, ISBN: 978-960-474-253-0, pp.192-197.
[5]. Syarif, T. N. Rohmannudin, M. Z. Omar, Z. Sajuri, A. R. Daud, Study nn Formation of Single Phase of a Ti-10at%Mo-10at%Cr Alloy on Sintering and Solution Treatment",Conf. Proc. of the 1st WSEAS International Conference on Materials Science (MATERIALS'08), pp.48-52.
[6]. B. Hausnerova, T.I Kitano, P. Saha, "Effect of Particle Size Distribution on the Flow Behaviour of Powder Injection Moulding Materials", *2010 Conf. Proc. EMESEG '10., 3rd WSEAS International Conference on EMESEG '10*, .pp. 192-194
[7] Mihaiella Cretu, Victoria Teleaba, Silviu Ionescu, Adina Ionescu, Pollution Scenarios through Atmospheric Dispersion Modelling based on Real Measurements - Impact on Human Health, WSEAS TRANSACTIONS on ENVIRONMENT and DEVELOPMENT, Issue 8, Volume 6, August 2010, ISSN: 1790-5079.
[8] Mihaiella Cretu, Victoria Teleaba, Silviu Ionescu, Adina Ionescu, *Pollution Scenarios through Atmospheric Dispersion Modelling based on Real Measurements - Impact on Human Health,* WSEAS TRANSACTIONS on ENVIRONMENT and DEVELOPMENT, Issue 8, Volume 6, August 2010, ISSN: 1790-5079.
[9] Ovidiu-Marcel Murarescu, Gica Pehoiu, *Environmental Impact of Landslides in Sub-Carpathian Area between the Valleys of the Rivers Dambovita and Prahova (Romania)*. WSEAS TRANSACTIONS on ENVIRONMENT and DEVELOPMENT, Issue 2, Volume 6, February 2010, ISSN: 1790-5079.
[10]. Gabriela Popa, Cristiana-Zizi Rizescu, Ofelia-Valentina Robescu, Cezarina Necula, ENVIRONMENTAL POLICY AND INDUSTRIAL INNOVATION: INTEGRATING ENVIRONMENT AND ECONOMY THROUGH ECOLOGICAL MODERNIZATION. Mathematics And Computers In Science Engineering archive Proceedings of the 11th WSEAS international conference on mathematics and computers in business and economics and 11th WSEAS international conference on Biology and chemistry, Iasi, Romania,pp: 285-289 ,Year of Publication: 2010 ,ISBN ~ ISSN:1790-2769 , 978-960-474-194-6.World Scientific and Engineering Academy and Society (WSEAS) Stevens Point, Wisconsin, USA.

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- Ph.D. candidate, Research Theme – *Heavy Metals on Environmental and Health Risks*
Publications
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EDUCATION
- 1972 – 1977 Polytechnique Universirty of Bucharest – Metallurgy Faculty.
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- 1977 – 1997 ICEM Bucharest;
- 1997 – 1998 – 2002 "Valahia"University from Targoviste - Chief of Papers,
- 1997 . Ph.D in technical Sciences from Polytechnique Institute of Bucharest – Thesis Research regarding influence of manufacture and processing parameteres on the physico-chemical and technological properties of alloys from platinic materials.
- 1998 – 2002 "Valahia"University from Targoviste, Reader
-Supervisor of doctoral studies – Fundamental field: Engineering sciences; Field: Materials Engineering
- 2002- until now Professor, Ph.D, "Valahia"University from Targoviste.
Job endorsed /Professional field: Didactic, scientific research, academic administration
OTHER SKILLS AND COMPETENCES:
Representative Awards selected
- Attestation CNRS – Vitry – Centre d'Etudes de Chimie Metallurgique, iulie 2002
- Excellence Award for outstanding and new materials in Microtechnologies, November, 2003.
- Handbooks, courses, guidelines – 8 (author)
- Patents – 4 (co authors)
- Composite materials;
- Emergent materials;
- Nano technologies;
- Phyisico-chemical processes in heterogeneous systems;
- Presious metals
- Member of professional associations:
-Member of "Institute of Standards Engineers" India since 1995.
- Member of Romanian Society for Metallurgy since 1990.
- Member of the Society of Chemistry, Romania since 2006.
- Member of the Romanian Society for Biomaterials since 2006.
Professional Skills selected
- Improvement of existing technologies in metallurgy and specific introduction of new advanced materials technologies
- Making and processing special alloys.
- Teaching (holder of disciplines in the last five years):

- Smart materials
- Shape memory alloys
- Procedures for obtaining advanced materials unconventional
- Non-crystalline materials
- Powder metallurgy
- Composite materials
- Emergent materials
- Nanotechnologies
- Physico-chemical proceses in heterogeneous systems
- Scientific activity (syntheses)
- Academy reviews – 1 (co author)
- Profile Society Reviews – 7 (co author)
– University gazettes – 19 (authors)
- International conferences in the country – 4 (co author)
- International conferences abroad 9 (author), 7 (co author)
- Foreign specialized reviews – 1 (author), 5 (co author).
- National Scientific Events – 15 (author), 1 (co author)
- Grants since the last promotion: Academy – 1 project (member); CNCSIS – 1 project (director); MATNANTECH – 1 project (director) and 2 (member); RELANSIN – 2 (member); CEEX – 2 projects (director).

– Research fields approached selected
- Thermodinamics and kinetics of physico - chemical processes from alloys manufacture
- Chemical equilibrium, diffusion phenomena, surface properties of heterogeneous systems at manufacture and processing of special alloys
- **Composite materials**

Publications

- Articles ISI: 110

– Standardisation. Standard Promotion, Report of The Twenty –Eight International Training Programme in Standardisation and Quality Systems for Developing Countries, New Delhi, 11 October to 8 December 1995, India (Bureau of Indian Standards);

Didactic papers for higher education: 10

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