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Mineralogical factors affecting the separation behavior of Ta-Nb-bearing minerals in the gravity field: mineral grain size, liberation, and association relationship

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Abstract: Gravity separation is the primary method used to beneficiate Ta-Nb-bearing minerals, however, it performs poorly in low-grade and fine-grained ores. A comparative study of gravity separation products (concentrate, middlings, and tailings) reveals the factors affecting the separation behavior of Ta-Nb-bearing minerals in the process combined with a spiral chute and shaking table from the perspective of mineralogy. The results reveal that columbite-tantalite is the principal Ta-Nb-bearing mineral. As the grinding time increases, the grade of Ta and Nb in concentrate increases significantly. The grain size of columbite-tantalite in the concentrate is the coarsest, followed by that in the middlings, and the finest in the tailings, which are mainly distributed in the range of -150+38 μm, -75+20 μm, and -38 μm, respectively. The liberation degree of columbite-tantalite in the concentrate and tailings is positively correlated with grinding time, while that in the middlings is negatively correlated with grinding time. The density of columbite-tantalite-bearing particles in concentrate is mainly distributed above 3 or even 4, due to the high liberation degree of the columbite-tantalite in the concentrate, as well as the high amount of rich intergrowth associated with heavy minerals. The density of Ta-Nb-bearing mineral particles in the middlings and tailings is predominantly distributed in D<3, owing to columbite-tantalite mainly associated with lighter gangue minerals such as quartz, albite, and orthoclase. It demonstrates that the liberation degree is not the most essential factor in determining columbite-tantalite separation behavior in the gravity field, and the mineralogical characteristics of columbite-tantalite including grain size, association relationship, and particle density, may be more important. The results of this investigation can provide theoretical support for the strengthening separation of low-grade tantalum-niobium ore.

Keywords: separation behavior, gravity field, mineralogical characteristic, columbite-tantalite, association relationship, liberation

1. Introduction

Niobium and tantalum have the characteristics of good heat resistance, high melting point, good ductility, strong corrosion resistance, and high thermal conductivity. They are crucial raw materials for the chemical, aerospace, steel, electronics, atomic energy, and other sectors. In the realms of energy, advanced technology, and medical care, they are indispensable (Linnen et al., 2014; Schulz et al., 2017b). Niobium and tantalum are consequently listed as critical metals in China, Japan, the United States, and the European Union (Gulley et al., 2018; Mathieux et al., 2017; Schulz et al., 2017a; Zhai et al., 2019).

The beneficiation of tantalum-niobium ore generally involves roughing and cleaning(Li et al., 2023). The density of main valuable minerals in tantalum-niobium ore is above 4×10^3 kg/m^3, and the density of gangue minerals is relatively low (Bulatovic, 2010). Gravity separation allows for the removal of the

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majority of the gangue minerals, yielding low-grade mixed crude concentrate. The separation is challenging due to the mixed crude concentrate's complicated mineral composition, which often contains a range of valuable minerals(Nzeh et al., 2022a; Nzeh et al., 2022b). To obtain qualified tantalum and niobium concentrate products, several mineral processing techniques, including gravity separation, flotation, magnetic separation, and other procedures, are typically utilized (Bale and May, 1989; Lv et al., 2010; Nheta and Ruwizhi, 2019).

Currently, gravity separation is the primary method used to beneficiate tantalum niobium ore (Ghorbani et al., 2017). The devices utilized in the gravity separation of tantalum-niobium ore include shaking table, spiral chute, jigging, etc. However, gravity separation performs poorly in low-grade and fine-grained ores (Ghorbani et al., 2017; Lv et al., 2022; Lv et al., 2012). It is necessary to determine the process mineralogy reason for the poor gravity recovery rate of tantalum niobium ore. Research on the process mineralogy of tantalum niobium ore currently focuses primarily on the state of occurrence of useful elements such as tantalum and niobium (Habinshuti et al., 2021; Lv et al., 2022; Yuan et al., 2015), and the dissemination characteristics of main occurrence minerals(Meirone et al., 2021), but there is not enough research on the microscopic characteristics of Ta-Nb-bearing mineral particles. The correlation between the separation behavior of particles and their mineralogical characteristics such as grain size, liberation, and association relationship is still unclear(El-Midany et al., 2011; Zhou et al., 2021).

The specific purpose of this study is to identify the mineralogical factors affecting the separation behavior of Ta-Nb-bearing minerals from the typical granite-type tantalum-niobium ore samples in Jiangxi Province in the gravity field. The separation behavior of Ta-Nb-bearing mineral in a gravity field was investigated by adopting the process combined with a spiral chute and shaking table. The separation mineralogical characteristics of Ta-Nb-bearing minerals in the gravity field were studied employing an optical microscope, Mineral Liberation Analyze (MLA), chemical analysis, X-ray diffraction analysis, and electron probe(Huang et al., 2022). And the corresponding relationship between the microscopic characteristics of mineral particles and the separation behavior was obtained, which can provide theoretical support for the strengthening separation of low-grade tantalum-niobium ore.

2. Materials and methods

2.1. Materials

The tantalum-niobium ore sample utilized in the current experiments was acquired from the Yichun Tantalum Niobium Mine in Jiangxi Province, China. For the experimental study, a representative sample of about 500 kg was chosen, of which about 50 kg was used for characterization and testing.

Inductively coupled plasma-atomic emission spectroscopy (Intrepid II XSP, Thermo Electron, USA) was used to determine the chemical composition of the raw ore. Table 1 displays the chemical compositions and contents of the ore. The contents of Ta_2O_5, Nb_2O_5, Li_2O, and Rb_2O are 160ppm, 83ppm, 1.03%, and 0.36%, respectively. Table 2 displays the mineral composition and content of the ore sample as determined by methods including optical microscopy and mineral liberation analysis (MLA 650F). As can be observed from Table 2, the main minerals in the ore are albite (49.10%), quartz (20.58%), and orthoclase (19.48%), followed by lepidolite (5.12%), topaz (1.81%), and hornblende (1.71%). The major mineral that contains Ta and Nb is columbite-tantalite, which has a 0.015% concentration. Gangue minerals are mainly feldspar(albite and orthoclase) and quartz, accounting for nearly 90% of the total mineral content.

Table 1. Chemical analysis of ore samples

Component	Ta_2O_5*	Nb_2O_5*	Li_2O	Rb_2O	SiO_2	Al_2O_3	Na_2O	K_2O	CaO
Content wt.%	160	83	1.03	0.36	69.25	17.42	5.88	2.67	0.31
Component	MnO	MgO	P_2O_5	TFe	Cs_2O	SnO_2	TiO_2	ZnO*	U_3O_8*
Content wt.%	0.13	0.025	0.35	0.14	0.15	0.011	0.013	62	10

* ppm

Table 2. Mineral composition and content of the ore

Mineral	Content wt.%
Columbite-tantalite	0.015
Microlite	0.008
Cassiterite	0.02
Quartz	20.58
Albite	49.10
Orthoclase	19.48
Lepidolite	5.12
Hornblende	1.71
Topaz	1.81
Grossularite	0.49
Apatite	0.32
Calcite	0.31
Variscite	0.21
Magnetite	0.13
Limonite	0.10
Goyazite	0.04
Zircon	0.01
Biotite	0.01
Others	0.537
Total	**100.00**

The mineral characteristics analysis of raw ore and mineral particles mentioned below was conducted by methods including optical microscopy analysis, and Mineral Liberation Analyzer (MLA 650F). Optical microscope analysis was conducted on ZEISS Axioskop 40. The EPMA-1720 (Shimadzu) was used for electron probe microanalysis, with a beam current of 10 nA and an accelerating voltage of 20 kV.

2.2. Gravity separation test

The gravity separation procedure of a spiral chute and a shaking table was employed to separate the raw ore in order to study the separation behavior of columbite-tantalite in the gravity field. Fig. 1 depicts the gravity separation procedure. A three-roll, four-cylinder rod mill was used for grinding, and the grinding concentration was selected to be a solids ratio of 66.7%, and the mill model was XMB-70.

3. Results and discussion

3.1. Mineralogical characteristic of raw ore

Table 3 displays the modes of occurrence of tantalum and niobium as well as the distribution of these elements in different minerals. According to Table 3, tantalum and niobium primarily occur as columbite-tantalite and microlite and are dispersed throughout cassiterite, lepidolite, feldspar (albite and orthoclase), and quartz. Among them, tantalum concentrates on distributing in columbite (45.95%), microlite (39.52%), and cassiterite (4.88%), whereas niobium prefers to disperse in columbite (65.69%). The most significant tantalum-niobium mineral in the ore is columbite-tantalite, which is also the most valuable metallic mineral. The separation of columbite-tantalite directly affects the recovery index of tantalum and niobium elements. Therefore, the dissemination characteristic of columbite-tantalite was investigated. The grain size distribution and dispersion form of columbite-tantalite are shown in Fig. 2 and Table 4, respectively. As illustrated in Fig. 2, the grain size of columbite-tantalite is mainly

Table 3. The modes of occurrence of tantalum and niobium, and their distribution

Mineral	Content wt.%	Ta_2O_5 wt.%		Nb_2O_5 wt.%	
		Grade	Distribution rates	Grade	Distribution rates
Columbite-tantalite	0.015	46.79	45.95	34.28	65.69
Microlite	0.008	75.46	39.52	1.58	1.61
Cassiterite	0.02	3.73	4.88	1.38	3.53
Lepidolite	5.12	0.0096	3.22	0.015	9.81
Feldspar and Quartz	89.16	0.0011	6.42	0.0017	19.36
Total	94.323	0.0162	100.00	0.0083	100.00

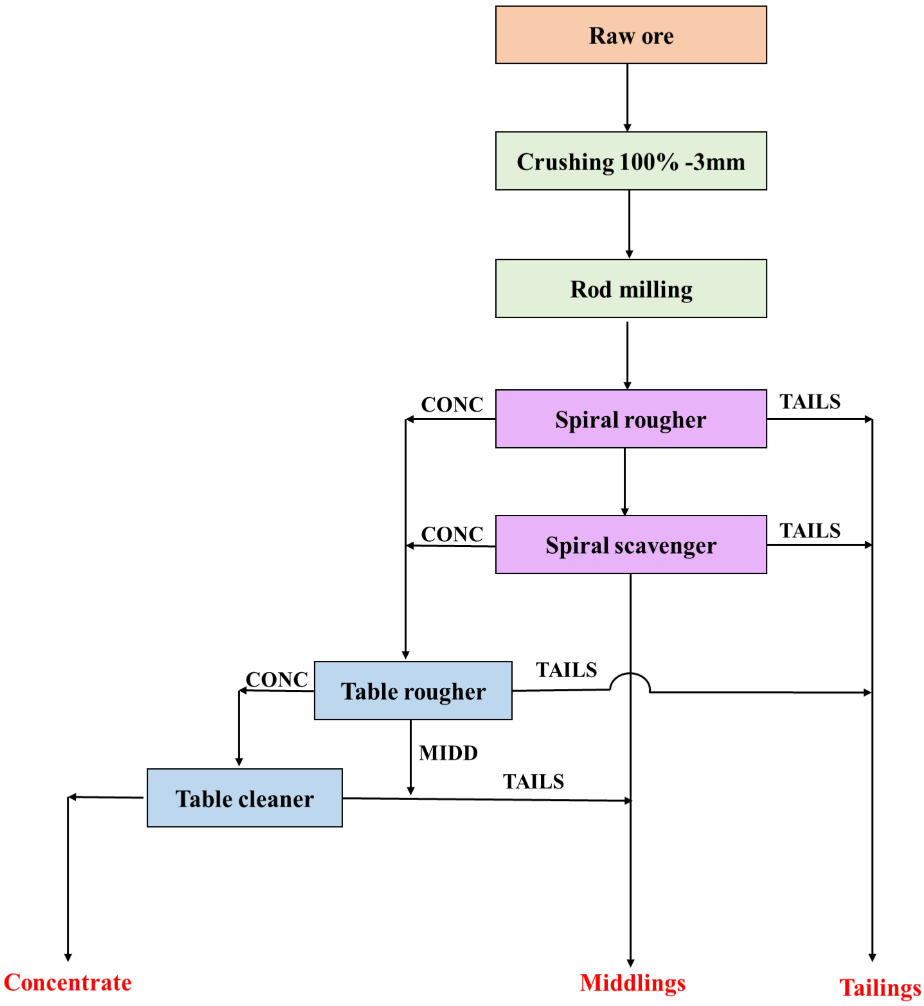

Fig. 1. The procedure for gravity separation of tantalum-niobium ore

distributed in -0.3+0.045 mm. Table 4 indicates that columbite-tantalite has two disseminated forms, the most common being interparticle distribution, which accounts for 54.23 percent of the total, and inclusion distribution, which represents 45.77 percent. The interparticle dissemination forms of columbite-tantalite are various; they are generally spread between two or three mineral particles composed of albite, lepidolite, topaz, orthoclase, and quartz, with a few between gangue minerals and biotite or variscite particles, as shown in Fig. 3. As seen in Fig. 4, the inclusion part is mainly embedded in albite, followed by quartz, orthoclase, and lepidolite, and a small amount is encapsulated by topaz.

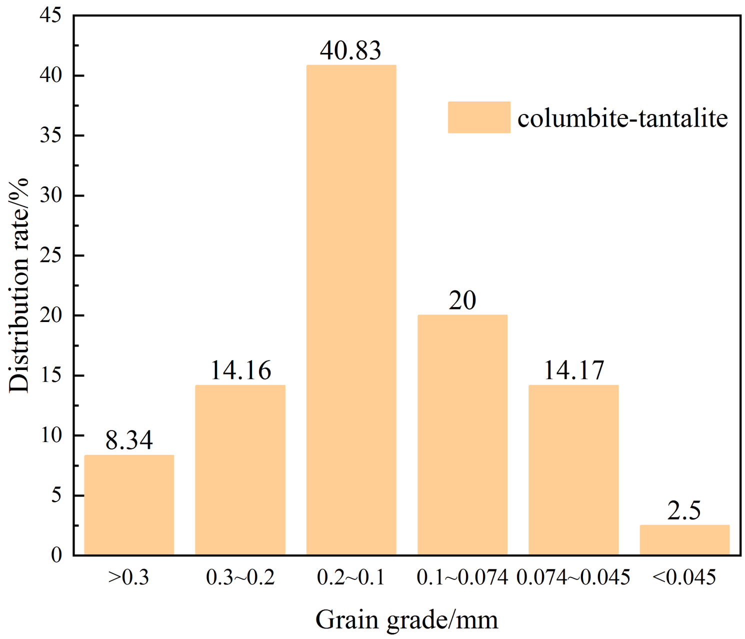

Fig. 2. The grain size distribution of columbite-tantalite

Table 4. The dissemination form of columbite-tantalite

Dissemination form	Mineral	Content %	Total (%)
Intergranular distribution	Quartz	6.11	54.23
	Albite	9.56	
	Albite and quartz	14.33	
	Albite and lepidolite	9.59	
	Albite and topaz	5.40	
	Albite and Orthoclase	3.83	
	Albite and grossularite	3.60	
	Apatite and biotite	1.45	
	Apatite and variscite	0.36	
Inclusion distribution	Albite	17.49	45.77
	Quartz	14.49	
	Orthoclase	11.03	
	Lepidolite	2.27	
	Topaz	0.49	
Total (%)	/	100.00	100.00

3.2. The separation behavior of Ta-Nb-bearing mineral in the gravity field

The separation behavior of Ta-Nb-bearing mineral in the gravity field was investigated by adopting the process of Fig. 1 and the results are shown in Table 5. The particle size distribution of feeding at different grinding time is shown in Fig. 5 (a). It indicates that the d80 of the feeding is 245μm, 191μm and 166μm under the grinding conditions of 3min, 5min and 7min, respectively. Table 5 indicates that the grade of tantalum-niobium concentrate increased significantly with the increase of the grinding time. When the grinding time increased from 3min to 5min, the grades of Ta_2O_5 and Nb_2O_5 in the concentrate were increased from 440 ppm and 213 ppm to 2,116 ppm and 1,555 ppm, respectively, and the enrichment ratio was increased from 2.75 to 13.22. The particle size distribution of gravity products under different grinding time is shown in Fig. 5 (b)~Fig. 5(d). The results show that there is no significant difference in particle size distribution between concentrate and middlings. The particle size in the tailings product is finer, especially the -38μm particle size mainly enters into the tailings product.

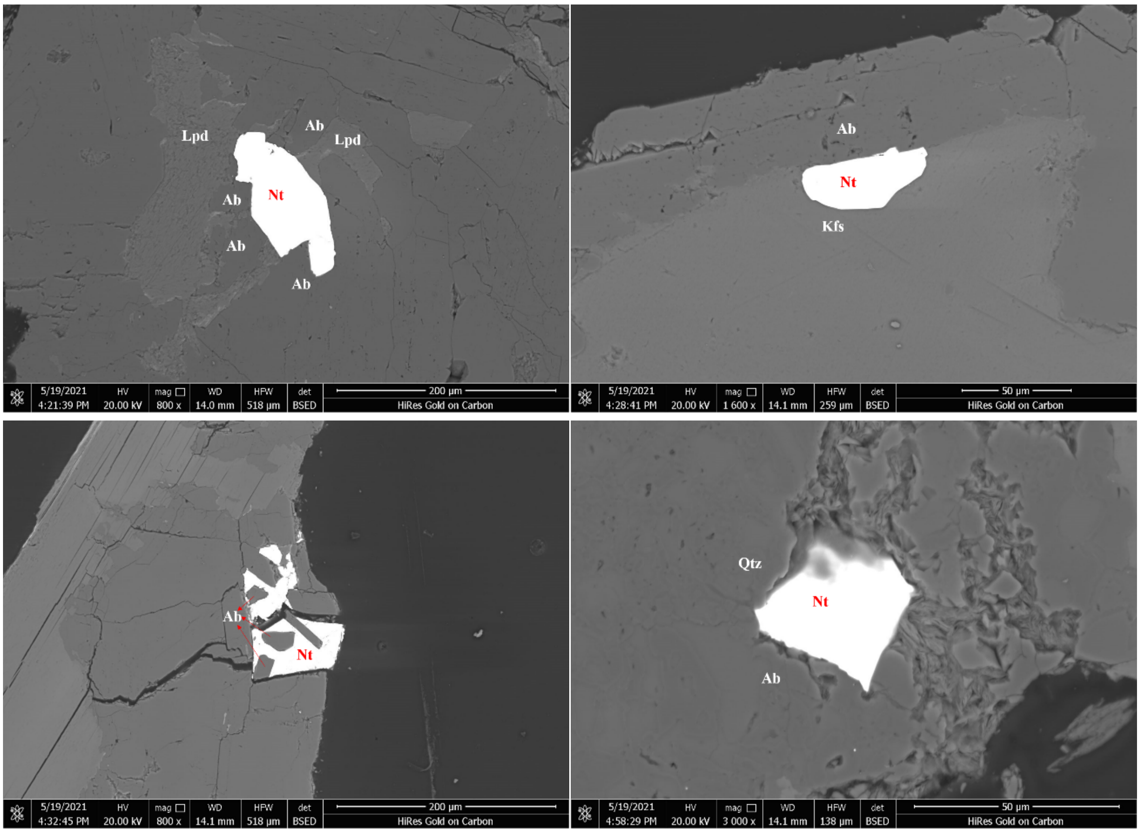

Fig. 3. SEM images of the intergranular distribution of minerals of columbite-tantalite (Nt– Columbite-tantalite, Ab– Albite, Lpd– Lepidolite, Qtz– Quartz, Kfs– Orthoclase)

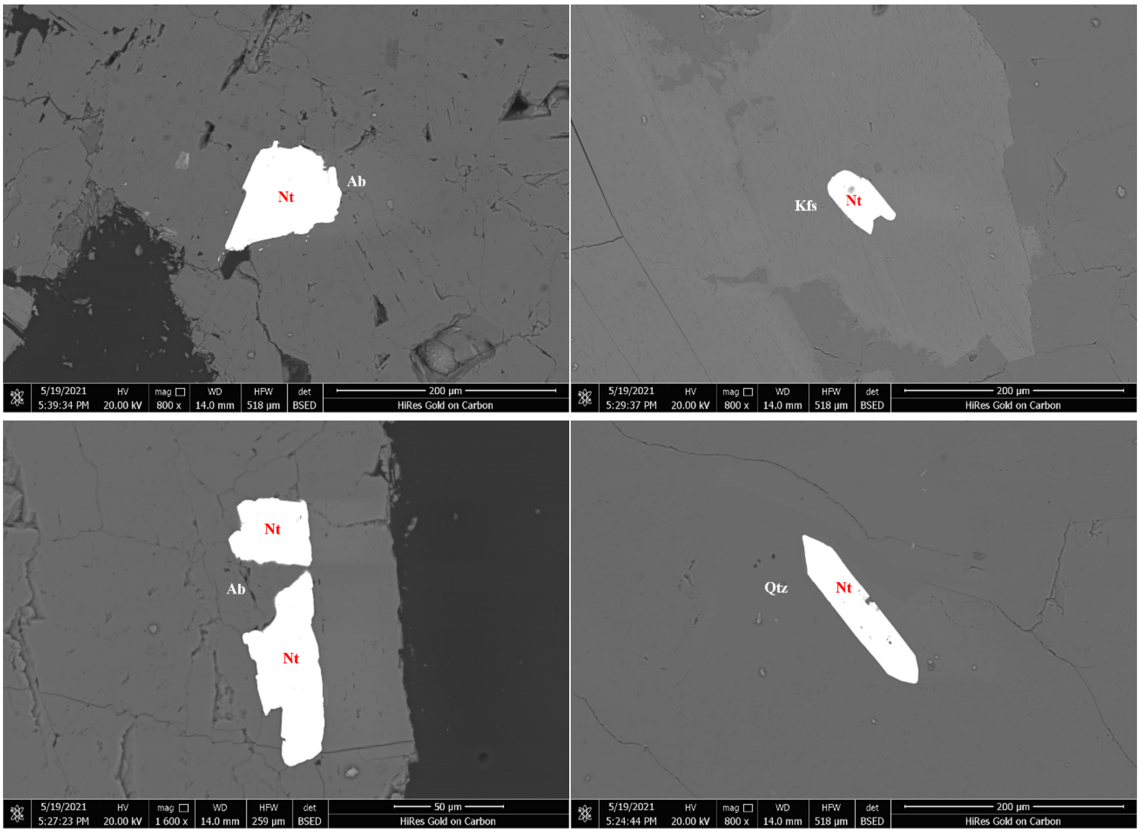

Fig. 4. SEM images of columbite-tantalite distributed as inclusions (Nt– Columbite-tantalite, Ab– Albite, Qtz– Quartz, Kfs– Orthoclase)

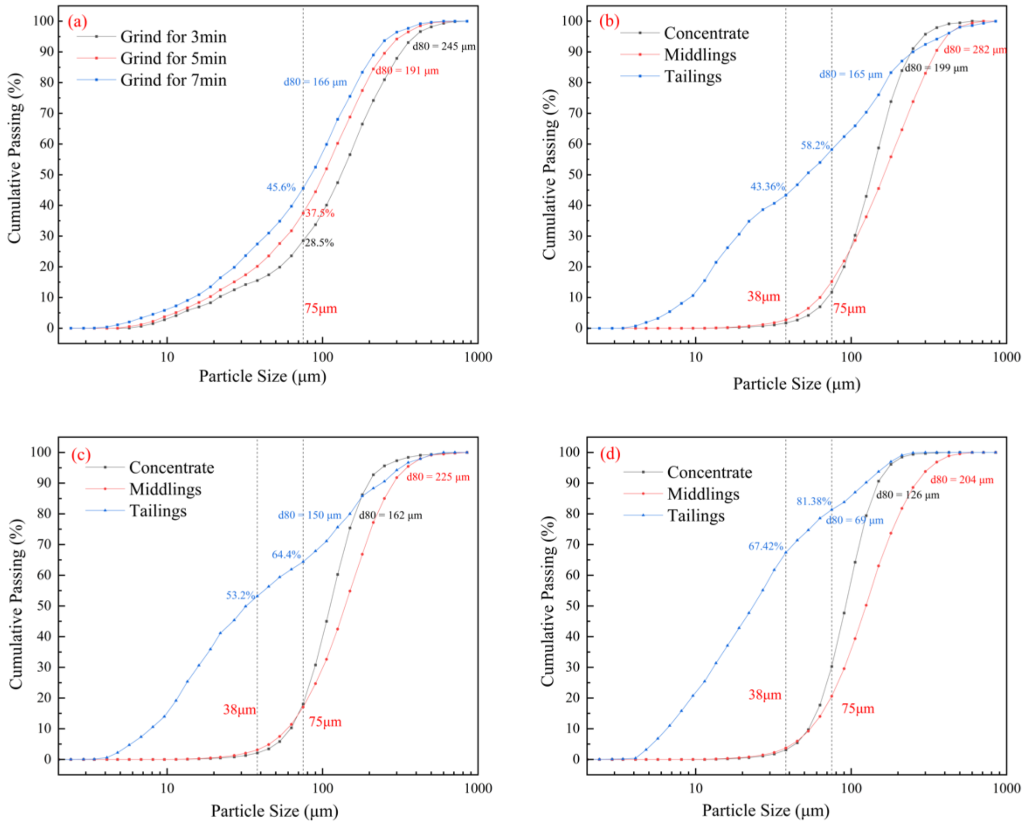

Fig. 5. The particle size distribution of feeding and gravity separation products at different grinding times: (a) Particle size distribution of feeding; (b) Particle size distribution of separation products after grinding for 3min; (c) Particle size distribution of separation products after grinding for 5min; (b) Particle size distribution of separation products after grinding for 7min

Table 5. The results of gravity separation of tantalum-niobium minerals

Grinding time/min	Product	Yield/ %	Grade/ppm		Recovery/%	
			Ta_2O_5	Nb_2O_5	Ta_2O_5	Nb_2O_5
3min d80 = 245 μm	Concentrate	7.10	440	213	22.64	18.88
	Middlings	70.35	120	77	61.18	67.61
	Tailings	22.55	99	48	16.18	13.51
	Feed	100.00	138	80	100.00	100.00
5min d80 = 191 μm	Concentrate	2.54	1638	861	30.52	27.88
	Middlings	68.50	104	64	52.27	55.88
	Tailings	28.96	81	44	17.21	16.24
	Feed	100.00	136	78	100.00	100.00
7min d80 = 166 μm	Concentrate	1.34	2116	1555	21.21	30.33
	Middlings	72.97	112	48	61.11	50.97
	Tailings	25.69	92	50	17.68	18.70
	Feed	100.00	134	69	100.00	100.00

3.3. The mineralogical characteristics of gravity separation products

In order to further investigate the influence of mineralogical factors on the separation behavior of Ta-Nb-bearing mineral in the gravity field, the mineral composition of gravity separation products, the grain size of columbite-tantalite, the liberation degree of columbite-tantalite, and the association characteristics of columbite-tantalite were studied, respectively(Engström, 2010; Marion et al., 2018).

3.3.1. The mineral composition of gravity separation products

The mineral composition of gravity separation products is shown in Fig. 6. The results indicate that when the grinding time is 3min, 5min, and 7min, the content of columbite-tantalite in the concentrate is 683ppm, 7644ppm and 12486ppm respectively. The main minerals in the concentrate are quartz, albite, and orthoclase, mainly because the content of these minerals in the raw ore is high, and columbite-tantalite is closely combined with them. Besides, the topaz content in the concentrate is considerable, up to 36.75%, which can be easily introduced into the concentrate product due to the high specific gravity of topaz and its close embedding connection with columbite-tantalite. The content of columbite-tantalite in the middlings decreased significantly, and the main minerals were quartz, albite, and orthoclase. The content of columbite-tantalite in the tailings is further reduced, and the content of columbite-tantalite increases when grinding for 7 min. It may be that the grain size of columbite-tantalite is too fine and lost in the tailings.

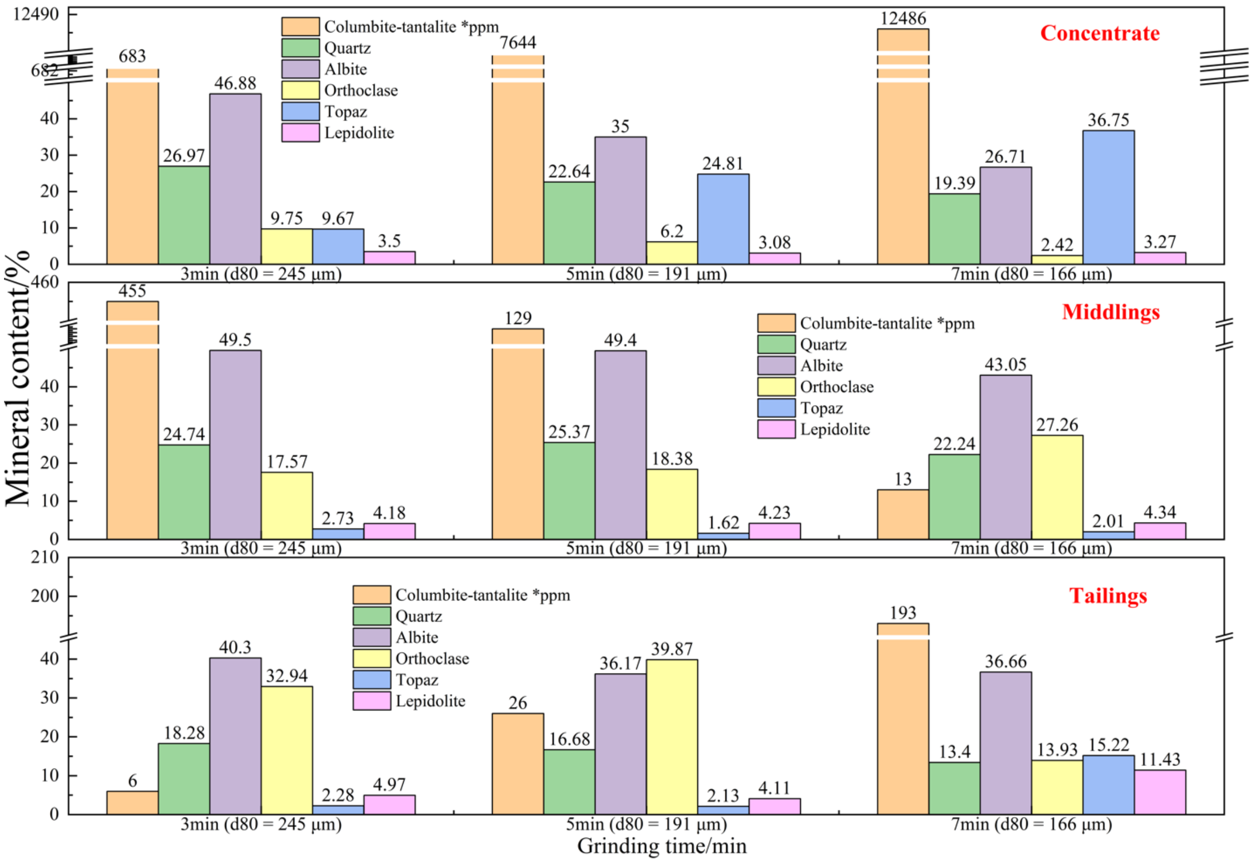

Fig. 6. The mineral composition of gravity separation products

3.3.2. The grain size of columbite-tantalite in gravity separation products

Fig. 5 shows that the -38 μm particles mainly enter the tailings, and there is almost no -38 μm particle size in the middlings and concentrate, indicating that the performance of gravity separation on -38 μm is unsatisfactory. Most of the particles in the middlings and concentrate are above 75 μm, and the particles in the middlings are even coarser than those in the concentrate. In order to further examine the mineralogical characteristics of Ta-Nb-bearing mineral particles in each gravity separation product, we studied the grain particle size distribution of columbite-tantalite in gravity separation products. Fig. 7 depicts the columbite-tantalite grain size distribution in gravity separation products. The grain size of columbite-tantalite in concentrate is the coarsest, followed by that in middlings, and the finest in tailings. The grain size of columbite-tantalite in concentrate is mostly -150+38 μm. The grain size of columbite-tantalite in middlings is primarily dispersed -75+20 μm. The grain size of columbite-tantalite in tailings is almost distributed in -38 μm. It can be observed that the grain size of columbite-tantalite is a crucial factor influencing its behavior in the gravitational field (Cao et al., 2020; Rajak et al., 2022).

In order to clarify whether the loss of columbite-tantalite mineral in tailings is due to particle size and gravity device suitability or other reasons. We studied the particle size distribution of mineral intergrowth particles containing columbite-tantalite in tailings, as shown in Fig. 8(a). It suggests that the particle size of particles containing columbite-tantalite in tailings is rather coarse, with the majority being larger than 75 μm. The grain size of columbite-tantalite in tailings is less than 38 μm, as shown in Fig. 7. This is because they are not completely liberated but are connected with other minerals, resulting in a relatively high particle size of the whole intergrowth particle. It illustrates that the loss of this portion of columbite-tantalite in tailings may be attributable to factors other than particle size and gravity device suitability. Fig. 5 demonstrates that some +38 μm particles enter the tailings. The mineral composition in Fig.6 shows that the primary minerals in the tailings are quartz, albite, and orthoclase. The grain size distribution of quartz, albite, and orthoclase in the tailings is illustrated in Fig. 8(b)~Fig. 8(d), indicating that some coarse-grained quartz and feldspar entered the tailings.

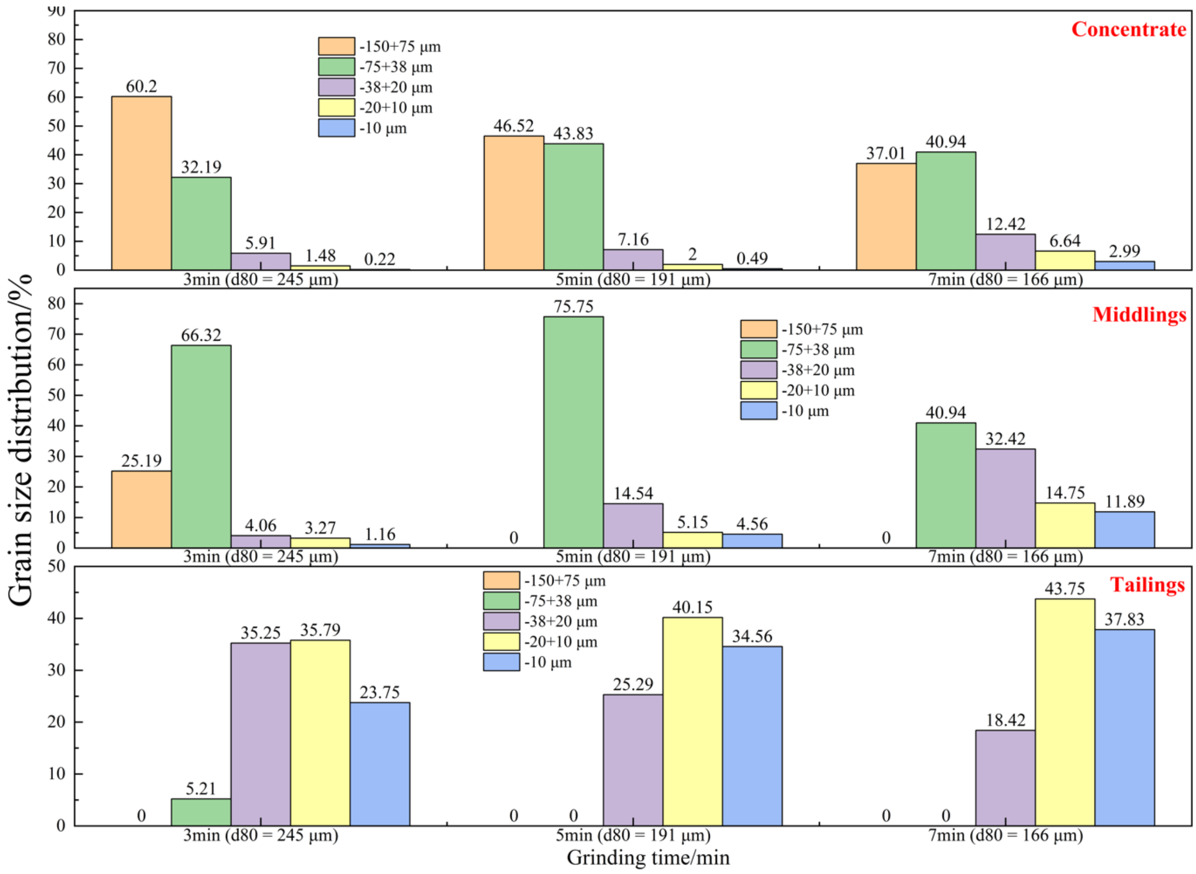

Fig. 7. The columbite-tantalite grain size of gravity separation products

3.3.3. The liberation degree of columbite-tantalite in gravity separation products

The liberation degree of columbite-tantalite in gravity separation products is shown in Fig. 9. It shows that the liberation degree of columbite-tantalite in the concentrate and tailings gradually increases with the increase of grinding time, while the liberation degree of columbite-tantalite in the middlings gradually decreases. This is due to the fact that the middlings contain a large number of intergrowths, which will either enter the concentrate or the tailings after grinding and liberation. In addition, it can be seen that the liberation degree of columbite-tantalite in the tailings is similar to that in the concentrate when the grinding time is 5min and 7min. Combined with the analysis in Fig. 7, it can be seen that it is because the liberated fine-grained columbite-tantalite enters the tailings. It demonstrates that the liberation degree is not the most essential factor in determining columbite-tantalite separation behavior and that the grain size is equally relevant.

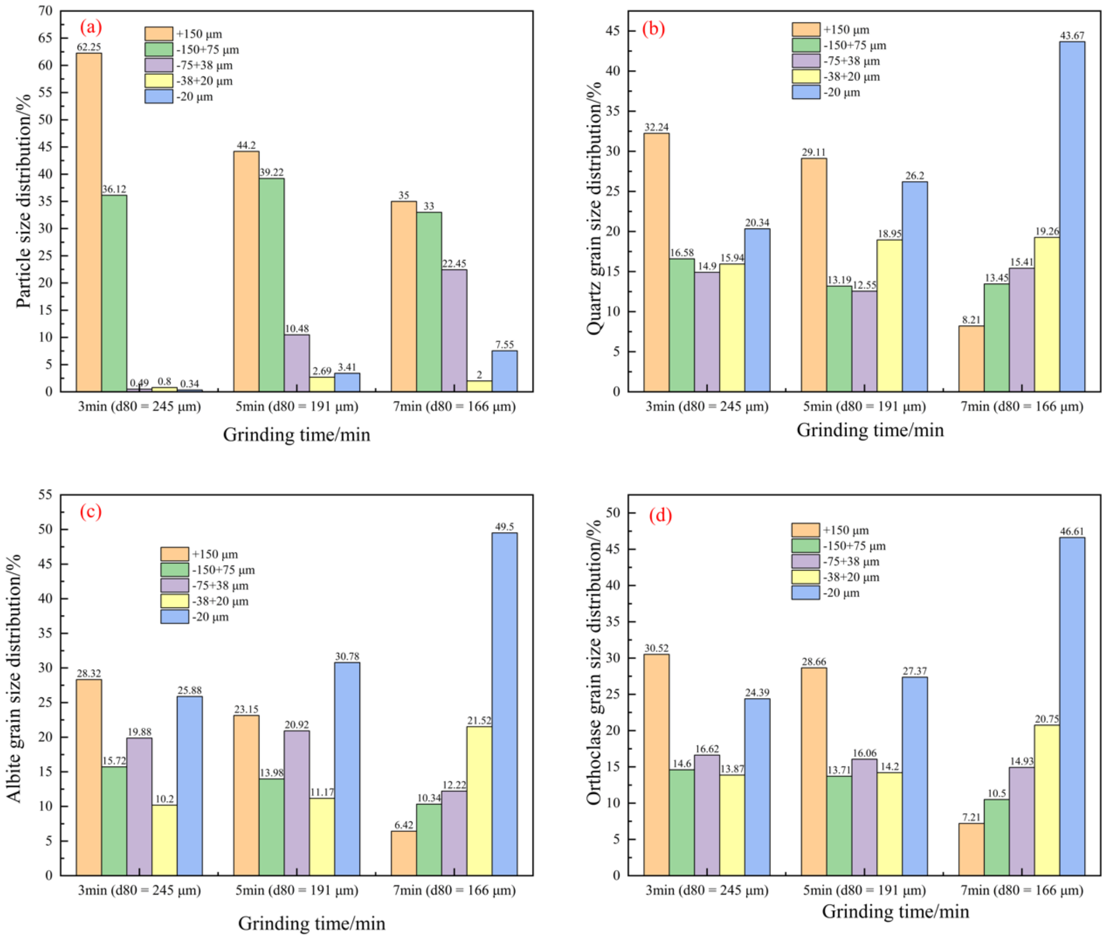

Fig. 8. The particle size distribution and minerals grain size distribution of tailings at different grinding times: (a) Size distribution of particles containing columbite-tantalite; (b) Grain size distribution of quartz in tailings; (c) Grain size distribution of albite in tailings; (b) Grain size distribution of orthoclase in tailings

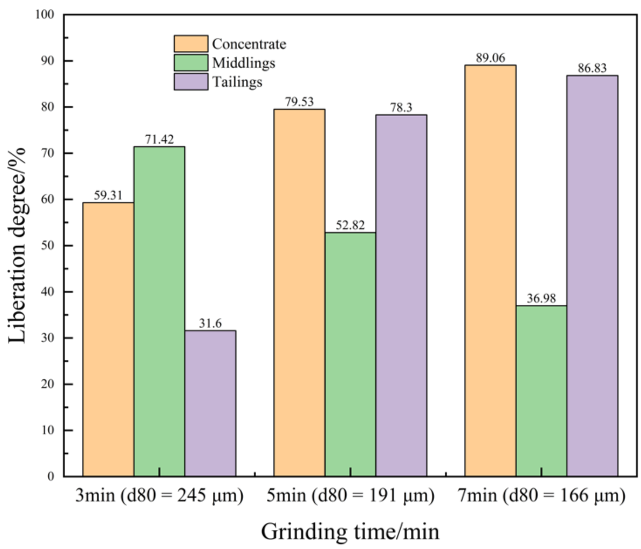

Fig. 9. The liberation degree of columbite-tantalite in gravity separation products

3.3.4. The association relationship of columbite-tantalite in gravity separation products

The association relationship of columbite-tantalite with other minerals in gravity separation products is shown in Fig. 10. As can be seen from Fig. 10, the unliberated columbite-tantalite in the gravity concentrate is mainly associated with quartz, albite, orthoclase, topaz, and goyazite, and the intergrowth type is mainly adjoining (Fig. 11). In the gravity middlings, columbite-tantalite is no longer connected with topaz, and other heavy minerals. The unliberated columbite-tantalite is mostly associated with quartz, albite, goyazite, and zircon, and the associated forms are primarily encapsulated (Fig. 12). In gravity separation tailings, quartz, albite, orthoclase, and lepidolite are the most common minerals associated with unliberated columbite-tantalite. The most common forms of association are encapsulated and adjoining (Fig. 13).

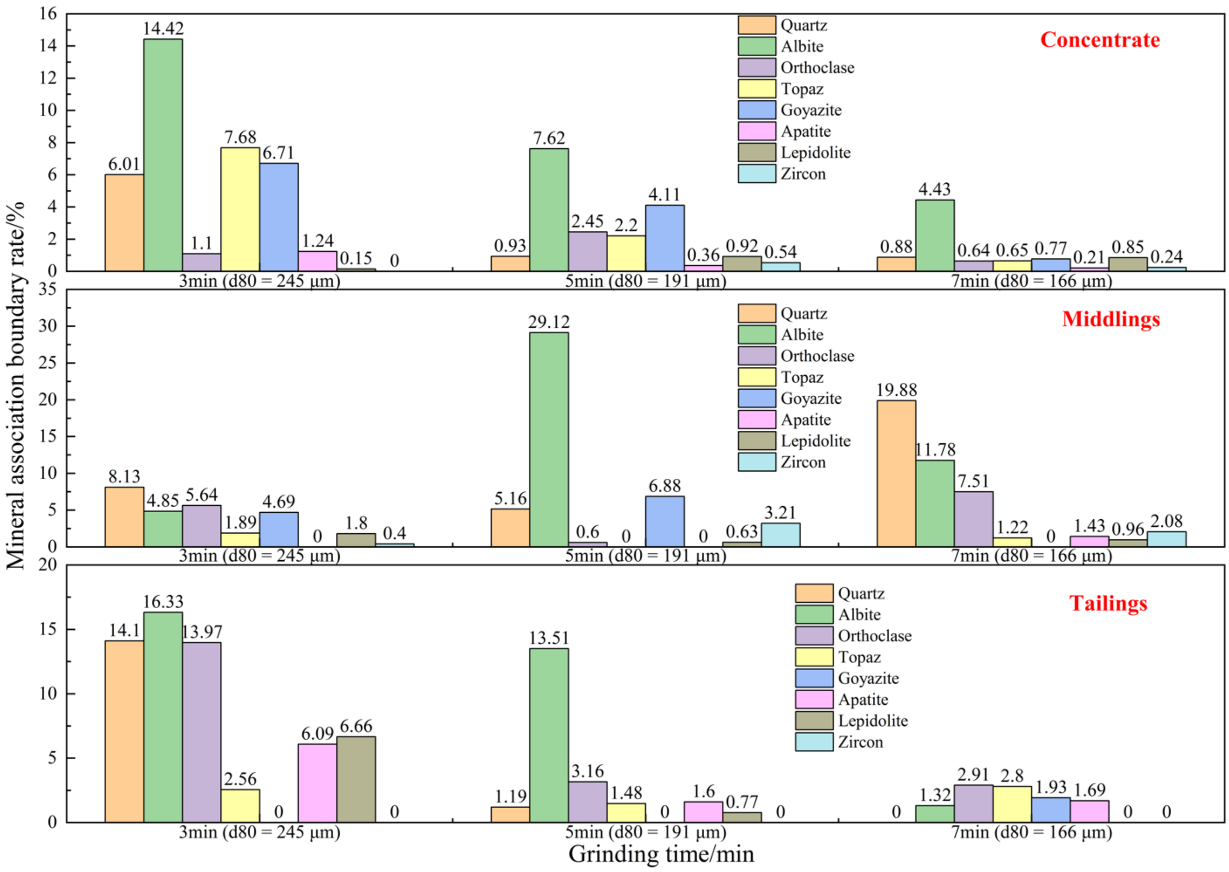

Fig. 10. The association of columbite-tantalite with other minerals in gravity separation products

Fig. 14 displays the density distribution of columbite-tantalite-bearing particles in gravity separation products. With the increase of grinding time, the density of Ta-Nb-bearing mineral particles in the concentrate increases gradually. The density in concentrate is mainly distributed above 3 or even 4, When the grinding time is 7min, the content of Ta-Nb-bearing mineral particles with a density greater than 4 in the concentrate is 82.64%. This is due to the high liberation degree of the columbite-tantalite in the concentrate, as well as the high amount of rich intergrowth associated with heavy minerals. The density of Ta-Nb-bearing mineral particles in the middlings and tailings is predominantly distributed in D<3, owing to the greater content of gangue minerals in the poor columbite-tantalite intergrowth. Furthermore, a minor quantity of D > 3 particles enter the middlings and tailings, probably due to the particle size being too fine.

4. Conclusions

To ascertain the mineralogical factors influencing the separation behavior of Ta-Nb-containing minerals in the gravity field . Investigations have been performed on the separation behavior and mineralogical

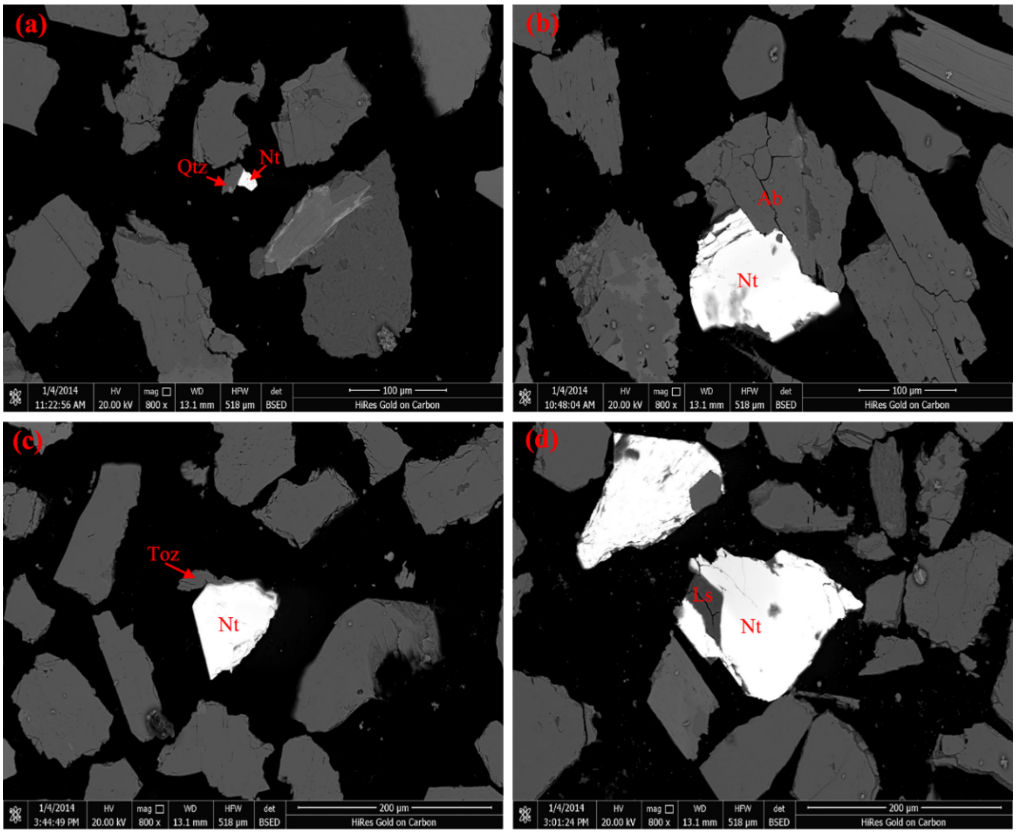

Fig. 11. SEM image of adjoining columbite-tantalite intergrowth (Nt– columbite-tantalite, Qtz– Quartz, Ab– Albite, Toz– Topaz, Ls– Goyazite)

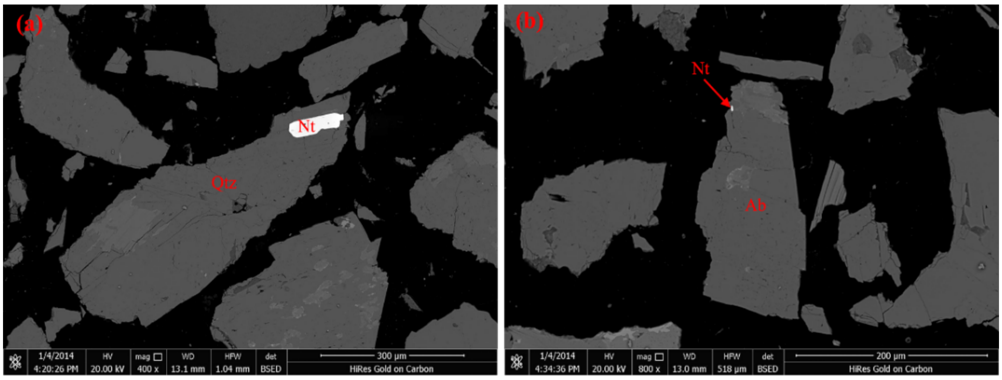

Fig. 12. SEM image of encapsulated type columbite-tantalite association (Nt– columbite-tantalite, Qtz– Quartz, Ab– Albite)

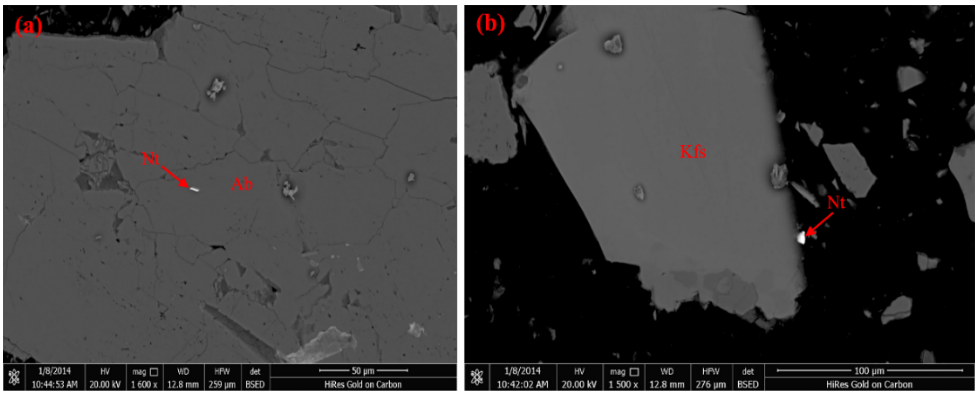

Fig. 13. SEM images of encapsulated type and adjoining type columbite-tantalite association

(Nt– columbite-tantalite, Ab– Albite, Kfs– Orthoclase)

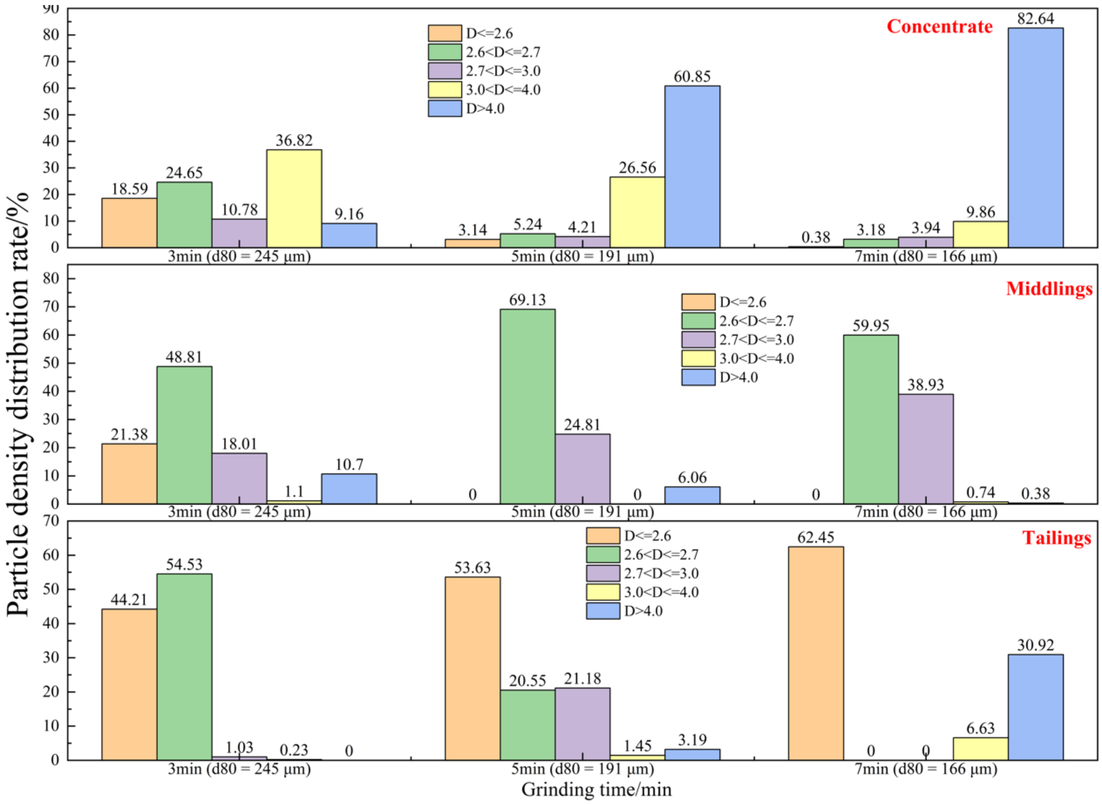

Fig. 14. Columbite-tantalite-bearing particles density distribution in gravity separation products

characteristics of Ta-Nb-containing minerals in the gravity field. It has been shown that the separation behavior of columbite-tantalite in the gravity field is related not only to the liberation degree but also tothe mineralogical characteristics of columbite-tantalite including grain size and association relationship.

The grain size of columbite-tantalite in the concentrate is the coarsest, followed by that in the middlings, and the finest in the tailings, which are mainly distributed in the range of -150+38 μm, -75+20 μm, and -38 μm, respectively. The liberation degree of columbite-tantalite in the concentrate and tailings is positively correlated with grinding time, while that in the middlings is negatively correlated with grinding time. This is due to the fact that the middlings contain a large number of intergrowths, which will either enter the concentrate or the tailings after grinding and liberation. The liberation degree of columbite-tantalite in tailings is also relatively high because the grain size of partially liberated columbite-tantalite is too fine to enter the tailings.

The density of columbite-tantalite-bearing particles in concentrate is mainly distributed above 3 or even 4, due to the high liberation degree of the columbite-tantalite in the concentrate, as well as the high amount of rich intergrowth associated with heavy minerals. The density of Ta-Nb-bearing mineral particles in the middlings and tailings is predominantly distributed in D<3, owing to columbite-tantalite mainly associated with lighter gangue minerals such as quartz, albite, and orthoclase.

It demonstrates that the liberation degree is not the most essential factor in determining columbite-tantalite separation behavior in the gravity field, and the mineralogical characteristics of columbite-tantalite including grain size, association relationship, and particle density, may be more important. The results of this investigation can provide theoretical support for the strengthening separation of low-grade tantalum-niobium ore.

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