MICROESTRUTURAL AND MICROANALYSIS STUDIES OF INTERMETALLIC PHASES IN A SAMPLE OF CHEMICAL GRADE SILICON

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Abstract

In this work a methodology combining the techniques SEM (Scanning Electron Microscopy) and EDS (Energy Dispersive X-Ray Spectrometer) are utilized in the characterization of 9L10R, a sample of chemical grade silicon produced by the Metallurgy Departament of IPT (Instituto de Pesquisas Tecnológicas).

Differents phases were found in the sample and the presence of elements as Fe, Ti, Al, V. Ca,.Mn, Zr, Ni and Cr was detected.

Introduction

Silicon is widely used in a number of applications including , the foundry industry, steel manufacture , aluminum alloys, ceramic components , silicone's industry , and the electronic industry. These applications requires differents levels of silicon purity ranging from the metallurgical grade for the foundry industry to a very high purity silicon grade for the integrated circuit industry (1).

Studies to make a characterization of the silicon's purity are important since in much of its apllications a higher purity is needed(2).

Optical microscopy and microanalytical techniques can help in the interpretation of variables like grain size, grain shape and phase distribution. Informations obtained with these techniques can improve the production of materials with a better performance.

The SEM gives high resolution and greater depth of the field image. Metal silicides of Fe, Ti, Al, V, Ca, Mn, Zr and Ni have been fully characterized using a combination of these electron optical instruments and microanalytical techniques (3).

Methods of analysis

Scanning Electron Microscopy (SEM)

In this method , the Si sample surface is analysed by an electron microprobe. The effective magnification is about 20.000 , the resolution is 5 nanometers and the depth of field is significant (10 nanometers for a 10.000 magnification). SEM can be coupled with an X-ray analysis or an image processing system. Samples must be carefully prepared, generally using mechanical polishing(3)

The equipment utilized was JXA-6400 – Electron probe microanalyser – JEOL with an EDS NORAN Instruments

Images

The amount of secondary or backscattered electron emitted from the material depends in particular on the average atomic number of the constituents and responsible for the differential contrast on the electron images. The emitted X-rays characterize the elements of the material and can be used to obtain repartitioning maps.

Chemical analysis

By local analysis (fixed probe) , elements from boron to uranium can be determined into the different phases. The emitted characteristic X-rays are analysed using two types of spectrometers:

a) Energy Dispersive Spectrometers (EDS) – using a SEM provides a spectrum from a volume 2 to 5 microns deep depending upon the excitation potential used. It is an excellent technique to obtain a qualitative analysis, however it can be used for quantitative analysis only to a limited extent.

b) Wavelenght Dispersive Spectrometers (WDS) – which give the chemical composition of the analysed phases. In this method standards of well known composition (generally single phase) are used.

Image Processing Method

In general , the different phases can be differentiated due to their atomic number contrast obtained on the electron images and their relative area fractions can be determined using an image processing system.

Microscopie Examination with Scanning Electron Microscopy

With the electron images, precipitated phases of different nature are revealled. Some of them are very small and are easily distinguishable by contrast images.

Qualitative analysis by EDS

The complexity of the compounds is exhibited in the spectras. In a same area of the sample , the compounds always contain silicon associated with two, three or even four main constituints.

Results and Discussion

Figure 1 shows the intermetallics presents in the silicon sample. The differents phases can be observed by the constrast of secondary electron image from SEM.

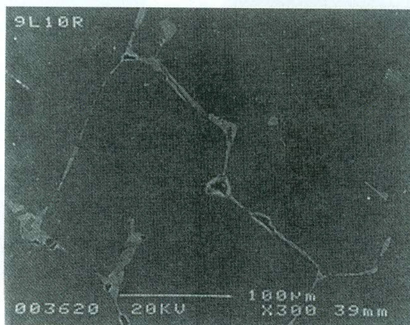

Fig. 1. Secondary Electron Image of a silicon sample (SEM)

Figure 2 and 3 presents the secondary electron image of differents phases of the silicon sample where its was possible, through constrast differences, to determine four regions observed on Figure 2 and six in the Figure 3 . For each region, an EDS analysis were realized and the elements found in each one are presented in the table I.

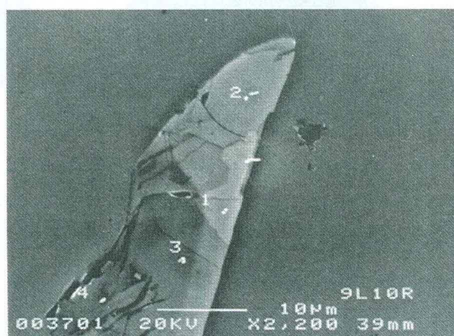

Fig.2. Secondary eletron image of a intermetallic of the silicon sample(SEM)

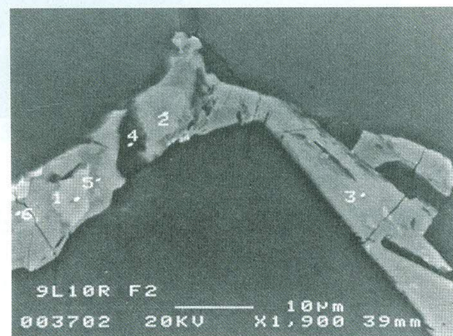

Fig.3. Secondary eletron image of a intermetallic of the silicon sample(SEM)

Elements	F21	F22	F23	F24	F31	F32	F33	F34	F35	F36
Fe	30.46	33.00	14.77	5.62	30.35	33.03	33.08	5.57	28.58	26.92
Ti	22.06	*	*	*	20.47	0.06	0.18	0.08	0.32	2.68
Al	17.20	25.92	11.20	7.60	19.59	26.55	26.14	34.89	34.56	35.85
V	0.13	*	0.09	*	0.28	*	*	*	*	*
Ca	0.08	6.37	2.74	*	0.29	6.57	6.22	24.01	2.35	0.60
Mn	0.67	0.47	0.80	0.19	0.56	0.50	0.32	0.20	0.92	0.88
Zr	0.27	*	*	*	0.20	*	*	*	*	*
Ni	*	*	0.27	*	0.40	*	0.22	0.20	0.21	*
Cr	*	0.21	0.09	0.17	0.08	*	*	0.12	0.26	0.31
Si	29.14	34.03	70.73	86.42	27.79	33.29	33.84	34.93	32.80	32.75

Table I – Semi-quantitative results of elements presents in the phases of fig. 2 and 3 (Wt%) with ZAF correction, in the silicon sample (9L10R), obtained by EDS.
obs.: F2 – figure 2 , F3 – figure 3, * - element not found

Figures 4 and 5 presents the X-Rays images from the phases presented in the figure 3 . These images show the distribution of the elements Ca and Fe present in some regions of the sample.

It was observed in the Figure 2 that the elements Fe, Al, Mn and Si are present in all the analysed regions, but in differents contents. The elements Fe and Si have the higher percentual in weight.The variation of the element Fe was 5.62% to 30.46% and the variation of silicon was 29.14% to 86.42%. The element Mn presents lower contents in all analysed regions, less than 1% in weight.

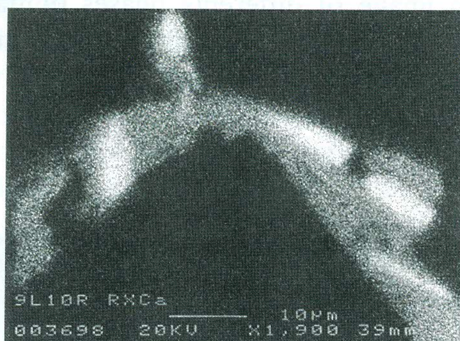

Fig.4 – X-Rays images from Ca distribution present in the Figure 3.

Fig 5 – X-Rays images from Fe distribution present in the Figure 3

In Figure 3 it can be seen that in the region 1 all of the elements were present. The elements Fe, Ti, Al, Ca, Mn and Si were present in all the six regions studied. The lowest concentrations were found to the Mn. The highest concentrations were found to the elements Fe, Al, and Si.

Comparing the two Si samples we observe that contents of Si in the second Si sample (figure 3) are similar in the six regions, while in the first Si sample (figure 2), a great difference between the contents in the four analysed regions.

Conclusions

Analysis using electron optical tools and techniques such as Scanning Microscope (secundary electron images),electron microprobe (analyzed by EDS spectrometer) has given good results in the characterization of a sample of chemical silicon grade.

The results obtained from this study indicate the heterogenity of the material. It was possible to detected Fe, Ti, Al, V, Ca, Mn, Zr, Ni and Cr in the analysed sample, although in each phase presented differents contents of each element were found.

Bibliography

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