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CHEMICAL GRADE SILICON
A STUDY USING THE TECHNIQUES OF EDS, SEM AND NAA

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The purity's study of the chemical grade silicon are so important due its application in the production of silicones. In Brazil it is necessary to produce materials that assists to the specifications of the chemical grade silicon (CGSi) and to get this aim the study of the composition, degree of cleaning(low level of non-metallic inclusion), and microstructural parameters (intermetallic composition and distribution) are needed (Rodrigues *et al.*, 1999).

In the present work the results obtained using the techniques EDS(Energy Dispersive X-Ray Spectrometer), SEM (Scanning Electron Microscopy) and NAA (Neutron Activation Analysis) are shown. The study presented here, objects a characterization and chemical microanalysis of the material 9L20Fe (a sample of CGSi produced by the Metallurgy Department of IPT).

The sample was carefully prepared, using mechanical polishing (Dubrous *et al.*,1990) and images obtained by the combination of SEM (backscattered electron image) and EDS. The equipment utilized was a JXA-6400 – Electron probe microanalyser – JEOL with an EDS NORAN Instruments.

The results by NAA were obtained after irradiation of about 80 mg of the sample and the standards of the elements analyzed in IEA-R1m reactor of the IPEN-CNEN/SP. The short half-live radioisotopes like ^{51}Ti, ^{49}Ca, ^{56}Mn, ^{52}V and ^{66}Cu were determined after a irradiation of 1 min at a flux of about 10^{11} n cm^{-2} s^{-1}. The elements Cr, Fe, Zr and Ni were determined after 8 h of irradiation at a thermal neutrons flux of about 10^{12} n cm^{-2} s^{-1}. After an adequate cooling period the measurements of the induced gamma-ray activity were made in a spectrometer of gamma-ray. The concentration of the elements were calculate by the comparative method

Analyzing the results for the element Ca obtained in SEM-EDS, table 1, and the results obtained in NAA, table 2, we can observe that this element appears in the five regions analyzed in SEM-EDS (figure 1) and was not detected by NAA. Analyzing the intermetallic phase(pontual region analyze) a variation (1.49 to 12.15) in weight is observed for Ca.

The element Zr was detected in the sample by NAA (12 ± 7 ug g^{-1}).In this intermetallic phase the element Zr was not detected by SEM-EDS.

The elements V, Cr, Ni , (respectively in the concentration of 5.5 ± 0.8; 14.1 ± 0.1; 13 ± 3, in μg g^{-1}) were detected by NAA and also found in some of the analyzed phases by SEM/EDS.

The elements Ti, Mn and Fe were detected for both techniques employed. The element Cu was not detect by NAA or by SEM-EDS.

A characterization of the intermetallics was made using the techniques employed. The knowledge of chemical composition of the material analyzed are important to predict silicon behavior. This kind of study can be helpful in these material production.

References

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Rodrigues, D.; Filho, C.A.F.R.; Ferreira, J.B.; Kashiwaba, J.; Salgado, L.; Nogueira, P. Chemical grade silicon particles produced by atomization, *Materials Science Forum*, v.299-300, p.182-189, 1999.

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Tabel 1 - Results of CGSi $9L20_{Fe}$ using EDS (% wt) – micrography 3898

Elements/regions	1	2	3	4	5
Al	26.38	22.18	41.63	37.08	41.09
Si	32.65	31.93	30.59	31.54	29.89
Ca	6.77	5.06	3.05	12.15	1.49
Ti	0.11	7.19	0.22	2.41	0.12
V	0.00	0.00	0.00	0.15	0.00
Cr	0.09	0.00	0.20	0.15	0.27
Mn	0.21	0.29	0.96	0.28	0.49
Fe	33.68	33.35	23.52	16.24	26.65
Ni	0.10	0.00	0.10	0.00	0.00
Cu	0.00	0.00	0.00	0.00	0.00
Zr	0.00	0.00	0.00	0.00	0.00

Table 2 - Results of CGSi $9L20_{Fe}$ using NAA

Elem.	Ca	Cr	Cu	Fe (%)	Mn	Ni	Ti	V	Zr
$\mu g\ g^{-1}$	*	14.1 ± 0.1	nd	0.237 ± 0.011	39 ± 1	13 ± 3	207 ± 52	5.5 ± 0.8	12 ± 7

Obs.: * - element not found

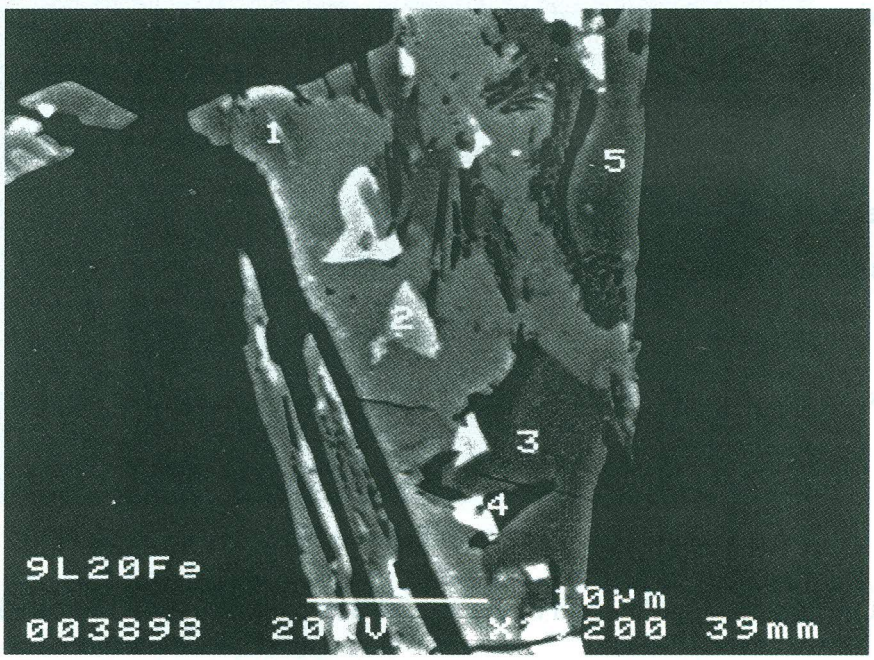

Figure 1 – SEM photomicrograph of a intermetallic CGSi