CHARACTERIZATION AND BENEFICIATION OF LOW GRADE Nb AND Ta ORE FROM KIBARA BELT

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ABSTRACT

Due to their chemical, physical and mechanical properties, niobium (Nb) and tantalum (Ta) are strategically used in various technical applications. About 130 different minerals contain tantalum and niobium, of which only about 80 are found in economical deposits. The remainder contains Nb and Ta as impurities. The most commercialised source of Nb and Ta is a columbite-tantalite ore often referred to as coltan containing pyrochlorite. The Central African Mesoproterozoic Kibara belt in Katanga (Democratic Republic of Congo) forms a metallogenic province that hosts a variety of granite-related mineralization, rich in cassiterite, columbite–tantalite, wolframite/ferberite, spodumene and beryl. This study focuses on the characterization of Nb/Ta ores from the Kibara belt by using the XRF, SEM/EDS, XRD and ICP-OES. It was observed that Nb/Ta ore that occurs in this belt has a high Nb/Ta ratio. The grade of the ore is 0.01wt% Ta and 0.04wt% Nb. The major minerals phases are Tapiolite, Tantalite, Simpsonite, Pyrochlore and Ilmenorutile. The major gangue minerals present locking Nb/Ta are silica, feldspars with small amounts of mica and quartz. The size of the particles containing Nb/Ta are 8-12microns. Basing on these results, flotation is proposed at as the suitable way of concentrating this ore.

Keywords: niobium, tantalum, flotation, Pyrochlore and Kibara belt

INTRODUCTION

Niobium (Nb) and tantalum (Ta) are classified on the periodic table as elements of transition group belonging to the refractory group. The high melting point of these metals makes them useful in the construction of parts of equipment which are required to withstand high temperature. In the case where Nb and Ta are alloyed with other metals, they pass their characteristic of being superconductive materials, which make them valuable in the manufacturing of electronic devices used in telecommunications, computers and aerodynamic reactors [1]. Due to these particular properties, the demand of these metals is very high and they are coveted in the world and push emerging economy such as China and Japan to sustain their economic with their contribution in the new technologies [2].

The actual estimated global reserves and resources of Nb and Ta are more than sufficient to satisfy the world global demand for the next 500 years [3]. It might be noted according to U.S. Geological survey of Mineral Commodity Summaries of January 2017 that the world mine production and reserves from 2015 to 2016 was estimated to an average of 4300000 and 100000 tons of Nb and Ta respectively [4]. Therefore, geological availability is not considered as a major concern for the supply of niobium and tantalum. To date countries like Brazil, Canada, and Australia have already developed their technologies in the processing and production of Nb and Ta concentrates with Brazil producing 90% of the word production.

A number of African countries such as Burundi, Democratic Republic of Congo (DRC), Ethiopia, Mozambique, Nigeria, Rwanda and Uganda are known for artisanal mining of tantalum minerals in the form of columbite-tantalite also called Coltan. [3]. Over 80% of the world's supply of tantalum comes from Africa particularly from the DRC and Rwanda more precisely in the Kibara belt (KIB) shown in Figure 1. Due to strong social, political and environmental conflicts, metallurgical processes of beneficiating Nb/Ta ores are not well developed in this region. This has led to lack of appropriated investigations on their mineralogical occurrences and characteristics [5]. Consequently, the present work is an investigation on the characterization of the Nb-Ta bearing ore from this region with an aim of improving its processing.

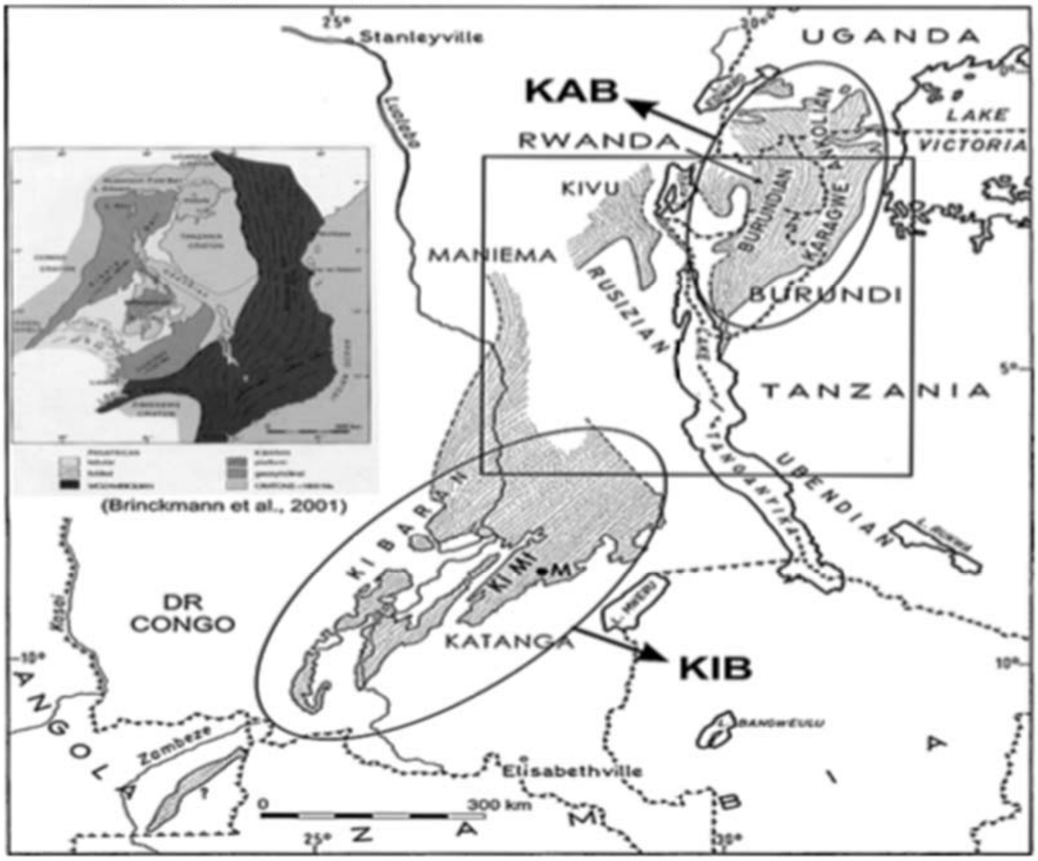

Fig 1: Map of Karagwe-Ankole belt (KAB) and the "Kibara belt" (KIB) [6]

MATERIALS AND METHODS

Material

The sample used in the present investigation was obtained from the Kibara belt located in the eastern part of the DRC bordering Rwanda Republic where the mining activities are basically artisanal.

Sample preparation

The as received sample was primary crushed to 100% passing 4000 µm, blend and split into 1000g. It was then sieved according to the ASTM procedure using sieves sizes from -38 to 4000 µm. After sieving, the particle size distribution was determined using the trompe curve. Selected size ranges (-38, +38 -75, +75 -300 µm and coarser) of the sample was then characterized using XRF, SEM/EDS, XRD and ICP-OES in order to assess for the amount of Nb and Ta, the mineralogical phases Nb and Ta present in the sample and the nature of the gangue mineral hosting them.

Methods

Chemical composition

The sample was pulverized, mix with a wax binder and pelletized. It was then oven dried at 105^{O}C. The compressed sample was then analyzed using X-Ray fluorescent (XRF) Rigaku ZSX Primus II with SQX analysis software (Japan) for chemical composition.

0.2 g of the Nb-Ta bearing ore was placed in a plastic beaker which can withstand temperature around 135^{O}C and cannot be corroded by the digesting media. 5 mL HNO_3 + 2 mL (40%) HF + 5 mL H_2O were added to the sample and heated at 50^{O}C up to dryness then refilled with 5 mL HNO_3. The process was repeated 3 time in order evaporate HF from the solution and avoid any corrosive effects on the instrument during analysis. The sample was filtered and diluted in the 250-ml volumetric flask for analysis. The solution was analyzed with the inductively coupled plasma –optical emission spectrometry ICP-OES Spectro Acros Instrument for chemical composition.

Mineralogical phases

The sample was placed in an aluminum sample holder on the goniometer of the X-ray Powder Diffraction (XRD) Rigaku UltimaIV with PDXL analysis software (Japan). The diffraction beam monochromator operates at 40 KVA and 30 mA with step size of 0.02 to generate the x- ray patterns with enough intensities to produce lines in order to identify minerals in the scanning range of 5^{O} to 90^{O}. Scanning rate was 1 degree per minute while the source of energy was copper with CuK_{α} radiation = 1.5418Å. Mineral phases were identified using the PDXL analysis software of the International Centre for Diffraction Data (ICDD).

Surface morphology

The representative sample was prepared using epoxy resins, polished and made conductive by carbon coating in a Quorum vacuum, Q 150T E. The morphology of the Nb-Ta bearing ore was analyzed in a TESCAN scanning electron microscope (SEM) incorporated with EDX analysis software performance in Nano space at accelerating voltage of 20KVA. Images, grain boundaries and grain sizes were made using the back-scattering electron detectors while chemical elements of the sample were determined by the EDS. The images were shown with point analysis at the positions of the ore particles.

RESULTS AND DISCUSSION

Particle size distribution

The sample was milled for 30minutes in a rod mill and analysed for particle size distribution in comparison to the as received sample and the results are shown in Figure 2. It can be seen that 80% were passing 710 µm and 4000µm for milled and unmilled samples respectively and the ore can be milled easily.

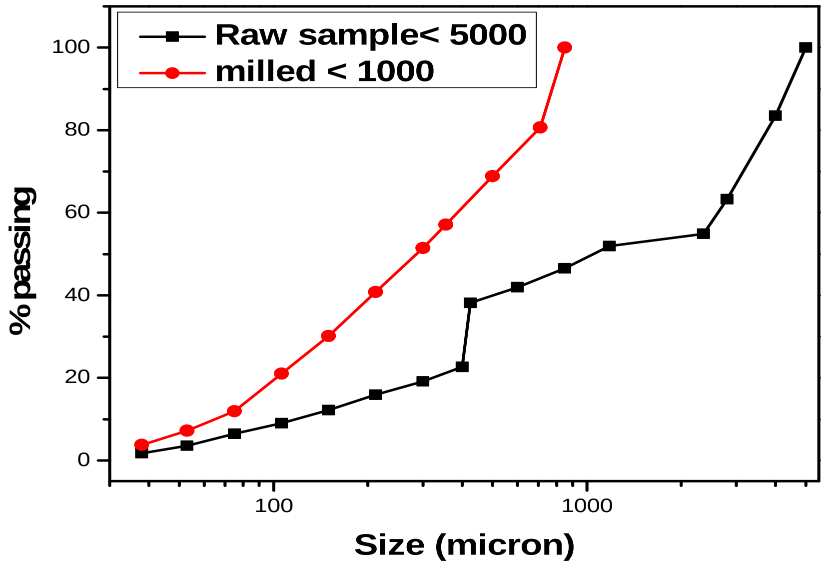

Fig 2: Particle size distribution of the raw and milled samples

Chemical analyses of the sample

The sample was analysed for chemical composition in different size ranges using XRF and the result are displayed in Table 1. It can be seen that Nb grade was 0.05 and 0.1017 %; 0.0366 and 0.0444 % and 0.0388 and 0.0851% in the raw and milled for the size ranges of -38, 75<X<106, 300<X<350 respectively. A similar comparison was done with Ta and it could be seen that the Ta grade was 0.2541 and 0.2854 %; 0.0686 and 0.5938 % and 0.0868 and 0.1504 % in the raw and milled for the same size ranges. Results indicate that the liberation of Nb and Ta is promoted by the milling process and this can be explained by the fact that the sample is dominated by mica minerals which after milling liberate values easily in the bulk of the fines.

The sample was further analysed using ICP-OES. The results of the elemental analysis showed that Nb and Ta content form the raw sample were 0.01675 % and 0.2532 % respectively which correlate with results obtained from the elemental analysis from the XRF. Other elements found in the samples were Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Sr, Zr, Pb and U which may have considerable impact on the processing and beneficiation of these ores.

The mineralogical component of the crushed ore bearing Nb and Ta was carried out by X–ray diffraction technique and results are presented in Figure 3. The main minerals found in the sample were quartz with ICDD card numbers 01-083-2467, muscovite with the ICDD card numbers 00-001-1098 and albite and constitute the main gangue bearing minerals. Nb and Ta appear in the sample in several mineralogical forms where they are either together or separated. Therefore, the following phases which are Simpsonite [$AlTaO_4$], Tantalite [(Fe, Mn) Ta_2O_6], Pyrochlore [$(Na, Ca, U)_2(Nb, Ta)_2O_6(OH, F)$], Niobium Silicide [$(Nb_4Si)_{0.4}$], Tapiolite [(Fe, Mn) $(Ta, Nb)_2O_6$], Ilmenorutile [FeO. $(Nb, Ta)_2O_5.5TiO_2$] were identified as shown in Figure 3.

Table 1: Chemical composition of the raw and milled samples -XRF results

	Raw			milled		
	size (µm)					
Elements	< 38	75<X<106	300<X<350	< 38	75<X<106	300<X<350
Na	3.6514	2.4145	3.5172	2.0934	1.4404	2.2423
Mg	0.3949	0.328	0.2017	0.503	0.4005	0.152
Al	21.7036	20.8857	16.4147	18.7493	20.9553	16.0342
Si	54.8624	50.7139	61.479	47.2724	45.1365	59.7105
P	0.996	0.4002	0.495	0.8685	0.1857	0.3519
S	0.0302	0.0337	0.0237	0.3284	0.3309	0.0325
K	8.6229	17.1289	11.0098	11.4703	21.6786	13.8935
Ca	4.0636	2.7909	2.4052	5.8009	2.6334	2.6687
Ti	0.1312	0.1362	0.0885	0.257	0.1887	
Cr	0.0733	0.1143	0.1336	0.434	0.2508	0.1268
Mn	0.0892	0.0673	0.0409	0.2258	0.0747	0.0705
Fe	4.2044	3.4046	2.6189	9.6375	4.5296	2.6875
Ni	0.0382			0.0896		
Cu	0.0207			0.0855		
Zn	0.0188	0.0239	0.0163	0.344	0.0699	0.035
Ga	0.0117	0.0284	0.0187	0.0219	0.0511	1.7063
Rb	0.3394	0.7164	0.8644	0.8922	1.3451	0.0529
Sr	0.0514	0.0823	0.0484	0.1178	0.0905	
Zr	0.0091	0	0	0.0119	0	0
Nb	**0.05**	**0.0366**	**0.0388**	**0.1017**	**0.0444**	**0.0851**
Ta	**0.2541**	**0.0686**	**0.0868**	**0.2854**	**0.5938**	**0.1504**
Pb	0.0265			0.4095		

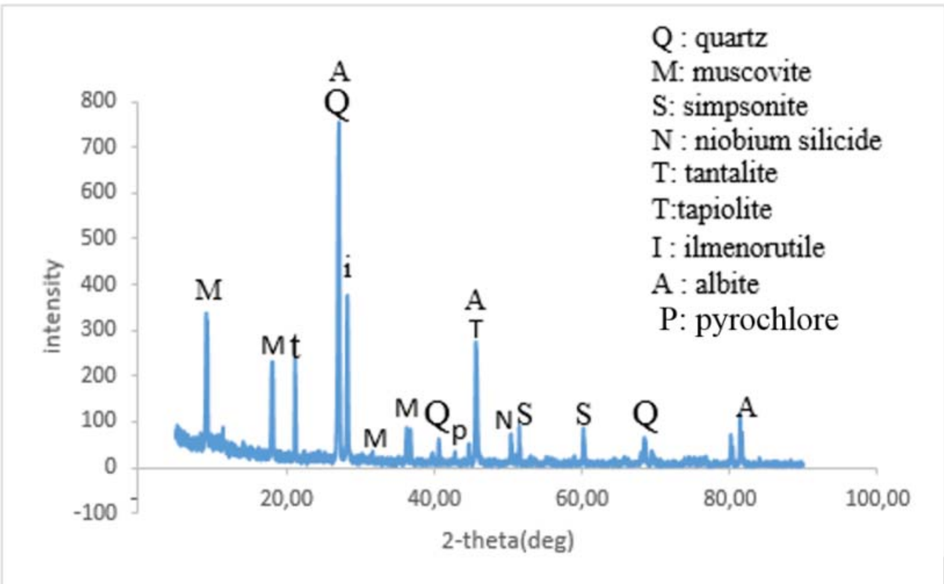

Fig 3: XRD pattern of the as receive sample

The morphological characteristics and the mineral distribution of the sample were determined on fine and coarse particles mounted on the resin (thin section) using SEM while the chemical composition of each particle based on the contrast was determined by EDS. The results are shown in Figure 4 and summarized in Table 2.

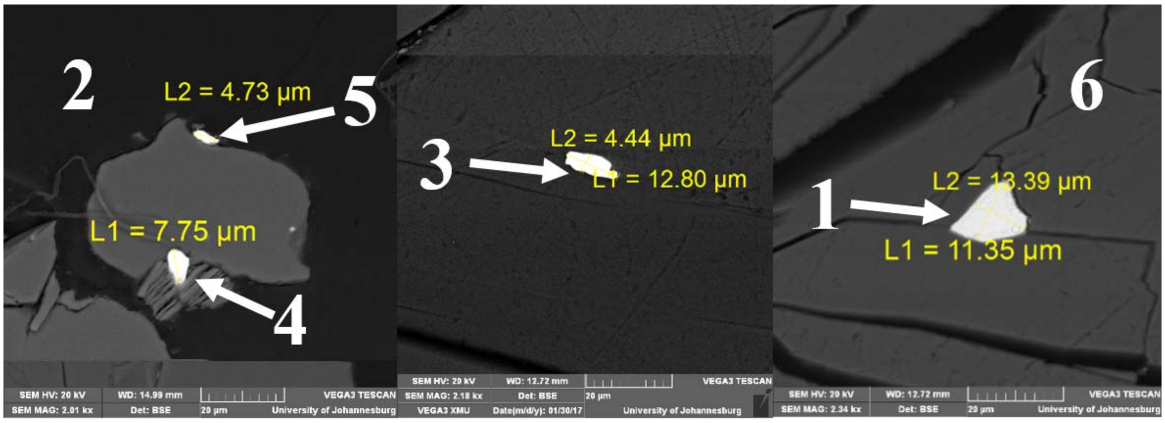

Figure 4: SEM image of the minerals distribution in the raw sample

Results obtained from the sample mounted in the resin show that sizes of the particles bearing Nb and Ta varied from 4 µm to 13µm as presented in figure 4. The sizes of Nb and Ta bearing phases show that their liberation will required fine grinding at a risk to render their beneficiation complex. The EDS spot point analysis showed that the elements that abound in the ore bearing Nb and Ta were Fe, Mn, Nb, Ti, Zr, Al, Y, Si, K, La, Ce, P, Mg, Ca, U and O and results are summarized in Table 2.

Table 2: EDS of minerals distribution in the sample

Element		Na	Al	Si	K	Ca	Ti	Fe	Nb	Ta	U	O
Weight %	1	0.6	0.46	4.46	0.11	5.54	0.66	1.02	2.49	58.46	3.03	23.23
	2	9.59	10.08	31.5		0.16						48.5
	3	0.83	17.46	21.5	6.88	0.91		0.65	0.23	7.51		44.03
	4	0.71	6.42	10.28	2.92	5.35		0.36	1.15	40.79		30.15
	5	0.81	18.45	23.18	6.93	0.6	0.04	0.47	0.08	3.06		45.79
	6	9.59	10.08	31.5	0.16							48.5

The morphological characteristic of the fine sample were as well performed and presented in Figure 5. The results revealed that the sample is basically dominated by a sheet like material which indicated that it has a high amount of mica.

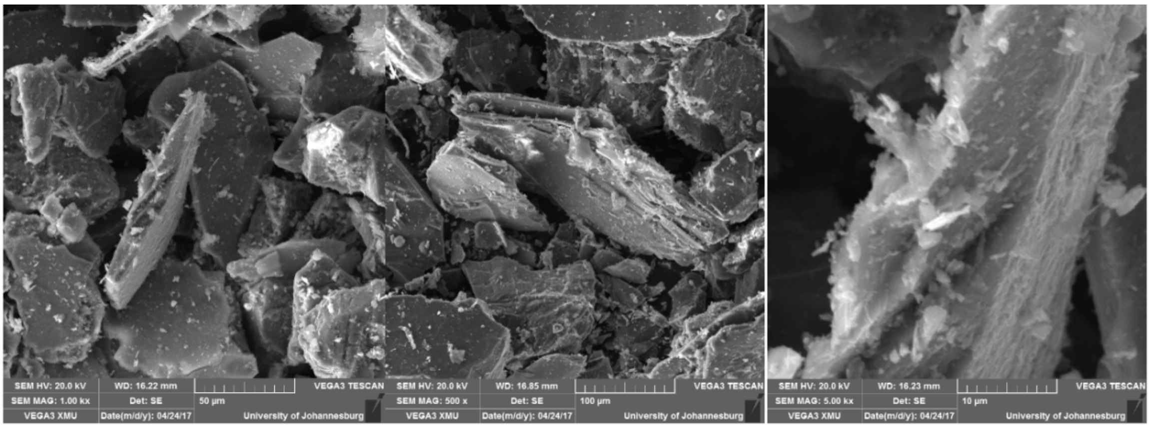

Figure 5: SEM morphology of the raw sample

CONCLUSIONS

The primary objective of this work was to characterize the Kibara belt ore containing Nb and Ta. The mineralogical studies carried out with SEM point imaging showed the presence of different aggregates of minerals bearing Nb and Ta. The SEM image gave the information on the way the phase bearing Nb and Ta are interlocked in the gangue which is basically composed of quartz, granite and mica minerals. The chemical elemental composition determined by EDS, XRF and ICP –OES shows the presence of elements such as Na, Mg, Al, Si, P, S, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Ga, Rb, Sr, Zr and Pb. The XRD reveals the way some of these elements are associated with Nb and Ta. Hence, its phase patterns confirmed the availability of phases bearing Nb and Ta such as Simpsonite, Tantalite, Pyrochlore, Niobium Silicide, Tapiolite, Ilmenorutil. The preliminary liberation study has shown that Nb and Ta ca be physically concentrated at particle size of -38 µm. based on the presented characteristics of this ore, the method and route of processing and the beneficiating Nb and Ta can be flotation.

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