Mineral processing of low quality zircon concentrates and preconcentrates

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In NE Brazil, Millennium Inorganic Chemicals do Brasil S/A produces zircon concentrates with variable contents of ZrO_2. One of such concentrates, named zirconite B, presents a low ZrO_2 value and high content of contaminants, being considered as a low quality concentrate. The deposit consists of coastal dunes, and the minerals are located in the base, coated by a clayey–ferruginous pellicle, which interferes in the zircon beneficiation. Besides, two other causes influence the production of the low quality concentrate: mineral inclusions and the development of metamictes in the zircons. The present work, conducted on the zirconite B concentrate and in the preconcentrate, characterises the pellicle's nature, identifies the mineral inclusions in the zircons, illustrates the occurrence of metamictes in the zircons and suggest alternative routes to the production of a high quality zircon good, from the zirconite B concentrate and the zircon's preconcentrate.

Keywords: Mineral processing, Zircon, Ilmenite, Tailings

Introduction

The zircon is an accessory mineral in a variety of igneous rocks, especially in those containing sodic feldspars, such as granite, senate and diorite. This mineral is one of the first to crystallise in the cooling of magma and in some cases, incorporates inclusions. Zircon is found in metamorphic rocks like gneisses and schist. Owing to the rare occurrence in the primary rocks, zircon has economic relevance in sedimentary deposits (mineral sands), where it appears with ilmenite, rutile, staurolite, monazite and other high density.[1–4]

Mineral sands' deposits are common in the seacoast in many countries. Australia concentrates the biggest reserves and it is at the same time the world's largest producer and exporter of zircon concentrate. In 2001, Brazilian reserves were equal to 3·4% of the world's and the amount produced in the country (21 thousand tons) represented 2·2% of the world's production. Millennium Inorganic Chemicals do Brasil S/A produced around 11 thousand tons, exclusively destined to the domestic market.[5]

In the majority of the producing countries, the more interesting deposits from the economical point of view are unconsolidated or partially consolidated placers. The processing of the run of mine (ROM) ore removes quartz and other light minerals. In the size classification, Reichert cones, spirals or other hydraulic classification devices are used. In some deposits, a step of attrition is necessary for the removal of possible adherent substances from the minerals. After the drying, electrostatic and magnetic separators are employed to generate concentrates of various heavy minerals, such as ilmenite, rutile, zircon and others.[2,4]

The relevance of zircon as a raw material is related to its chemical and physical properties, such as high hardness and refraction index, melting point above 1800°C and others.[2,3] These features favour the zircon's utilisation as a raw material in different industrial sectors, and the main applications are in ceramic products, melting and refractories. In Brazil, the average cost of a superior quality zircon concentrate is about $290. In the international market, the same product costs $350/ton.[5]

In the zircon crystal lattice, a process known as metamictisation occurs, which consists on the replacement of Zr by other chemical elements. Smith *et al.*[6] verified that the zircon's structure can accommodate external ions in the process and in some grains, the authors found that 40% of all Zr content was replaced by Y, Th, Nb, U and Ca cations, among others, and that a low amount of silicon was replaced by aluminium and phosphorus. Gorz and White[7] showed that the iron enters the crystal lattice replacing silicon, and that its concentration can vary between 400 and 15000 ppm. Hartmann *et al.*,[8] in a study with metamictic zircons from the Caçapava granite, in Southern Brazil, identified chemical exchanges between the zircon and the metamorphic environment, and found that the metamictised portions were strongly enriched in Fe ($\times$ 100) and in other elements.

The Millennium Company's deposit, known as Guaju Mine, is located in the municipality of Mataraca, in Paraíba State, NE Brazil. The main mineral good

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produced is ilmenite. Zircon and rutile are byproducts. The heavy minerals are disseminated in mineral sands deposits, as 30 m high dunes (mean value). The mining takes place by mechanical dismounting (bucket wheel dredge) and the mineral processing occurs in gravity, electrostatic and magnetic devices.

Studies made by Sabedot and Sampaio[9] on the deposit have indicated that 98% of the minerals in the base of the dunes are opaque due to the existence of a strongly adhered pellicle. On the top of the deposit, conversely, around 95% of the minerals have a vitreous glow and there is no pellicle on the particles. After the characterisation, Sabedot and Sampaio[10] applied an attrition process to the zirconite B concentrate, in laboratory scale, to optimise the operation parameters and the removal of pellicle. The process was efficient and removed the pellicle adhered to the minerals. The attritioned product was passed by the electrostatic and magnetic separators once again and the majority of the contaminant minerals were removed, yielding a high ZrO_2 product.

In the ore processing, zircon concentrate is obtained from a current name zircon preconcentrate (PCZ), which is the reject from the ilmenite concentration. From the mineralogical point of view, it is a material with more than 80% in zircon, contaminated by rutile, ilmenite, cyanite, staurolite and others. The quality of PCZ varies according to the portion of the deposit being mined: if the works are in the base, the majority of the heavy minerals will present the adhered pellicle. This phenomenon will not appear if the top sediments are those being extracted. In the first case, the beneficiation of PCZ will yield low quality zircon concentrates and in the second one, high quality products will be generated.

The PCZ is processed by batches, in concentrating tables, electrostatic and magnetic separators. Zircon is a non-conducting and non-magnetic mineral. The pellicle around the mineral reduces the efficiency of electrostatic and magnetic devices in the separation of the contaminants. As a consequence, part of those is incorporated in the zircon rich fraction what results in a low quality final product.

Considering that the PCZ is the main source of all the concentrates produced at the Guaju Mine, an operation in the semipilot scale was conducted to verify if the results with the attrition experiments in laboratory would reproduce in a largest scale. The attritioned PCZ was then processed in the electrostatic and magnetic devices, and the results are commented in this paper.

The mineralogical characteristics of the Guaju's zircons also determine the yielding of low quality concentrates. Studies conducted with these concentrates indicate that several zircons contain mineral inclusions of variable dimensions.

Zircon is a mineral often utilised in the detection of rocks. One of the aspects referred in the literature is a process named metamictisation, which disorientates the zircon's crystal structure and incorporates alien substances to it. The low quality concentrate was also investigated in relation to the occurrence of this process.

Since mineral inclusions also determine the final quality of the zircon concentrate, physical procedures were tested on the PCZ to evaluate the performance in the operations of heating, milling and removal of inclusions. The results of these investigations are also presented and discussed in this paper.

Materials and methods

Attrition in the PCZ

Twelve 40 kg samples of PCZ were tested in semipilot attrition equipment. The tests and results in laboratory scale were optimised: concentration of solids in the pulp, concentration of NaOH in the pulp and stirring time. In Table 1, the parameters' values according to the runs are listed. Run 1 was executed without NaOH because the company, due to environmental issues, wanted to know about the performance without the chemical.

Aliquots of the attritioned products were analysed in the binocular magnifying lens and subjectively qualified in relation to the presence of grains with the adhered pellicle. So, the performance of the experiments was qualified as regular, good or outstanding.

To corroborate the efficiency of the treatment made, the spectrum of reflectance was measured, with a Minolta CM 508d spectrophotometer coupled to the OJ Holder accessory, in three samples: PCZ (without attrition) and runs 7 and 11 of the attrition studies. Each sample was measured 11 times and the results were submitted to a statistical treatment (t test),[11] to determine the difference between two mean values, considering the same variance for the samples, in a level of confidence equal to 95%. Considering that the clayey–ferruginous pellicle gives the grain's surface a reddish colour and an opaque appearance, the pellicle's removal alters the colour of the attritioned material and as a consequence, also the values of the colorimetric parameters are altered in the sample, before and after the attrition procedure.

The performance of the runs with and without the addition of NaOH was verified in attritioned samples (runs 1 and 5), tested in electrostatic and magnetic equipments. The electrostatic separator utilised was a Carpco, model HT 111-15, with a 14 in. induced roll. The wet magnetic separator was an Inbras/Eriez, model 4L, with a 2×4 mm matrix, operating in a closed gap with 17 000 G. The chemical analyses of the PCZ and the products of beneficiation were made in a Philips X-ray spectrophotometer, model PW1660.

Samples from the reject of the attrition process were separated, dried and analysed in a Philips X-ray diffractometer to identify the mineral phases present in

Table 1 Operation parameters in PCZ attrition tests (semipilot scale)

Run	Pulp, % solids	NaOH, g t^{-1} pulp	Time, min
1	80	0	20
2	80	400	20
3	80	400	40
4	80	500	20
5	80	600	20
6	70	500	20
7	70	600	20
8	80	500	30
9	80	600	30
10	70	500	30
11	70	600	30
12	80	600	10

the pellicle. Once dried, the samples appeared like a clayey brown mass.

Mineral inclusions

Approximately 300 zircon grains with mineral inclusions were selected from a sample of the zirconite B concentrate for the mineralogical characterisation of the inclusions. After the selection, the grains were collated on a glass support and abraded, exposing 68 mineral inclusions that were photographed and analysed under an optical microscope and under an analytical microprobe.

Metamictisation

The metamictisation process was investigated in around 800 grains of the zirconite B concentrate, randomly collected. The grains were pasted on a glass support and then they were abraded and coated with graphite for analysis in a SX50 Cameca electron microprobe.

Physical treatments in the zirconite B concentrate

The mineral inclusions in the zircons determine the elevated contents of contaminant substances in the zirconite B concentrate. Physical treatments, such as heating in the microwave oven, milling, electrostatic and magnetic separation, were implemented in the concentrate, to evaluate the performance in the removal of inclusions, by analysis after the treatments.

Heating in the microwave oven

The use of the microwave oven in the heating of the zircon's concentrate was considered due to the discussions by Haque[12] and Kingman and Rowson,[13] in their reviews on the microwave energy in ore processing technologies. According to these researchers, the heating of minerals varies according to their chemical compositions and physical properties. Generally, metallic minerals, such as pyrite, heated by microwaves for 7 min, can reach temperatures above 1000°C. The temperature for non-metallic and transparent minerals, such as quartz, under the same conditions and time of exposure, remains close to 80°C. Still according to the cited authors, the quick heating with microwaves is important for the release of economic minerals associated with a mineral gangue. Owing to the difference between the mineral's dilatation coefficients, microwaves act to provoke a fracture network in the contact zones between the different minerals and make their separation easier, in a posterior step of grinding and milling. Since zircon is a transparent mineral, the microwave oven was utilised to cause the fracturing in the zones of contact between the mineral and the inclusions. The operation parameters were: power 500 W, heating time 7 min and mass of sample per batch 600 g.

Milling

Milling was utilised to promote the breakage of the inclusion bearing zircons, previously fractured by the microwave's action and thus the release or exposure of the inclusions. An impact mill was used, with continuous feed and withdrawal of material, designed to avoid the excessive comminution of the material. After the milling, the material was sieved at 0·063 mm. The retained fraction was processed in electrostatic and magnetic equipments. The passing fraction, with a size distribution inadequate to the operation in the mentioned equipments, was just chemically analysed.

Electrostatic and magnetic separation

The fraction +0·063 mm was initially treated in the Corona type Krupp Laboratory electrostatic separator. Operation parameters were 30 kV and roll rotation of 100 rev min^{-1}. The non-conducting fraction was treated in a Carpco induced roll magnetic separator, model MIH-13·111-5. The equipment was regulated at its maximum magnetic intensity and with the roll speed set to 33 rev min^{-1}.

Chemical analysis

From all the steps in the processes, samples were taken to the chemical analysis, conducted in a Philips X-ray spectrometer, model PW1660. The following oxides were determined: Fe_2O_3, TiO_2, ZrO_2, Al_2O_3 and P_2O_5.

Results and discussion

Attrition on the PCZ

Subjective evaluation

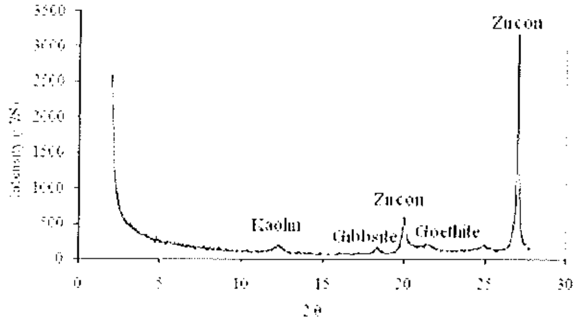
In a subjective evaluation, the performance of the attrition studies was qualified as regular, good or outstanding. The regular performance indicated the presence of grains with clay adhered to shallow or deep cavities, or grains with remains of pellicle, and/or grains with opacity. Runs 1, 6 and 12 in Table 1 received this qualification. The good performance indicated the presence of grains with clay adhered only in some deep cavities, but without remains of pellicle and opacity. Runs 2–5, 7 and 10 in Table 1 were qualified as good. The outstanding performance indicated that the grains were entirely free from pellicle or clay, with shining surfaces. Runs 8, 9 and 11 in Table 1 fell into this qualification.

In the global evaluation of results, the parameters from these runs qualified as good were admitted as ideal values (runs 2, 4, 5 and 7), due to the smaller operation times. Run 7, with a smaller concentration of solids in the pulp, could be elected as the most adequate of all, if this parameter represents diminished energy consumption, but this aspect was not evaluated in the tests. Although runs 8, 9 and 11 presented an outstanding performance, their operation times were considered too long for the process. Runs 1, 6 and 12 were not efficient in the removal of the pellicle, which indicates that the operations without NaOH (run 1) or with a 10 min residence time (run 12) were far from the ideal. Run 6 presented an atypical result, probably as a consequence of some operation problem that was not detected during the experiment.

Objective evaluation

The chemical analysis by X-ray fluorescence proved to be inadequate to evaluate the results, because the pellicle's mass removed by attrition was of little significance, if compared with the original PCZ mass. The results of analyses made before and after the attrition showed differences that coincide with the values of the confidence limits of the analytical method (margin of error) and due to this fact, the colorimetric method was selected as the objective tool to evaluate the process.

In Table 2, the values of mean and standard deviation for brightness and yellowness are listed (calculated by



1 Diffractogram of reject of attrition

2 Mineral inclusions in zircons from low quality concentrate

the ISO 2470 and ASTM E313 norms respectively), referring to the statistical treatment of PCZ, run 7 and run 11 samples. The statistical treatment included a t test to compare the mean values obtained in runs 7 and 11 in relation to the PCZ sample. The t values and the associated probabilities can equally be found in Table 2. Both the t values, above the critical value of 2·05, which corresponds to a 95% confidence interval, and the p values, below 0·1%, confirm that both treatments were effective both in the increase in brightness and in the decrease in the yellowness in the studied samples.

Reject of the attrition

The mineralogical analysis of the reject of the attrition experiments identified peaks of kaolinite, gibbsite, goethite and zircon, as shown in the diffractogram from Fig. 1. The first three peaks characterise the pellicle's composition and are typical of the natural alteration processes that occur in the sediments, mainly when submitted to the percolation of water, either by the phreatic as by the infiltration. The peaks for zircon in the diffractogram represent torn fragments of zircon and/or zircon powder, resulting from the comminution caused by intergranular shocks occurring during the process.

Attrition with and without the use of NaOH

The less efficient performance in the semipilot attrition process was identified in run 1, conducted without the addition of NaOH. A comparative test applied to the attritioned products from this run and from run 5 has shown, in a qualitative way, that the use of the chemical is important in the attrition process. Aliquots from these runs were processed in the electrostatic and magnetic separators, in this sequence. The final, the non-conductor and non-magnetic portion of each aliquot, were analysed by X-ray fluorescence. Table 3 shows the results. The PCZ sample refers to the non-attritioned product and the samples' 'run 1' and 'run 5' refer to the attritioned fractions, processed in the electrostatic and magnetic separators. Rutile and ilmenite are the main contaminants for the PCZ. The values for TiO_2 presented more significant differences between the samples processed with and without NaOH, and there is around 100% more TiO_2 in the product attritioned without NaOH. For the remaining oxides, the differences fall within the operational range of error of the equipment.

Generally, the results of semipilot attrition tests evidenced that the operational performances were similar to those obtained in the laboratory scale. Considering the simplicity and the efficacy of the attrition process, it appears to be a viable alternative to be introduced in the industrial scale at the Guaju mine, objectively to the PCZ product, to promote the production of zircon concentrates of higher purity.

Mineral inclusions

Around 28% of the zircon from CZB presents a significant amount of mineral inclusions which can occupy a considerable volume of grains as shown in Fig. 2. By mineralogical study of inclusions, several species were identified (Table 4) among which the magnetic ones, the biotite, the quartz and the ilmenite are the most abundant ones. These minerals, without considering the quartz, represent around 50% of the mass of the inclusions.

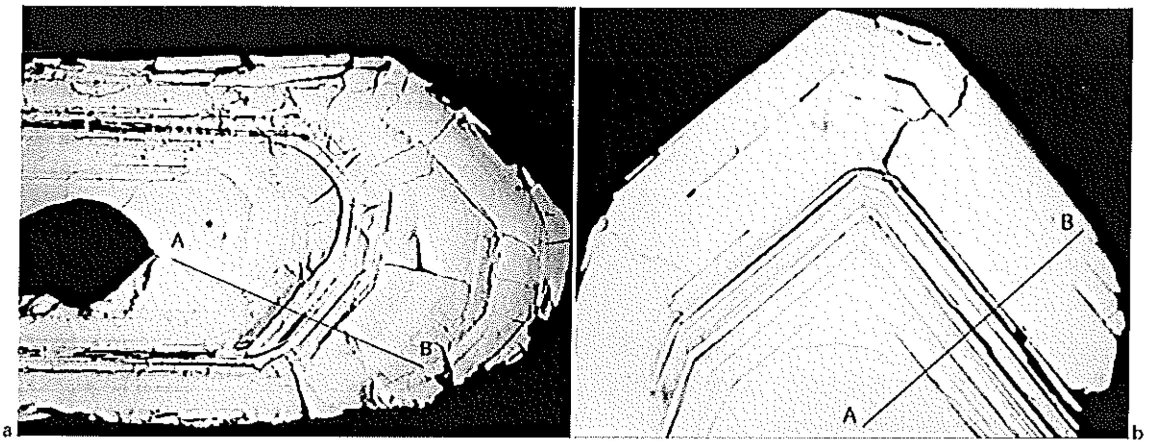
Analysis of inclusions in five magnetic minerals, carried out by SEM, showed that around 60·2% of the mass, on the average, corresponds to the iron element. Inclusions in biotite and ilmenite have, on the average, 30·2 and 46·1%FeO respectively. On the other hand, apatite is the fifth more frequent mineral species found as inclusion. In addition, Al_2O_3 can be found as inclusion in biotite, feldspar and plagioclase. Inclusions may represent an important contaminant fraction of the

Table 2 Statistical comparison of measured colorimetric parameters

	Brightness			Yellowness		
	PCZ	Run 7	Run 11	PCZ	Run 7	Run 11
Mean	16·77	17·31	17·75	28·91	26·24	26·82
Standard deviation	0·34	0·32	0·42	0·90	1·60	1·38
t		3·87	5·98		4·84	4·23
p value, %		0·041	0·001		0·010	0·096

Table 3 Chemical analyses (%) for PCZ and samples attritioned with and without NaOH, processed in electrostatic and magnetic equipments

Sample	TiO_2	Fe_2O_3	P_2O_5	Al_2O_3	ZrO_2
PCZ	7·46	1·49	0·19	1·31	58·36
Run 1: without NaOH	0·65	0·24	0·12	1·09	66·17
Run 5: with NaOH	0·30	0·28	0·11	1·07	66·03



a highly fractured; *b* slightly fractured
3 Images (BSE) of zircons with metamictisation bands

CZB and may explain the fact that after purification, PCZs present high grades of Fe_2O_3, P_2O_5 and Al_2O_3 rendering them low quality products.

Metamictisation

Metamictisation process produces alteration which modifies the zircon chemical composition. The process is carried out in two ways: by the transformation of the elements of the crystal lattice during the process and by the ulterior alterations provoked by solutions which penetrate the mineral through fractures originated during the process.

Figures 3 shows the spectra of the elements Fe, Th and Zr in the structures of two zircon grains picked at random. In each grain, the spectra show that in the dark bands (metamictised areas of the mineral), Fe and Th concentrations appear high while Zr concentration appears low. On the other hand, in the clear bands (areas not metamictised), it appears the contrary, i.e. Fe and Th concentrations appear low and that of Zr appears high.

This characterisation does not allow determining absolute values of alterations in the concentrations of the elements inside the grains. Nevertheless, from the spectra, it can be inferred that alterations must be considerable. For example, in the spectrum corresponding to Zr (Fig. 4) which corresponds to the zircon sample of Fig. 3*a*, the Fe counts of clear bands is ~2500, while in the dark bands, it is around 32 000. For Th, counts are around 2000 in the clear bands and around 15 000 in the dark bands. In the contrary, Zr counts are around 100 000 in the clear bands and drop down to around 63 000 in the dark bands.

The spectra also indicate a variation between 60 000 and 100 000 counts for the Zr. However, the counts in the spectra of Fe and Th appear different in the grains. Those differences must be related to the intensity of the metamictisation process in the interior of the grain which produces more or less alteration of the Zr to some other element. Figure 5 illustrates the spectra of Fe, Th and Zr of the zircon sample shown in Fig. 3*b*. These results confirm the data of Hartmann *et al.*[15] and Nasdala *et al.*[16] who concluded that the areas of zircons with high metamictisation degree generally contain high quantity of the trace elements and they are characterised by higher variations in the concentrations of those elements than in the neighbouring areas which are not metamictised.

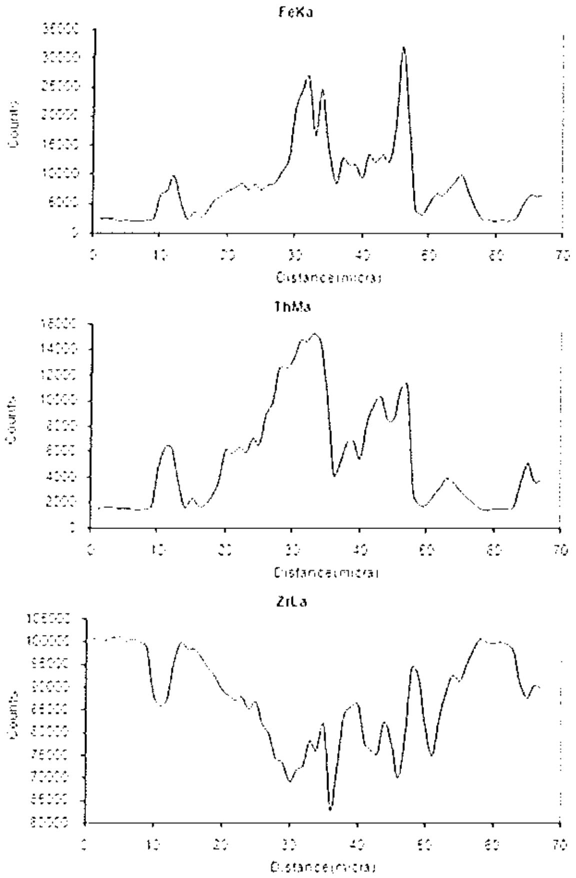
Zircon fractures are related to the metamictisation according to Wopenka *et al.*[14] and Nasdala *et al.*[16] According to them, physicochemical variations during zircon growth produces non-uniform U and Th concentrations in the mineral, causing volume expansions at different rates. In general, the regions rich in U and Th expand faster. Those heterogeneous expansions produce internal pressures throughout the mineral, high enough to produce fractures. Fractures become potential paths where fluids penetrate, allowing leaching of the elements. Figure 3 shows fractures which are common to the zircons analysed by SEM. Those characteristics lead one to conclude that metamictisation is also a factor which determines high grades of contaminant substances and hence a low quality of the raw CZB is produced in the Guaju Mine.

Table 4 Mineral inclusions in zircons, identified by analytical microprobe

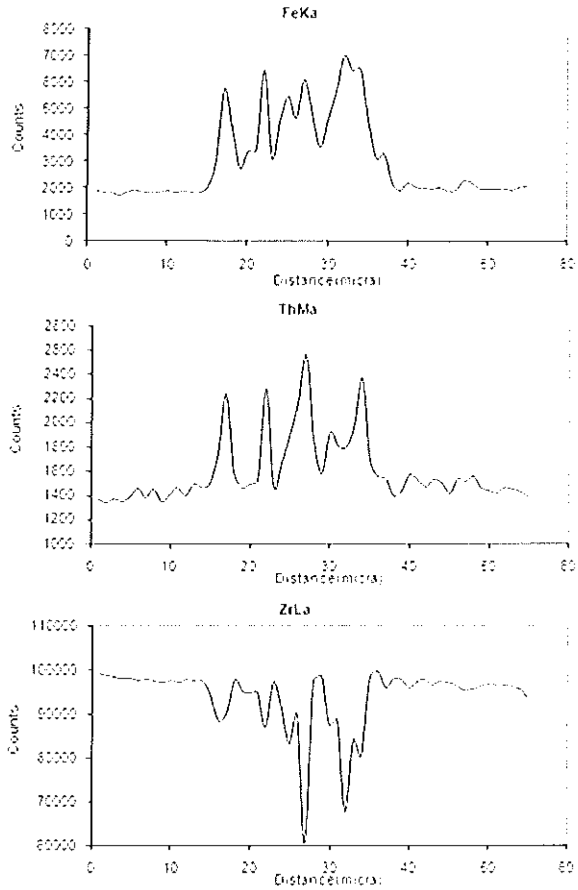
Inclusions	Total	% Grains
Magnetic	14	21
Biotite	13	19
Quartz	10	15
Ilmenite	7	10
Apatite	6	9
Oxide Te–Ti	4	6
Feldspar	4	6
Plagioclase	3	4
Titanite	2	3
Limonite	2	3
Copper minerals	2	3
Amphibole	1	1
Sum	68	100

Physical treatments on the zirconite B concentrate

Table 5 shows a mass balance and chemical analysis after physical processes were applied to CZB. Grinding was considered satisfactory because it generated low amount of fines. As the aim was to fracture zircon grains carrying inclusions in order to liberate or to expose them, it was important to produce the less fine material due to restrictions in electrostatic or magnetic processes. After grinding, many broken grains were found: most of



4 Spectra of distribution of Fe, Th and Zr, obtained by electron microprobe analysis, correspondent to AB scanning shown in Fig. 3*a*



5 Spectra of distribution of Fe, Th and Zr, obtained by electron microprobe analysis, correspondent to AB scanning shown in Fig. 3*b*

them were free from inclusions, and some of them had the inclusions adhered but were exposed as the grains showed in Fig. 6.

In general, ground samples showed low number of grains having big size inclusions. However, it appears that many grains have little size inclusions which appeared incorporated to the mineral. Those facts may indicate that microwave heating is adequate to liberate big inclusions but not so for little ones. High content of Fe_2O_3 and P_2O_5 in the −0·020 mm size fraction (Table 5) also support the idea that heating/grinding processes are effective for liberating inclusions. Analysis of inclusions by SEM showed that most part of inclusions is formed by minerals which contain mainly Fe and P in a minor extent. The inclusions appeared concentrated in the −0·020 mm as a consequence of the processes heating/grinding.

The product +0·063 mm separated by sieving, submitted to an electrostatic process, produced a low amount of conductive material (5·0%) and 80·2% of non-conducting substance. This was considered as a positive factor due to the little amount that would be lost.

The magnetic separation of the conducting material produced a relatively low amount of magnetic product (3·4%) and a significant amount of mix fraction (24·6%). A reduction of the mean diameter D_{50} of the original mass by grinding could have affected the performance of the equipment giving rise to a high volume of mix or could have been due to the exposure of the inclusions of the broken grains which allowed the separation of zircon grains having mineral inclusions.

The non-magnetic product was the final product. As a mass, the recovery of 52·2% is considered poor. However, from quality standpoint, chemical analysis showed that the composition is very close to that of

6 Zircons with exposed inclusions, after comminution and magnetic separation

Table 5 Processes, mass balance and chemical analyses: zirconite B concentrate

Process/product		Mass, %	TiO_2	Fe_2O_3	P_2O_5	Al_2O_3	ZrO_2
Zirconite B concentrate sample		100	0·69	0·27	0·08	1·52	64·1
Sizing of zirconite B	+0·063 mm	85·2	0·2	0·17	0·08	1·32	64·56
Concentrate sample	−0·063, +0·020 mm	12·5	0·12	0·24	0·13	1·01	64·56
	−0·020 mm	2·3	0·19	2·62	0·25	1·62	62·68
Electrostatic separation of +0·063 mm	Conductor	5·0	*	*	*	*	*
	Non-conductor	80·2	0·16	0·17	0·08	1·16	65·29
Magnetic separation of non-conductor	Magnetic	3·4	0·64	0·45	0·19	2·6	63·0
	Mixed	24·6	0·22	0·21	0·1	1·32	65·15
	Non-magnetic	52·2	0·1	0·15	0·06	0·97	65·94

*Sample not evaluated.

zirconite E, the best product produced in the Guaju Mine. As the product did not have the qualities of that product, it could be compared with zirconite I, also produced by the mine which represents a value of 5 in a scale from 1 to 6 in which the number 6 represents zirconite E (zirconite B has a value of 2). From here, it can be stated that the treatment implemented on the CZB produced a quality increase of 3 points leading to a considerable gain in quality as well as in commercial value. The product did not reach the value of 6 due to the contents of contaminant substances which surmount those found in zirconite E, but the ZrO_2 produced had a final value 1% above the minimum necessary to get quality of 6.

Conclusions

1. The high contents of contaminants found in the zirconite B concentrate are caused by three main product's characteristics:
   (i) clayey–ferruginous pellicle in the PCZ obtained from the sediments mined at the base of the deposit. Some minerals, such as ilmenite, cyanite, rutile and monazite, are impregnated by the pellicle and are not removed in the magnetic and electrostatic separations, remaining as contaminants in the zirconite B
   (ii) mineral inclusions in around 28% of the zircons. Most of the identified inclusions consist of minerals that present the contaminant substances to the zirconite B
   (iii) metamictisation in around 15% of the zircons: this is a process that causes the replacement of Zr by other elements, markedly Fe.

2. The attrition in the semipilot scale had performance similar to the tests in the laboratory scale, proving that the operation parameters defined are optimised and that the attrition operation on the PCZ is important for the removal of the clayey–ferruginous pellicle that is impregnated in most of the grains from the inferior part of the deposit. The pellicle is constituted by kaolinite, gibbsite and goethite.

3. The physical treatments applied to the zirconite B concentrate were efficient in the removal of contaminants. The contents of ZrO_2 and contaminants were very similar to those achieved in the zirconite E concentrate, a high quality product elaborated at the Guaju operation.

4. The mass balance of the physical treatments carried out on the zirconite B pointed at a mass recovery of 52·2% as the final product (non-magnetic). Commercially, this product can represent (in terms of weight), a value three times superior to that of zirconite B, because its chemical specifications are very close to the superior quality product also obtained at the Guaju Mine. The finer particle size, caused by the comminution, can represent a higher value aggregated to the final product. Besides the final product, the mixed (24·6%) and −0·063, +0·020 mm (12·5%) fractions presented better contents than the zirconite B, and will probably present more attractive commercial values. The losses, or the products with commercial values smaller than the zirconite B value, due to their chemical specifications, represent the conductor (5·0%), magnetic (3·4%) and −0·020 mm (2·3%) fractions.

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References

1. J. D. Dana: 'Handbook of mineralogy'; 1978, São Paulo, Livros Técnicos e Científicos Editora S/A.
2. T. E. Garnar: in 'Industrial minerals and rocks', 6th edn, (ed. D. D. Carr), 1159–1165; 1994, Littleton, CO, Society for Mining, Metallurgy and Exploration Inc.
3. J. Gambogi: 'Zirconium and hafnium'; 1997, Reston, VA, USGS.
4. H. V. Muijen, and R. A. Massee: Proc. XVII Natl Meet. for Mineral Processing and Extractive Metallurgy, Águas de São Pedro, Brasil, August 1998.
5. DNPM: 'Sumário mineral'; 2002, Brasileiro, DNPM.
6. D. G. W. Smith, L. Jorre, S. J. B. Reed and J. V. P. Long: *Can. Mineral.*, 1991, **29**, 301–309.
7. H. Gorz and E. W. White: *Contrib. Mineral. Petrol.*, 1970, **29**, 180–182.
8. L. A. Hartmann, M. A. Z. Vasconcellos and A. P. Rosa: *Pesquisas*, 1999, **26**, (1), 59–66.
9. S. Sabedot and C. H. Sampaio: Proc. 55th Cong. Anual da ABM, Rio de Janeiro, Brasil, July 2000, ABM, Vol. 1, no. 1, 525–533.
10. S. Sabedot and C. H. Sampaio: Proc. VI Meet. of the Southern Hemisphere on Mineral Techonoly/XVIII Encontro Nacional de

Tratamento de Minérios e Metalurgia Extrativa, Rio de Janeiro, Brasil, May 2001, CETEM/MCT, Vol. 3, 79–82.
11. Walpole and R. H. Myers: 'Probability and statistics for engineers and scientists', 5th edn; 1993, New York, Macmillan Publishing Company.
12. K. E. Haque: *Int. J. Miner. Process.*, 1999, **57**, 1–24.
13. S. W. Kingman and N. A. Rowson: *Miner. Eng.*, 1998, **11**, (11), 1081–1087.
14. B. Wopenka, B. L. Jolliff, E. Zinner and D. T. Krenser: *Am. Mineral.*, 1996, **81**, 902–912.
15. L. A. Hartmann, J. A. D. Leite, L. C. da Silva, M. V. D. Remus, N. J. McNaughton, D. I. Groves, I. R. Fletcher, J. O. Santos and M. A. Z. Vasconcellos: *Aust. J. Earth Sci.*, 2000, **47**, 829–844.
16. L. Nasdala, R. T. Pidgeon and D. Wof: *Geochim. Cosmochim. Acta*, 1996, **60**, (6), 1091–1097.