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Characterization, Classification, Dry High Intensity Magnetic Separation (DHIMS) and Re-Grinding Techniques to Improve the Mineral Performance of a Sn-Ta-Nb Mineral Concentrate

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Abstract: Ta and Nb are considered critical raw materials; due to their properties and potential applications in wide sectors. This study deals with Sn-Ta-Nb minerals from the Penouta mine (Orense, Spain), the only active mine in Europe producing tantalum minerals. These are obtained from mining wastes accumulated during old mining jobs in tailing ponds. The industrial processing flowsheet is based on successive gravimetric stages followed by low intensity magnetic separation to reduce ferromagnetic contaminants. Sn-Ta-Nb concentrate, with grades between 35-45% Sn and 4-7% Ta_2O_5 and Nb_2O_5, is obtained in this stage with plant recoveries around 60-70% respectively. A chemical-mineralogical characterization by size fractions, XRF and XRD was carried out to implement a size classification stage in the processing plant. The finest fractions, containing higher grades of well liberated Sn, Ta, Nb minerals, were the feeding for dry high intensity magnetic separation (DHIMS) multifactorial tests, while, coarse fractions were re-grinded to maximize performance. The good results obtained in these tests demonstrate that two products with commercial quality could be obtained, a cassiterite concentrate with grades between 70-78% SnO_2 and a tantalite-columbite concentrate with grades ranging between 12 and 14% Ta_2O_5 and Nb_2O_5, also increasing the overall recovery of the plant.

Keywords: high intensity magnetic separation; Sn-Ta-Nb; critical raw materials; Penouta mine.

1. Introduction

The modern and competitive economy relies heavily on a range of critical raw materials owing to the risk in their supply and their economic importance [1,2]. Among these critical raw materials are Ta and Nb, with significant applications in the technological and aeronautical fields [3]. These elements are mainly contained in minerals such as columbite and tantalite, which come mainly from the mines of Brazil and Central Africa respectively [2]. These mines are frequently managed by armed groups in conflict areas, so that commercialization of Ta and Nb as well as their forming minerals are regulated to avoid minerals of illegal mines to enter into the supply chain [4]. In order to reduce dependence on imports of Ta and Nb in Europe, it is necessary to increase the investigation of deposits that contain these elements and the processes for their concentration in Europe, within the framework of the Circular Economy [5].

The Penouta mine (Orense, Galicia) is the only active mine in Europe that produces concentrates of Sn, Ta and Nb as main products, from the exploitation of the old tailings ponds and dumps by Strategic Minerals Spain (SMS) [6,7,8]. The industrial processing flowsheet is based on a grinding-classification stage, multiple stages of gravimetric concentration such as a battery of spirals and shaking tables, followed by a low intensity magnetic separation to reduce ferromagnetic impurities such as iron oxides. A primary concentrate of Sn-Ta-Nb is obtained as a product, with grades between 35-45% Sn and 4-7% Ta_2O_5 and Nb_2O_5, and plant recoveries around 60-70% respectively.

This study aims the chemical-mineralogical characterization by size fractions of the Sn, Ta and Nb primary concentrate to study the feasibility of continuing their mineral processing by carrying out operations such as classification by circular vibrating screen and dry high intensity magnetic separation (DHIMS, hereinafter) multifactorial tests. The objective is to explore the possibility of obtaining independent concentrates of Sn and Ta-Nb that could be commercially more competitive, thus contributing to increase grades and the overall recovery at the industrial plant of the Penouta mine.

2. Material and methods

A representative sample of the Sn-Ta-Nb primary concentrate from the Penouta mine were taken to carry out these tests, characterizing chemically and mineralogically by size fraction through sieving and X-ray Fluorescence (XRF, hereinafter) using a Bruker 4 kW set up in the ALS laboratory of the Penouta mine. Three size fractions were obtained, >150; 150/90 and <90 μm. Fractions 150/90 and <90 μm were used for the DHIMS multifactorial assays. The fraction >150 μm was regrinded in order to achieve maximum yields of Sn, Ta and Nb.

DHIMS multifactorial tests were developed using a Felemamg laboratory-scale induced rotor high intensity magnetic separator (*Figure 1*) at Felemamg facilities in Gijón, Spain.

Figure 1 - Induced rotor high intensity magnetic separator. Felemamg facilities in Gijón, Asturias, Spain.

Table 1 shows the operational variables studied by size fractions for the DHIMS tests works. The intensity of the magnetic field was varied, changing the intensity (A) of the electric field, where the maximum intensity is 21,000 G. Subsequently, the non-magnetic products resulting from all the tests were reprocessed again with the DHIMS in order to simulate an industrial equipment with two induced rotors. A first test work with a wide granulometry (150/0 μm, test 1 showed in *Table 1*) was carried out; five test works with the 150/90 μm fraction (tests 2-6) and another five test works with the <90 μm fraction (tests 7-11). Each product obtained, magnetic and non-magnetic, was analysed by XRF to know the grades and yields for the mineral species of interest, thus evaluating the feasibility of implementing a high intensity magnetic separator on an industrial scale in the Penouta mine. Besides, the phase compositions were characterized by X-ray Diffraction analysis (XRD, hereinafter) using a Siemens D5000 (Siemens, Berlin, Germany) apparatus and quantified by the Rietveld method on the tantalite-columbite concentrate <90 μm obtained, in order to identify mineral phases that could be responsible for decreasing grades of Ta and Nb, and then to study the possibility of continuing its processing.

Table 1 - DHIMS assays configuration. Due to SMS data protection, they are considered qualitatively.

Test Nº	Size fraction (μm)	Magnetic field intensity (A)	Roll speed (rpm)
1	150/0	Low	High
2	150/90	Low	High
3	150/90	High	High
4	150/90	High	Low
5	150/90	Low	Low
6	150/90	Changing split inclination	
7	90/0	Low	High
8	90/0	High	High
9	90/0	High	Low
10	90/0	Low	Low
11	90/0	Changing split inclination	

Afterwards, representative samples of the fraction >150 μm of the primary concentrate were prepared to carry out the regrinding test works in a 0.18 m diameter mill, 4.5 l capacity, charged with 7 kg of balls, resulting in 20.74% filling of balls (156 balls of 19.06 mm and 42 balls of 29.72 mm); 1020 g of mineral for 9.3% mineral filling and 75% critical speed (80.23 rpm). Subsequently, the grinded products were chemically and mineralogically analysed by size fraction after five different milling times: 0.5, 2.5, 5, 10 and 15 min, throughout sieving and XRF analysis, respectively. Finally, the evolution of the percentage passing through the 100 μm mesh was studied with respect to time, as this is the liberation size of tantalite [9,10,11], as well as the distribution of the minerals of interest.

3. Results and discussion

3.1. Characterization and classification of the Sn, Ta and Nb primary concentrate.

The results obtained from the assay by size study, as well as the metallic distribution of the elements of interest for the Sn, Ta and Nb primary concentrate , are reported in *Table 2*.

This primary concentrate shows a D_{80} of 99 μm. The calculated feed grades were 33.75% Sn, 5.24% Ta and 4.64% Nb, with significant MnO, Fe_2O_3 and SiO_2contents . It can be noted that Sn, Ta, Nb contents increase with decreasing particle size, so that the highest concentration of these metals are found below 106 μm; on the contrary, MnO, Fe_2O_3, SiO_2, MgO are commonly concentrated in coarser fractions, according to the findings of [10,11]. In order to try to remove these impurities as much as possible from the coarser fractions prior to carrying out the magnetic separation test works, a vibrating circular sieve was used, throughout 150 and 90 μm meshes (*Figure 2)*, thus obtaining three size fractions: > 150, 150/90 and <90 μm. The two finest factions (150/90 and <90 μm) would be the feed for the DHIMS multifactorial test works, while the fraction >150 μm would be regrinded.

Table 2 – Assay by size of Sn, Ta and Nb primary concentrate from the Penouta mine.

Size	Retained weight		Accumulated weight	Grades (%)						
(μm)	(g)	(%)	(%)	Sn	Ta_2O_5	Nb_2O_5	Fe_2O_3	Al_2O_3	MnO	SiO_2
300	0.00	0.00	100.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
212	3.30	0.28	99.2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
150	25.30	2.14	97.58	11.80	2.77	2.58	13.30	14.20	23.50	20.40
106	136.10	11.51	86.07	14.85	3.30	3.23	12.60	12.75	20.80	17.55
63	434.30	36.74	49.32	26.50	5.05	4.99	8.59	7.32	12.75	13.40
45	330.10	27.93	21.40	41.50	6.06	5.13	3.15	1.80	4.64	7.19
38	118.90	10.06	11.34	45.10	5.94	4.69	1.73	0.58	2.32	6.30
0	134.00	11.34	0.00	52.30	5.93	4.19	1.22	0.11	1.44	4.99
Total:	1182.00	100.00	**Calculated feed grade:**	33.75	5.25	4.64	6.08	5.04	9.28	10.59
			***D_{80}* (μm):**	99						

Size (μm)	Distribution of metal content (%)						
	Sn	Ta_2O_5	Nb_2O_5	Fe_2O_3	Al_2O_3	MnO	SiO_2
300	0.00	0.00	0.00	0.00	0.00	0.00	0.00
212	0.00	0.00	0.00	0.00	0.00	0.00	0.00
150	0.75	1.13	1.19	4.68	6.04	5.42	4.12
106	5.07	7.24	8.02	23.85	29.16	25.82	19.08
63	28.85	35.05	39.51	51.88	53.42	50.51	46.50
45	34.34	32.05	30.88	14.46	9.98	13.97	18.96
38	13.44	11.39	10.17	2.86	1.16	2.52	5.99
0	17.57	12.82	10.24	2.27	0.25	1.76	5.34
Total:	100.00	100.00	100.00	100.00	100.00	100.00	100.00

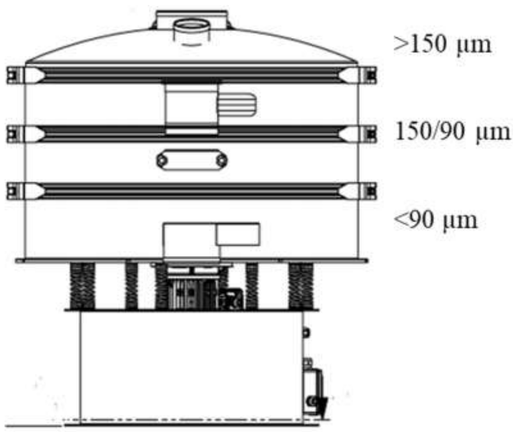

Figure 2 – Scheme of the vibrating circular sieve used to obtain three size fractions: >150, 150/90 and <90 μm.

3.2. Results of laboratory scale DHIMS multifactorial test works

3.2.1. Grades and recoveries of the new concentrates obtained.

The fractions 150/90 and <90 μm obtained in the previous classification stage were selected for the DHIMS multifactorial test works. Two products are obtained after DHIMS performance, a magnetic concentrate of columbo-tantalite and a non-magnetic concentrate of cassiterite, grades and recoveries are reported in *Table 3* and *Table 4*, respectively, for each fractions and variables considered in *Table 1*.

Table 3 shows that the best grades of Ta_2O_5 and Nb_2O_5 in the tantalite-columbite concentrate were obtained with test work #6 for the 150/90 µm fraction, reaching grades of 11.71% Ta_2O_5 and 12.21% Nb_2O_5; while, test work #7 was the best configuration for the <90 µm fraction, overtaking grades of 13.66% Ta_2O_5 and 13.21% Nb_2O_5. Specifically, test work #7 supposed a better Ta_2O_5 grade even than #1, carried out with a wider size 150/0 µm.

Table 3 - Tantalite-columbite concentrate yields resulting from DHIMS test works. G: grades; R: recovery.

Mineral	Ta_2O_5		SnO_2		Nb_2O_5		SiO_2		Fe_2O_3		MnO	
Test nº	G	R	G	R	G	R	G	R	G	R	G	R
	(%)		(%)		(%)		(%)		(%)		(%)	
1	12.20	57.62	7.10	2.95	12.65	76.76	8.97	30.87	8.74	65.87	12.71	79.18
2	10.33	68.18	3.34	2.09	11.10	89.39	13.44	52.81	12.01	83.44	17.36	96.14
3	10.15	68.09	3.33	2.11	11.02	88.59	13.83	55.02	12.13	83.38	17.51	95.22
4	8.80	69.82	26.99	19.18	8.79	85.61	9.41	49.87	7.62	78.34	10.84	89.55
5	8.79	68.96	28.09	19.82	8.78	85.97	9.31	50.78	7.50	78.38	10.66	90.97
6	11.71	70.11	7.29	3.87	12.21	91.88	10.13	42.86	9.47	80.89	13.86	97.86
7	13.66	67.78	10.09	4.23	13.21	89.42	6.30	25.49	6.83	71.40	10.04	92.31
8	13.47	67.80	10.77	4.61	13.61	90.95	6.15	24.76	6.65	70.24	9.75	91.11
9	8.89	76.07	39.01	28.59	8.23	91.63	5.47	36.89	4.37	76.75	6.01	94.01
10	9.03	76.34	37.93	26.69	8.42	94.08	5.38	35.81	4.38	77.93	6.03	95.78
11	13.13	68.24	13.65	5.93	13.24	91.15	5.36	22.51	5.89	69.52	8.69	90.58

Table 4 - Cassiterite concentrate yields resulting from DHIMS test works. G: grades; R: recovery.

Mineral	Ta_2O_5		SnO_2		Nb_2O_5		SiO_2		Fe_2O_3		MnO	
Test nº	G	R	G	R	G	R	G	R	G	R	G	R
	(%)		(%)		(%)		(%)		(%)		(%)	
1	1.84	27.12	75.06	97.51	0.41	7.78	6.56	70.89	0.62	14.61	0.13	2.53
2	2.01	26.37	75.57	93.73	0.58	9.25	6.36	49.62	0.84	11.59	0.29	3.19
3	1.95	25.00	76.96	93.40	0.49	7.50	6.34	48.28	0.72	9.47	0.21	2.19
4	2.13	24.93	73.91	77.48	0.78	11.18	6.56	51.28	0.88	13.35	0.51	6.21
5	2.15	25.13	73.91	77.61	0.80	11.67	6.58	53.42	0.86	13.37	0.52	6.61
6	1.89	25.76	78.23	94.39	0.41	7.07	6.27	60.37	0.61	11.86	0.12	1.93
7	1.85	26.81	76.58	93.96	0.37	7.13	6.67	78.87	0.57	17.43	0.11	2.96
8	1.85	26.93	77.34	95.57	0.38	7.26	6.70	77.81	0.58	17.67	0.11	2.97
9	1.65	18.67	74.17	71.30	0.35	5.12	7.52	67.25	0.53	12.35	0.11	2.28
10	1.65	19.17	74.68	72.33	0.34	5.21	7.63	69.90	0.54	13.23	0.10	2.19
11	1.79	26.26	75.57	92.72	0.36	6.96	6.73	79.90	0.55	18.36	0.10	2.95

Table 3 also shows that the best recoveries for the 150/90 µm fraction resulted from #6, with 70.11% and 91.88% of Ta_2O_5 and Nb_2O_5, respectively; in contrast, #10, with recoveries of 76.34% and 94.08% of Ta_2O_5 and Nb_2O_5, respectively, was the best configuration for the <90 µm fraction. Such results are accordingly with the liberation size below 100 µm reported for the tantalite [9,10,11], thus demonstrating the influence of the size particle on the increase of the grades and the recoveries of the species. Furthermore, the fact of a higher content of SiO_2, Fe_2O_3 and MnO in the tantalite-columbite concentrate in coarser granulometry stands out [12].

Regarding the non-magnetic cassiterite concentrate, the best grades and recoveries of SnO_2 were obtained in #6 and #8 test works, for the 150/90 and <90 µm fractions respectively, reaching grades of 78.23% and 77.34% SnO_2 and recoveries of 94.39% and 95.57% SnO_2, respectively (*Table 4*). Therefore, #6 test work is considered the optimal configuration for the 150/90 µm fraction, since it shows the best results for both grades and recoveries of SnO_2 and Ta_2O_5. Whereas, the fraction <90 µm through the configuration of #7 shows the best results to favour the increase of Ta_2O_5 and Nb_2O_5 grades and one of the best for SnO_2.

The XRD pattern of the tantalite-columbite concentrate <90 µm *(Figure 3)* shows the presence of minerals such as xenotime, cassiterite, aluminum silicates of Mn, goethite and zircon as main impurities associated with the predominant tantalite-columbite (Mn)[12]. These results complement the information obtained by XRF (*Table 3)*, confirming that elements such as Mn and Fe are intrinsically associated with the mineralogy of tantalite-columbite, indeed. Consequently, their physical separation is impossible.

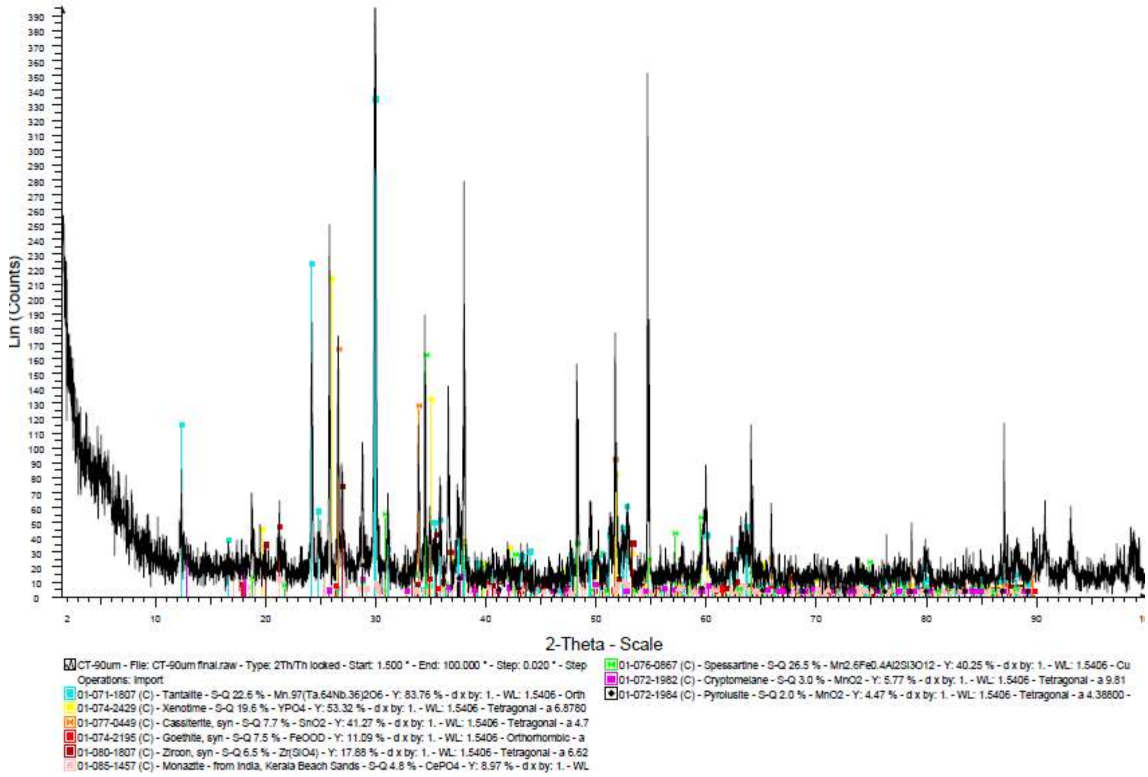

Figure 3 - XRD pattern of the tantalite-columbite concentrate sample <90 µm.

3.2.2. Influence of operational parameters in DHIMS test works.

According to the results shown in *Table 3-4*, a higher roller speed seems to favour greater recoveries not only for the cassiterite in the non-magnetic product, but also for tantalite-columbite in the paramagnetic product obtaining good grades, as a better separation of the streams occurs, then allowing the adjustment of the split in order to increase the cassiterite grades in the non-magnetic product and recovery of tantalite in the magnetic product.

Regarding the intensity of the magnetic field, as the particle size is smaller a greater intensity is necessary than with a coarser granulometry for the same roller speed. Likewise, the results suggest that a finer particle size favours both the grades and the recovery of Ta_2O_5 and SnO_2.

In summary, when the feed is classified in two fractions, the selectivity of the operation increases, obtaining better results than for larger size fractions such as 150/0 µm.

3.3. Regrinding of >150 µm fraction.

The assay by size of the resulting products passing the 100 µm mesh, after regrinding of the >150 µm fraction for each grinding time (0.5, 2.5, 5, 10 and 15 min) are shown in *Table 5*.

Table 5 - Evolution of the P_{80} with respect to time and assay by size results on the regrinded >150 µm fraction.

Time (min)	Pass weight through 100 µm (%)	P_{80} (µm)	Distribution of metal content (%)						
			Sn	Ta_2O_5	Nb_2O_5	Al_2O_3	Fe_2O_3	MnO	SiO_2
0.5	11.20	218	26.26	21.07	20.44	5.59	7.45	5.45	7.13
2.5	17.60	197	38.25	30.81	29.66	8.77	11.67	8.91	11.39
5.00	22.40	192	43.74	36.57	34.43	12.93	16.53	13.13	15.60
10.0	30.90	185	51.09	46.81	43.13	20.75	26.47	22.14	23.05
15.00	32.90	184	50.39	47.47	43.93	24.44	29.89	25.54	26.49

Table 5 shows that as grinding time increases, the amount of milled material passing through the 100 µm mesh increases. The P_{80} with respect to time highlights the little difference between 10 to 15 min, suggesting the optimum time as 10 min for grinding to avoid regrinding of fines and loss of energy efficiency. Besides, the distribution of the metal content in the species of interest of the milled product passing through the 100 µm mesh after the milling time shows an increase, reaching a higher liberation of at least 50% in the species of interest such as Sn, Ta and Nb after 10 min.

4. Conclusions

From the present research it is concluded that by implementing a size classification, better grades and recoveries of Sn, Ta and Nb are obtained when performing DHIMS instead of using a wide granulometric interval (150/0 µm), since selectivity increases during operation. Consequently, a circular vibrating screen was implemented to generate three size fractions; the finest fractions (150/90 and <90 µm) were used as the feed of DHIMS multifactorial test works; the coarsest fraction (>150 µm) was regrinded in a closed-circuit ball mill installed at the Penouta mine plant.

The multifactorial DHIMS test works on the Sn, Ta and Nb primary concentrate generated two new products, a magnetic columbo-tantalite concentrate and a non-magnetic cassiterite concentrate with better grades, being therefore rather commercially competitive, and then leading to better profitability and feasibility for the Penouta mine project. The results for this test works suggest that a finer particle size favours both the grade and the recovery of Ta_2O_5, Nb_2O_5 and SnO_2, hence, it is key to keep control on the grinding-classification circuit; maintain the D_{80} of the primary concentrate and a better liberation of the mineral species of interest, in order to reproduce the results obtained in the present investigation.

The present work proposes to conduct electrostatic separation multifactorial tests to cut down on minerals such as zircon, monazite and xenotime and to purify the paramagnetic and non-magnetic concentrates obtained; as well as passing the tantalite-columbite concentrate thorough the DHIMS to increase the grades of Ta_2O_5 and Nb_2O_5. Regarding the regrinding test works on the fraction >150 µm, the optimum time was 10 min to obtain a well liberation for the mineral species of interest Sn, Ta and Nb.

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