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URANIUM-COPPER MINERALOGICAL CHARACTERIZATION OF A FLOTATION CONCENTRATE USING QEM-SCAN

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ABSTRACT

This study investigates the mode of occurrence of uranium in a copper concentrate from the Prominent Hill mine. Mineralogical and liberation studies of a high grade Cu concentrate (monthly composite) containing uranium (U) were undertaken using QEM-Scan in combination with QXRD and ICP-MS. The study aimed at determining in what form U was recovered, and by which mechanisms. The deportment of U was studied in 3 size fractions; +20µm, -20+8µm and -8µm. QEM-Scan analysis showed that uranium was present as U(Cu)-silicates, U-silicates, UPbOx/FeOx or traces of UThOx and UTiOx, with the U-silicates present across the size fractions. The association of uranium bearing minerals with copper sulphides varied across the size ranges. Interestingly, most of the U (46% by weight) was distributed in the finest fraction (-8µm), in which the deportment of Cu was low (6%). Over 40% of the U mineralisation in the -8µm was liberated. In terms of the floatation response, while recovery of the uranium minerals as a result of their association with copper sulphides (as composite particles) was evident, recovery of UPbOx locked in hydrophilic haematite in the +20µm size fraction suggests, that other mechanisms (e.g. Inadvertent activation by Pb from radioactive U decay) are possible. Furthermore, the high amount of fine liberated U bearing particles in the concentrate points at entrainment as another major mechanism for uranium recovery, and therefore, a target for process optimisation.

Keywords: uranium, copper ore, QEMSCAN, liberation, entrainment

INTRODUCTION

Uranium is a reactive element with a negative reduction potential of -1.38mV and frequently occurs as oxides and/or oxysalts, elucidating their high affinity for oxygen. For nearly all of the oxysalts, uranium is oxidized to the soluble hexavalent state (U^{6+}) (Bowell *et al*, 2011). In this regard, uranyl minerals generally, occur as expressed in equation (1) (Rogers and Adam, 1967; Roger *et al*, 2009).

$$MUO_2 + (XO_4)_n \tag{1}$$

Where M = Na, K, Mg, Ca, Ba; Al, Pb, Bi, Fe, Co, Cu, Zn; REE (Y, La, Lanthanides), actinides (Th) and XO_4 = PO_4, AsO_4, VO_4, SO_4, MoO_4, WO_4, CO_3.

The primary minerals of U are uraninite or pitchblende (UO_2), the simple oxides; brannerite [(U, Ca, Ce)$(Ti,Fe)_2O_6$] and coffinite[$U(SiO_4)_{1-x}(OH)_{4x}$ which are refractory minerals (Elzbieta *et al*, 2009; Bowell *et al*, 2011). These primary minerals often occur in nonoxidizing and sub-surface environments (Clarke *et al*, 1966; Elzbieta *et al*, 2009). Generally, uranium occurs in several environments including most gold and copper deposits (Roger *et al*, 2009). However, only a few of these deposits contain economically extractable uranium such as the

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Witwatersrand Gold deposit of South Africa (Makanza *et al*, 2008) or Olympic Dam Copper deposit of South Australia (Ragozzini, 1986; Belpario *et al*, 2007). Under normal conditions, the oxides and oxysalts of uranium are not expected to float in sulphide minerals floatation. They are highly hydrophilic minerals compared to sulphides, and are expected to remain wet in the pulp together with other non-sulphides (i.e. Silicates, carbonates, oxides, sulphates, etc.) and eventually report to tailings. Reported floatation recovery of uraninite and brannerite for instance in Witwatersrand Gold ore, was promoted by their association with pyrite and naturally hydrophobic kerogen (Makanza *et al*, 2008), and not related to their inherent float ability.

In operations, where uranium is a valuable mineral (e.g. Olympic Dam), leaching is often the preferred method to economically recover uranium, and the process is well established (e.g. Ragozzini, 1986; Guettaff *et al*, 2009, Roshani and Mirjalili, 2009). Equation (2) depicts the leaching of primary uranium minerals (U^{4+}) which is usually catalysed by ferric ions (Fe^{3+}) unlike the secondary minerals that readily dissolve in sulphuric acid.

$$UO_2 + 3Fe^{3+} = UO_2^{2+} + 2Fe^{2+} \tag{2}$$

In the Prominent Hill Copper-gold-silver ore, naturally occurring uranium is present in sub economic concentrations. This is in contrast to other ore assemblages in South Australia, for example, Olympic Dam where uranium is recovered as a valuable mineral together with Copper, Gold and Silver (Ragozzini 1986; Belpario *et al*, 2007). The Prominent Hill mine employs conventional grinding and floatation circuit to recover Copper, Gold and Silver. The rougher floatation concentrate is reground in Isa Mill, to 80% passing 20µm, with the aim to liberate the valuable copper sulfides from the Gangue minerals including the uranium bearing minerals. The reground ore is then treated in a Jameson floatation cell, where the liberated and readily floatable particles are recovered. The Jameson cell tailings are treated in a 3 stage conventional mechanical cleaner circuit. The cleaner concentrate is then combined with the Jameson cell concentrate to produce the final concentrate for sale.

It is generally, understood that U recovery in floatation is greatly influenced, by the association with copper sulphides, even at extremely fine particle size, but no recovery mechanism has yet been established as dominant. This paper discusses the quantitative mineralogical work carried out in a study aiming to establish the mechanisms by which uranium is recovered.

MATERIALS AND METHODS

Materials

A 200 g sample of high grade 3rd cleaner concentrate (monthly composite) containing a significant amount of uranium received from the Prominent Hill Mine was used for this study. The sample was riffle split into two equal fractions, one of which was cyclone cut, using a cyclone rig with spigot diameter of ¼ inches to separate the ultrafine fraction (< 8 µm, refer Figure 1). The underflow fraction (> 8 µm) was further wet screened using a laboratory 20µm sieve.

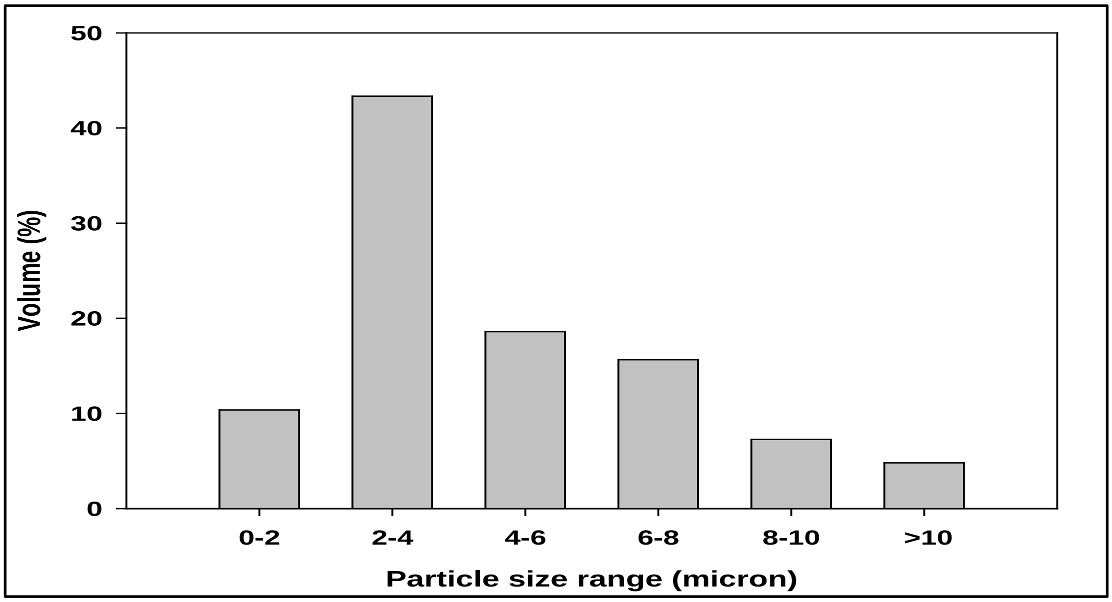

Figure 1. Particle size distribution of the cyclone rig cut overflow for the 3rd cleaner concentrate sample. Distribution determined with a Malvern laser particle sizer

The size fractions produced; +20µm, -20,+8µm and -8µm were collected, filtered and dried under controlled temperature to prevent oxidation. Polished sections were made per size fraction for QEMSCAN analysis. A total of 12 polished sections were made, 3 each for the coarsest (+20µm) and finest (-8µm) fractions and 6 for the intermediate fraction (-20,+8µm).

Methodology

QEMSCAN (Quantitative evaluation of minerals by scanning electron microscopy), was used primarily in this study, in combination with QXRD and ICP-MS.

A QEMSCAN apparatus with Zeiss EVO 50 SEM fitted with four Gresham thin-window EDX detectors, BSE and SE detectors available at The Wark^{TM} was employed for the mineralogical and liberation analysis of the samples. The measurement mode used was PMA, but SMS mode was also used for identification of uranium. The softwares Discover and iExplorer (Gottlieb *et al*, 2000; French *et al*, 2008; Aral 2010) were used to generate information on the chemical and mineral composition of samples.

The XRD analysis was done using a Scintag ARL X'tradiffractometer and CuKα radiation. XRD traces were collected between 5º and 90º 2θ at 0.02º intervals at the rate of 0.05º per minute. SIROQUANT V3 software was used to quantify the minerals.

RESULTS AND DISCUSSION

Modal Mineralogy

The mineral composition of the 3rd cleaner concentrate determined by QEMSCAN analysis is shown in Table 1. As expected, the major mineral phases are the copper sulphides, with chalcocite predominantly high across all sizes confirming a chalcocite-rich concentrate. Some secondary copper minerals were also present (e.g. Malachite, cuprite) but were mainly found in the -20+8µm size range. Apart from the copper minerals, other sulphides present are Pyrite, Pyrrhotite and Sphalerite, in varying proportions. Galena was found only in trace amounts. Fe oxides, quartz, Micas, Chlorite, Fluorite and Carbonates are the main Gangue minerals identified, most of which were recovered in floatation locked with the copper minerals.

Table 1. Mineral composition of the 3rd cleaner concentrate by size

Mineral Phases	**Weight Percent (%)**	**in Fraction**	
Size (μm)	**+20**	**-20,+8**	**-8**
UPbOx	0.001	0.000	0.000
USiCu	0.001	0.002	0.002
USi	0.000	0.002	0.002
Fe Sulphides	1.59	1.46	0.24
Chalcopyrite	0.84	3.54	0.36
Chalcocite/Digenite	18.77	31.42	2.45
Bornite	3.89	9.72	0.59
Cu-Fe interface	2.60	5.62	2.91
Cc/Silicates	0.20	0.30	0.31
Cu Oxides	0.12	0.13	0.00
Other Sulphides	0.01	0.03	0.02
Quartz	0.71	0.13	0.01
Mica and chlorite	0.23	0.10	0.01
Other Silicates	0.01	0.02	0.00
Carbonates	0.07	0.03	0.00
Fe Oxides	3.16	1.44	0.12
Other Oxides	0.04	0.06	0.01
Fluorite	0.04	0.01	0.00
Sulphates	0.01	0.01	0.01
Others	0.07	0.09	0.02
Total	32.36	54.10	7.07

The QEMSCAN results are consistent with quantitative XRD data conducted on similar size fractions (refer Table 2).

Table 2. Quantitative XRD analysis of the 3rd cleaner concentrate by size

Mineral Phase	**Weight Percent (%)**	**in fraction**	
Size Fraction (μm)	**+20**	**-20,+8**	**-8**
Chalcocite	26	32	18
Bornite	14	21	13
Chalcopyrite	6	9	10
Pyrite	8	17	18
Haematite	16	14	16
Quartz	5	4	5
Amorphous	25	3	21

Since the detection limit, of this XRD is 2.0 weight percent, uranium which occurred as trace mineral (less than 1 wt %) was difficult to identify (Mandile and Johnson, 1998).

However, QEMSCAN analysis identified some uranium bearing phases, based on elemental peaks present in the spectra per pixel. The majority of uranium occurred as U(Cu)- silicate, U-silicate and to lesser extent U-Pb oxide/FeOX. Traces of U/Th and U/Ti oxides were also found.

Whether the Cu in the U-silicate was present within the U minerals as elemental copper, or came from copper sulphide phase which U was associated with was unclear. However, from a previous preliminary QEMSCAN X-ray data on the same ore, elemental Cu was found in U-bearing oxide phases and assumed a quite similar occurrence.

Distribution and Association of Cu-Bearing Minerals

The distribution of copper minerals across the size fractions is depicted in Figure 2. Over 90% of all copper minerals present are in the +8µm size range, with only less than 10% in the finest fraction analyzed. Of the Copper minerals, Chalcocite is the predominant phase, constituting well over 50% of total copper minerals, elucidating the high grade nature of the concentrate.

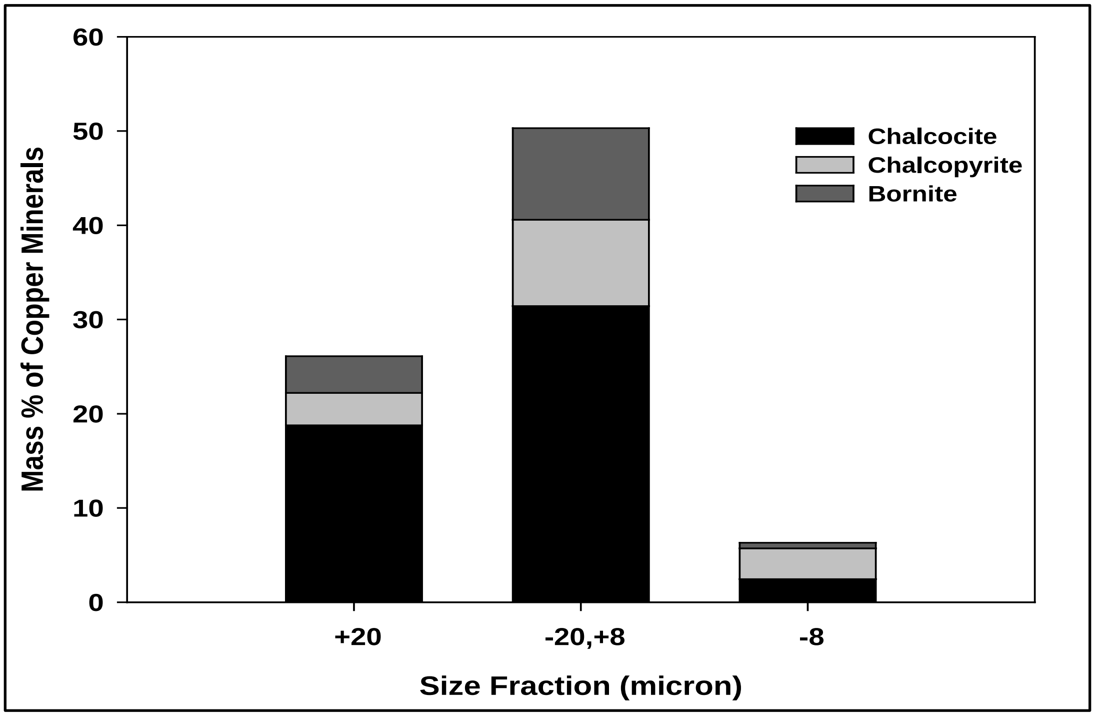

Figure 2. Deportment of copper minerals by size

The detailed association characteristic of these copper minerals per size is tabulated in Table 3. According to Table 3a, about 65% of the total, Chalcocite mineral is liberated with bulk of the remainder in binary with Bornite (28%). Only less than 3% is associated with Gangue minerals and in multiphase is about 3.7%. Bornite, on the other hand, was recovered mainly in binary composites with Chalcocite (73%), and slightly over 12% as liberated particles (refer Table 3b).

However, a similar trend in the association of Bornite and Chalcocite with the Gangue minerals was observed which is same for chalcopyrite (refer Table 3c). The majority of chalcopyrite is in binary composites with Chalcocite or Bornite, and only less than 2% with Gangue minerals.

The lower proportion of iron oxides and silicates (Gangue minerals) recovered in association with the copper minerals reflects the quality of the concentrate.

Table 3(a). Association characteristics of chalcocite in the 3rd cleaner concentrate

Chalcocite(Cc) status	**Weight Percent (%)**			
Size (μm)	**+20**	**-20,+8**	**-8**	**Sum(∑)**
Liberated	22.4	40.6	2.2	65.2
Cc-Bn	10.5	16.0	1.8	28.4
Cc-Cp	0.0	0.4	0.1	0.5
Cc-Py	0.0	0.1	0.0	0.1
Cc-FeOx	0.8	0.3	0.0	1.2
Cc-silicates	0.4	0.5	0.2	1.0
Multiphase	1.6	1.7	0.4	3.7
Total	35.7	59.7	4.7	100

Note: Cc: chalcocite; Cp: chalcopyrite; Bn: bornite; Py: pyrite; FeOx: iron oxide

Table 3(b). Association characteristics of bornite in the 3rd cleaner concentrate

Bornite(Bn) status	**Weight Percent (%)**			
Size (μm)	**+20**	**-20,+8**	**-8**	**Sum(∑)**
Liberated	3.8	8.3	0.4	12.5
Bn-Cc	17.3	52.9	2.9	73.1
Bn-Cp	0.5	2.9	0.2	3.8
Bn-Py	0.5	0.2	0.0	0.7
Bn-FeOx	0.7	0.6	0.0	1.4
Multiphase	4.5	3.6	0.6	8.7
Total	35.7	59.7	4.7	100

Table 3(c). Association characteristics of chalcopyrite in the 3rd cleaner concentrate

Chalcopyrite (Cp) status	**Weight Percent (%)**			
Size (μm)	**+20**	**-20,+8**	**-8**	**Sum(∑)**
Liberated	7.7	29.4	1.3	38.4
Cp-Cc	3.8	28.9	3.8	36.6
Cp-Bn	1.5	6.3	0.5	8.3
Cp-Py	0.7	3.0	0.7	4.4
Cp-FeOx	0.7	0.8	0.0	1.5
Cu(Fe)S-Py	1.7	4.1	0.5	6.3
Cu(Fe)S-FeOx	0.1	0.2	0.0	0.3
Multiphase	1.5	1.9	0.8	4.2
Total	17.8	74.6	7.6	100

Detailed liberation analysis of the copper minerals is shown in Table 4. The analysis describes the surface exposure (or areal grade) of the valuable copper minerals in an interval of 10%. Normally, mineral phases with surface exposure of ≤30% are considered locked, while surface exposure ≥90% is accepted as liberated. From 60 to 90%, surface exposure is termed high-grade middling, whereas 30 to 60% of mineral surface exposed is considered low-grade middling (Savassi, 2006).

Table 4. Liberation analysis of combined copper minerals in the 3rd cleaner concentrate

Size (µm)	**Areal grade (%)**											
	≤10 (%)	**10-20 (%)**	**20-30 (%)**	**30-40 (%)**	**40-50 (%)**	**50-60 (%)**	**60-70 (%)**	**70-80 (%)**	**80-90 (%)**	**90-100 (%)**	**100 (%)**	**Total**
+20	0.35	0.62	0.66	0.66	1.04	1.12	1.44	1.72	4.67	46.3	41.5	100
-20,+8	0.07	0.18	0.20	0.29	0.40	0.38	0.54	0.80	1.76	14.8	80.6	100
-8	0.02	0.11	0.18	0.36	0.75	0.37	0.85	1.42	2.20	4.01	89.7	100
Combine	0.15	0.31	0.34	0.41	0.63	0.61	0.85	1.14	2.70	23.7	69.1	100

All the copper minerals were essentially liberated across the size fractions (69%), with only small proportions recovered as middling (refer Table 4). Those in fully locked category are less than 1%. Such result also suggests, that any uranium mineral recovered in middlings with the copper sulphides is in smaller proportion.

Distribution and Association of U-bearing Minerals

Among 46,803 measured particles, 44 contained uranium bearing particles; 5 particles were present within the +20µm size range, 9 particles in the intermediate size range (-20+8µm) and 30 particles in the finest fraction (-8µm), which represents about 60 % of the total U found. The association characteristic of these U bearing particles is depicted in Figure 3.

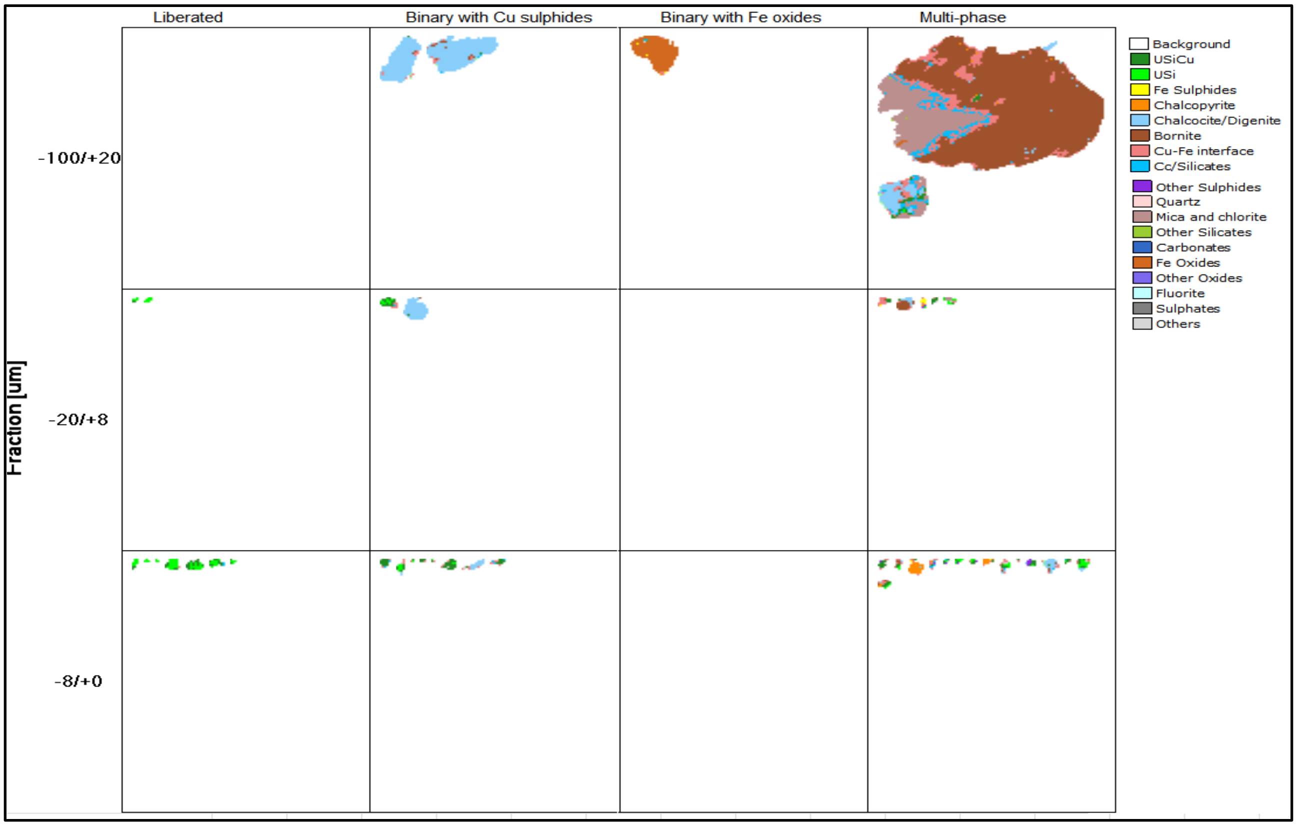

Figure 3. Image grid of the U-bearing particles sorted according to the size fraction from which they were measured and the texture they exhibit

In general, more U-bearing particles (green-colored) were present in the -8µm compared to the coarser fractions. U particles in the coarsest fraction (+20µm) were present as U-Cu silicate and U-Pb oxide. U-Pb oxide was fully locked in haematite, while U-Cu silicate was locked in the boundary between Chalcocite and Muscovite (refer Figure 4) as well as chalcopyrite-chalcocite multiphase. With the exception, of one UPbOx-FeOx particle, U recovered in the +20µm size range is a result of association with chalcocite and chalcopyrite. In comparison, the finer size fractions contained both liberated and locked particles indicating co-existence of different recovery mechanisms.

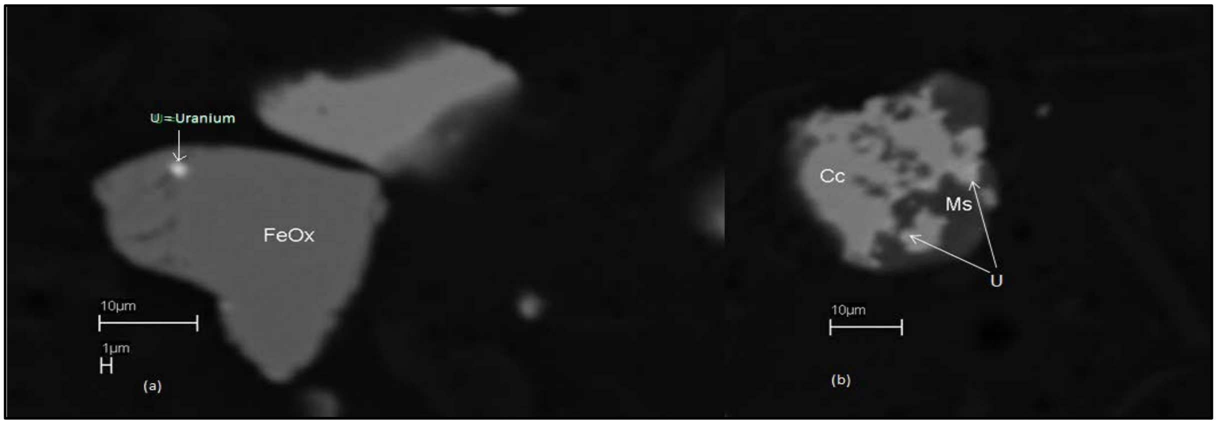

Figure 4. BSE image of U-bearing particles in the +20 um size fraction (a) U locked in haematite with magnification 1200X and (b) U on the boundary between Chalcocite (Cc) and Muscovite (Ms) with magnification 900X

The detailed liberation analysis of the U-bearing minerals is provided in Table 5. In the intermediate size (-20+8µm), about 22% are liberated particles while about 70% are high-grade middlings (60-90% exposed surface) and mainly associated with Chalcocite or Chalcopyrite. It is interesting, to note that the lesser copper sulphide surfaces (<40%) imparted sufficient hydrophobicity to enable their recovery. Finer grinding is the obvious option to liberate the uranium minerals from this kind of particle, in order to prevent their recovery.

Table 5. QEMSCAN Liberation analysis of U-bearing minerals in the 3rd cleaner concentrate

Size (µm)	**Areal Grade (%)**											
	≤10 (%)	**10-20 (%)**	**20-30 (%)**	**30-40 (%)**	**40-50 (%)**	**50-60 (%)**	**60-70 (%)**	**70-80 (%)**	**80-90 (%)**	**90-100 (%)**	**100 (%)**	**Total**
+20	100	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	**100**
-20,+8	3.2	5.0	0.0	0.0	0.0	0.0	17.2	52.6	0.0	0.0	22.0	**100**
-8	2.1	0.0	0.0	7.7	7.6	9.9	17.6	11.4	24.7	10.9	8.1	**100**
Combine	16.15	2.16	0.0	3.34	3.26	4.29	14.99	27.5	10.68	4.71	12.9	**100**

In the -8µm size fraction, approximately 43% of U are essentially liberated and less than 3% recovered in floatation fully locked. The rest (about 54%) are in middling mainly with Chalcocite, Chalcopyrite and to a lesser extent, silicates. Unlike the U-Th and U-Ti oxides which were fully liberated, the dominant UCuSi and USi particles were present in varying forms; fully liberated as well as in middling reflecting the nature of mineralization. Nonetheless, the recovery of the liberated U bearing particles is believed to be predominantly by entrainment.

Metallurgical Implications

The distribution of minerals in the 3rd cleaner concentrate (monthly composite) is shown in Figure 5. In general, only about 8% of the total minerals in the concentrate are found in the -8µm size range, of which copper minerals overall is less than 5%. Interestingly, U minerals distribution revealed a contrasting trend with their proportion increasing with decreasing particle size.

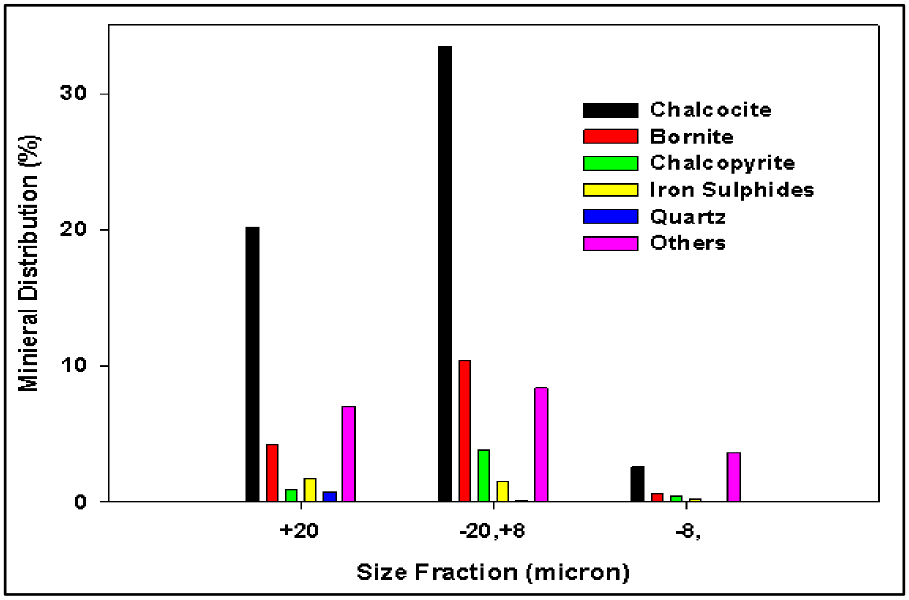

Figure 5. Distribution of minerals in the 3rd cleaner concentrate

Figure 6 depicts the metallic distribution of uranium and copper relative to the size. While there exist little difference in the amount of U between the intermediate size (-20+8um) and finest size (-8um) fractions where over 90% of uranium is distributed, what is significant is the proportion of Cu relative to U in the -8µm size range.

Relatively, more U (46.2%) than Cu (5.9%) is present in this finest fraction, which accounts for about 8% of the total mass of the sample. This result suggests that any rejection of this fraction may significantly reduce U in the final concentrate, at the expense, however, of reducing copper recovery by approximately 6%.

Future work will investigate the relative recovery mechanisms in the Jameson cell, which represents 50% of the final concentrate.

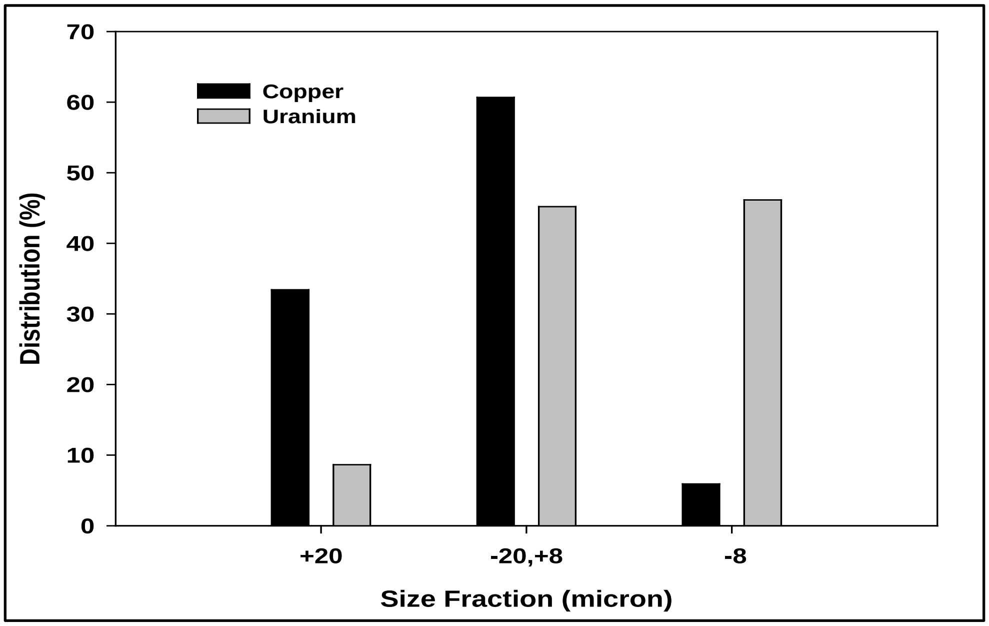

Figure 6. Distribution of metallic Cu and U in the 3rd cleaner concentrate

Possible factors influencing U recovery

While the recovery, of uranium obviously does have links to its association with Cu minerals (liberation issue), other mechanisms (e.g. Entrainment) are suggested by this study. Recovery of the liberated U particles (22% in -20+8µm and ~43% in -8µm) is possibly related to water recovery (entrainment). Several studies (Trahar and Warren, 1976; Zheng *et al*, 2006; Neethling and Cilliers, 2009; Yianatos and Contreas, 2010) found, that entrainment is more pronounced in particle size ranges less than 20 microns. The significant recovery of liberated silicates (refer Table 6) also affirms such unselective behavior.

Table 6. Liberation characteristics of silicates

Size Fraction	**Areal grade (%)**											
(µm)	**≤10 (%)**	**10-20 (%)**	**20-30 (%)**	**30-40 (%)**	**40-50 (%)**	**50-60 (%)**	**60-70 (%)**	**70-80 (%)**	**80-90 (%)**	**90-100 (%)**	**100 (%)**	**Total**
+20	2.3	3.69	2.53	2.16	2.91	4.53	6.44	9.18	12.21	18.32	8.07	72.36
-20,+8	0.68	1.00	0.89	0.96	1.45	1.32	1.68	2.12	2.85	2.83	8.90	24.67
-8	0.03	0.17	0.16	0.21	0.39	0.11	0.18	0.22	0.11	0.09	1.28	2.97
Combine	3.04	4.86	3.59	3.33	4.75	5.97	8.29	11.52	15.16	21.24	18.2	100.0

On other hand, the recovery of iron oxide containing U-particles in the +20µm size range (refer Figure 4) raises another question of whether recovery of such a particle is through entrainment or true floatation. Work is required to assess if Pb and possibly Cu in the U/Fe-Ox particle are enhancing uranium floatation. A study by Makanza *et al* (2008) found, that floatation of uraninite and brannerite in a Witwatersrand gold ore was activated by Pb in the uranium minerals. For this particle, the surface exposure of the U particle is small compared to that of iron oxide, limiting such phenomena; however, if similar particles will be found in further analysis (e.g. Jameson concentrate), Pb/Cu enhanced floatation will be considered a valid hypothesis for Prominent Hill ore as well.

CONCLUSIONS

Liberation and entrainment are the mechanisms contributing to the recovery of U-bearing minerals at Prominent Hill. Regrinding the rougher concentrate to finer size than the current P80 (20µm) is one strategy to address the liberation issue. In this way, U which was floated, in the roughers, due to the association with the Cu sulphides can be rejected in cleaning. The considerable distribution of U in the ultra- fine size fraction (-8µm) in this sample suggests, that entrainment is also a contributing factor. However, it is important to establish the relative contributions of each of these mechanisms before any practically applicable strategy is considered.

Further, QEMSCAN studies on the Jameson cell concentrate, and feed and plant survey activity will be carried out, to assess the relevance of these U recovery mechanisms and determine the best operating strategy for maximizing U rejection. Further, QEMSCAN studies on the Jameson cell concentrate and feed and plant survey activity will be carried, out to assess the relevance of these U recovery mechanisms and determine the best operating strategy for U rejection.

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