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The Impact of Microwave Pre-Treatment on the Liberation of Nb/Ta Minerals from a Tin Oxide Ore

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Abstract – Liberation of Nb/Ta minerals from Cassiterite ore during comminution is difficult. Microwave pre-treatment of cassiterite ore using batch multiple mode microwave was done. The ore was characterized and the effects of various parameters affecting the liberation of Nb/Ta minerals were investigated. It was found that the ore contains 76. 1%wt of SnO_2 and traces of Nb_2O_5 (0.62% wt) and Ta_2O_5 (0.81% wt). The major phases in the cassiterite ore after pre-treatment were Pyrochlore, Cassiterite, Sillimanite, Ferrotapiolite, Niobium Tantalum, Niobium Titanium, Iron Tin, Niobium Tin, Iron Tantalum and Niobium tin silicide. The ore displayed a significant liberation of the Nb and Ta minerals at 100% irradiation and the high temperature of 540 ℃ was achieved. Significant changes were observed from the SEM/EDS and the Mapping of the distribution of the Nb/Ta minerals showed that there was an improvement in the liberation of these minerals. It is concluded that the use of microwaves brings changes in the matrix pattern of the cassiterite ore, favouring the liberation of microwave susceptible minerals phases.

Keywords: Cassiterite, Microwave Pre-Treatment, Niobium Tantalum, Liberation.

1. Introduction

The potential of microwave selective heating of minerals is not well applied in the mining and mineral extraction industry. Although microwave energy hasn't been applied extensively in the metallurgical processes, it has the following advantages: rapid completion of the heat processes, high yields, low operating and capital costs and environmental benefits. Apart from that, use of microwaves can induce micro fractures in ores containing hydrated minerals thereby weakening the ore and reduce the comminution energy [1]. Macro and micro fractures are induced in ore segments where good heaters of microwave energy are located, such as metal oxides and sulphides. [2]. However, use of a single mode cavity has got its limitations. It is difficult to control and maintain the power since it depends on the packing density of the sample and the dielectric properties of the ore. According to [3], there is a critical power density (~ 109 W/m^3 for absorbing phase) below which microwaves will not efficiently fracture an ore. Very high-power densities combined with short residence times are more efficient. Apart from that, there are other important factors such as particle shape and size, mineral composition and texture of the minerals.

Due to increase in use of Nb/Ta in modern technology, there is need to increase the production of these metals. Global Ta demand in recent years has been driven up specifically by use of Ta capacitors in portable electronic devices including mobile phones and this demand is expected to increase [4]. Currently Nb/Ta metals are produced by gravity concentration, magnetic separation, electrostatic and some flotation [5, 6, 7]. The success of all these processes depend on the liberation of Nb/Ta minerals, hence comminution plays a very important role. Comminution processes are energy intensive and there is need to pre-treat the ore and reduce the Bond work index of the ore.

In this paper, the effect of microwave pre-treatment of cassiterite ore on the liberation of Nb/Ta minerals is presented. The main objective of this study is to investigate the phase changes during microwave processing of cassiterite ores bearing Nb/Ta minerals. This in turn would determine the grindability of the ore.

2. Materials And Methods

2.1. Materials

The cassiterite ore used in this investigation was supplied from a mine in the eastern part of the Kibara belt in the Democratic Republic of Congo (DRC). The chemical composition of the cassiterite ore was determined by the wavelength dispersive X-ray fluorescence spectrometry (XRF) using a Rigaku ZSX primus 2 instrument with SQX analysis the mineralogical phases were obtained using a Rigaku ultimal V X-ray diffractometer (XRD) equipped with graphite monochromator in the diffracted beam and using CμKα radiation, PDXL analysis software. The morphology of the sample was investigated using scanning electron microscopy (SEM) TESCAN performing in nanospace at 15kv.

2.2. Method

A Cassiterite ore sample weighing 45 kg was homogenized using a cone blender for one hour and split into halves using a Jones riffle splitter. After splitting, sub samples of 1250 g were obtained using a spinning riffle splitter and send for characterisation. Further samples of 50g were obtained from these samples and used for microwave pre-treatment experiments. A multiple mode microwave with a maximum power of 700 W was used. The samples were treated for 30minutes and irradiations of 16, 66, and 100 % of 700 W were applied respectively. After irradiation, the samples were air cooled for a day and then analysed for mineral phase changes.

3. Results and Discussion

3.1. Chemical Composition of the Cassiterite Ore

The chemical composition of the sample was analyzed using XRF and the results are shown in Table 1. Table 1 shows that the sample contains a high grade of SnO_2 (76.01%) and traces of Nb_2O_5 (0.62%) and Ta_2O_5 (0.81%). Usually the ore from this area contains Uranium (U_3O_8) but it was not detected in this sample. PbO (0.2%) detected was very low to cause any harm during microwave pretreatment and the subsequent smelting processes. Major gangue in the sample was SiO_2 (5.93%) and Fe_2O_3 (7.41%). Iron has the disadvantage that it goes into the metal during reductive smelting and contaminate the metal product.

Table 1: Chemical composition of the Cassiterite ore sample.

Comp	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	TiO_2	MnO	Fe_2O_3	ZrO_2
%wt.	0.13	3.19	5.93	0.40	0.29	1.33	0.25	7.41	1.56
Comp	Nb_2O_5	SnO_2	La_2O_3	CeO_2	Ta_2O_5	WO_3	ThO_2	-	PbO
%wt.	0.62	76.01	0.31	0.80	0.81	0.21	0.04	-	0.2

3.2. The Effect of Microwave Pre-Treatment on the Mineral Phases in the Ore Sample

The effect of microwave pre-treatment on the mineral phase changes of the Cassiterite ore was analysed using XRD and the results are shown in Figure 1. Figure 1 illustrates that as intensity of irradiation increases, new phases are formed. At 16% irradiation, a temperature of 162^0C was achieved and no major phase changes were observed. At 66% irradiation, a temperature of 232^0C was achieved and new phases formed were Magnesium tin oxide, Ferrotapiolite and Foordite. Further increasing the irradiation to 100%, a temperature of 540^0C was achieved and other new phases that were formed are Iron Tin Titanium oxide and Tantalum silicide. There was no change on quartz, feldspar and calcite phase during the pre-treatment and this might be due to the fact that minerals of value absorb microwaves whereas common host rock minerals don't .

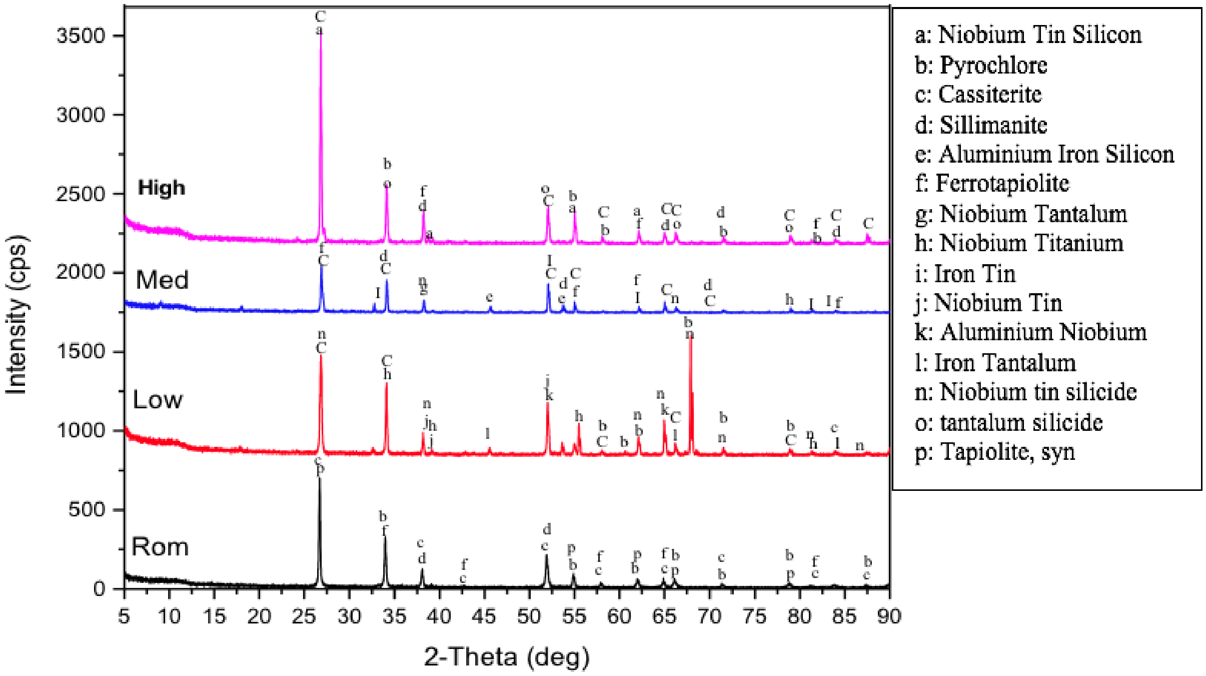
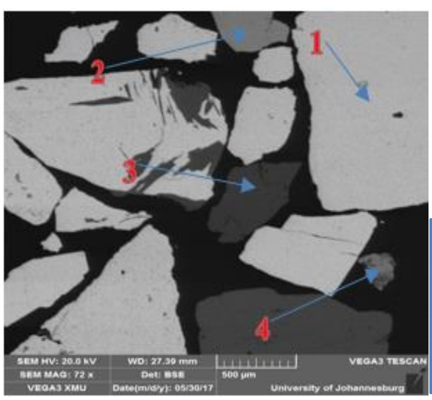


Fig. 1: Mineral phase changes of cassiterite ore samples after irradiation.

3.3. Surface Morphology of the Cassiterite Raw Sample

The surface topography of the ore sample was analysed using SEM and EDs and the results are shown in Figure 2. The results show that major phases in the ore are Cassiterite, Tapiolite, Ferrotapiolite, Sillimanite and quartz. This confirms the results that were obtained using XRD.

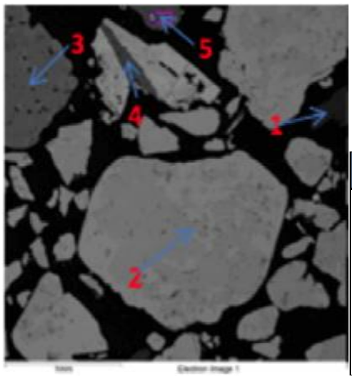


Elem	Mg K	Al K	Si K	Ti K	Fe K	Nb L	Sn L	Ta M	W M	Mineral phases
1	0	0,05	0,07	0,37	0,09	0,09	98,47	0,37	0,5	Cassiterite, Tapiolite
2	0,1	0	99,17	0	0,73		0	0	0	Quartz
3	0	0	99,76	0,07	0,16	0,01	0	0	0	Quartz
4	0	2,88	3,45	0,52	92,9	0,25	0	0	0	Ferrotapiolite, Sillimanite

Fig. 2: Micrograph of the raw Cassiterite sample.

3.4. The Effect of Microwave Irradiation on Surface Morphology

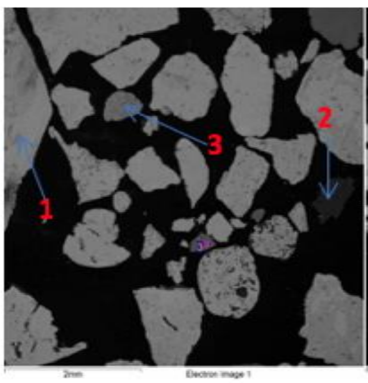
The effect of irradiation on surface morphology was investigated using SEM/EDS and the results are illustrated in Figures 3-5. From Figure 2, it can be seen that from the lower power of 16% irradiation, a temperature of 162^{0}C was achieved. There was no change compare to the untreatable ore. Cassiterite, Aluminium Niobium, Niobium Titanium, Quartz and sillimanite were the existing phases and they match the phases of the untreated ore.



		Elem	Mg K	Al K	Si K	Mn K	Ti K	Fe K	Zr L	Nb L	Sn L	Ta M	Mineral phases
65x a	1	Wt%	0	39	59,8	0	0	0,43	0	0,72	0	0	Quartz, Sillimanite
	2	Wt%	0	0	0,34	0	0	0,72	0	0	97,6	1,31	Cassiterite, Tantalum(I) oxide,
	3	Wt%	0	0,68	20,2	0	0	0,16	76,7	2,29	0	0	Aluminum Niobium, Quartz, Aluminum Niobium
	4	Wt%	2,27	2,23	0	0	0	89	0	0	3,45	3,04	Tantalum(I) oxide, Cassiterite, Sillimanite
	5	Wt%	0	0	0	0	53,4	44,5	0	0	0,3	0,46	ferrotapiolite

Fig. 3: Micrograph of the sample treated at 16% of full irradiation for 30 minutes.

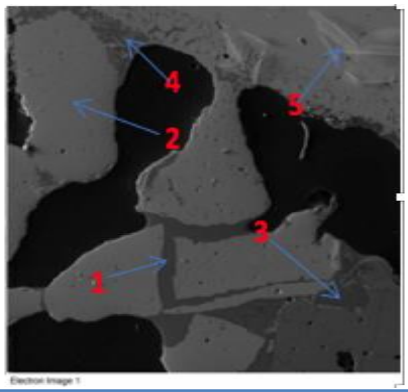
The sample was treated at 66% of full power irradiation for 30 minutes and the results are shown in Figure 3. A temperature of 232^{0}C was achieved and a considerable number of fractures could be seen. New phases were formed such as Magnesium Tin Oxide, Ferrotapiolite and Foordite.



		Elem	Al K	Si K	Zr L	Fe K	Nb L	Sn L	Ta M	Mineral phases
36x a	1	Wt%	0,11	1,73	0	0	0,08	97,2	0,87	cassiterite, Quartz
	2	Wt%	1,89	54,1	0	0,49	0	0,5	4,33	Ferrotapiolite, Sillimanite
	3	Wt%	0	20,1	77,3	0	2,53	0,15	0	Quartz, Foordite

Fig. 4: Micrograph of the sample treated at 66% of full irradiation for 30 minutes.

The sample was then treated at full irradiation and the results are shown in Figure 4. Figure 4 shows that partial melting occurred in certain areas. This indicates that the temperature reached in the matrix of the ore must have been quite high (540^{0}C). While the significant increase in fracture after 30minutes microwave treatment may give rise to significant reductions in Bond work index, this long exposure time and high temperature may prove to be detrimental to any further processing of the ore. The EDs results show evidence of formation of new phases such as Tin Titanium Oxide, Tantalum silicide, Niobium Titanium and sillimanite on the surface morphology of the sample.



		Elem	Mg K	Al K	Si K	Ti K	Mn K	Fe K	Nb L	Sn L	Ce L	Ta M	W M	Mineral phases
270 X	1	Wt%	0,85	14,9	39,5	2,06	4,21	15,5	0	1,89	0	7,72	13,4	tantalum silicide, Tin Titanium Oxide, Quartz, ferrotapiolite
	2	Wt%	0	0,12	0,06	0	0	0,09	0,14	98,9	0	0,72	0	dialuminum silicon oxide, Cassiterite
	3	Wt%	0,86	15,1	42,5	1,85	4,1	15		1,76	0	5,69	13,2	tantalum silicide, Tin Titanium Oxide, Quartz, ferrotapiolite, dialuminum silicon oxide
	4	Wt%	0		5,79	0	0,46	7,99	5,06	0,71	0	80	0	Quartz, tantalum silicide, ferrotapiolite
	5	Wt%	0	12,1	49,5	0,96	14,19	14,2	1,61	2,84	4,31	0,36	0	dialuminum silicon oxide, Cassiterite, Quartz

Fig. 5: Micrograph of the sample treated at 100% irradiation for 30 minutes.

3.5. Mapping for Full Power Irradiation

It can clearly be seen from the mapping that when a matrix containing the above minerals is heated large internal stress will occur. Therefore, for this material, significant increases in grade of Niobium and Tantalum were expected after microwave treatment. From the image we can see the distribution of different elements compared to each other after it has been expose to the high power irradiation where Niobium was almost distributed every way around the sample compare to tin oxide.

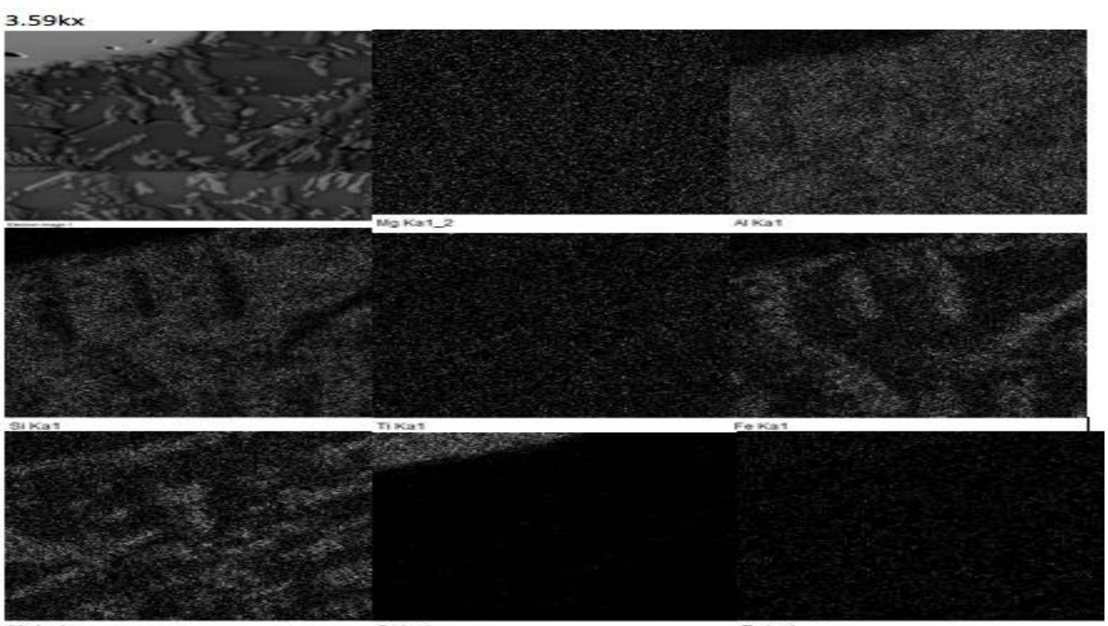


Fig. 6: Mapping of Cassiterite sample exposed to full power irradiation for 30 minutes.

4. Conclusion

The results of this study indicate that microwave, radiation has a significant effect upon the mineralogy and magnetic processing of the cassiterite ore. It has been shown that short periods of exposure can cause a partial melting in certain areas and fracture at grain boundaries which leads to the formation of intergranular fractures. These fractures reduces the Bond Working Index of the ore and comminution costs. However, the study has also indicated that process efficiency can be affected by over exposure of the ore to the microwave radiation. As energy is being added to the process, a detailed techno-economic analysis is required. This will form the next stage of this research together with further work on reducing the cost controlling factor of microwave exposure times. Points to consider are the use of higher powers for shorter times and pulsed delivery of radiation as both these methods will reduce the input of energy to the system overall and add support to the process economic.

Acknowledgements

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