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EPMA characterisation of strüverite from *amang* of Peninsular Malaysia

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Abstract: The heavy minerals from tin tailings or *amang* comprises a wealth of minerals which contain some of the most sought after metals in industry that includes tantalium and niobium, which are widely used in the computer and cellular phone industry because of their high reliability, high melting points and corrosion resistance characteristics. An extended investigation was carried out on the EPMA (Electronprobe Microanalyzer) into the complex nature of strüverite, the source of tantalium (Ta) and niobium (Nb) in *amang*, in particular its complex mineralogy, chemistry and associated phases/minerals. EPMA data of strüverite show that it is a complex intergrowth of rutile (TiO_2), tantalite (Ta_2O_5) and columbite (Nb_2O_5) with only very few grains devoid of any inclusions or exsolved phases. EPMA analysis of three "pure" strüverite grains, devoid of intergrowths, gave consistent ratios of Ti (19.58–29.08 wt%), Nb (5.45–7.99 wt%), Ta (25.43–33.46 wt%) and Fe (6.89–8.11 wt%). They also contain significant amounts of Sn (0.79–2.62 wt%) and Th (3.25–6.03 wt%) but very low W (0.11–0.47 wt%) and Mn (0.01–0.12 wt%) and no Y. Other strüverite grains which have inclusions or exsolved phases or intergrowths, all show very variable contents of Ti, Nb, Ta, Fe, W, Mn, Th and Y. BSE (backscattered secondary electron) images on the EPMA also picked up zoned strüverite crystals where great variations in compositions are clearly discernible in X-ray maps of these zoned crystals. EPMA analysis of fusion discs of strüverite concentrates from *amang* also show quite variable contents for Ti (12.60–54.38 wt%), Nb (1.71–13.12 wt%), Ta (2.88–7.40 wt%), Fe (1.43–15.65 wt%), W (0–3.48 wt%), Mn (0.05–1.51 wt%) and Th (0–29.76 wt%). The presence of Th with or without Y in some strüverite grains could be a useful criteria for determining their source area. Knowledge of the complexity of strüverite is useful in configuring the correct recovery processes for Ta and Nb.

INTRODUCTION

Strüverite, the ore mineral for tantalum and niobium, is a tantalum-niobium rich rutile mineral found in heavy mineral discards or *amang* after the tin ore, cassiterite, has been extracted (Wan Fuad,1989). It was first discovered in 1907 by Zambonini in Craveggia and Piemont, Italy and named after Giovanni Strüver, a mineralogist at University of Rome, Italy (Fleischer, 1983). Strüverite $(Ti,Ta,Fe^{3+})_3O_6$ is the tantalum-rich endmember of a series in which ilmenorutile $(Ti,Nb,Fe^{3+})_3O_6$ is the niobium-rich endmember.

The first discovery of "strüverite" (Ta-Nb rutile) in Malaysia was made in 1909 in alluvial grains from a tin mine near Salak North, in Perak (Crook and Johnston, 1912). An extended re-examination of the strüverite sample was carried out by Flinter (1959), which included physical and optical characteristics, polished section examination and X-ray powder data.

In Malaysia, Beh Minerals Sdn.Bhd. was the first *amang* factory to separate strüverite from other *amang* minerals. The mineral is mainly found in the Kinta Valley in localities like Bidor, Kampar, Gopeng and Papan (Figure1). The strüverite occurrences appear to decrease and disappear towards the south of the Kinta Valley so that in the Kuala Lumpur tinfields, except for some isolated occurrences, it is virtually absent (Wan Fuad, 1994).

The characterisation of strüverite in this study deals in great depth with the complex nature of the mineral, in particular its complex mineralogy and chemistry under the electronprobe microanalyzer or EPMA. The paper describes the complex nature of strüverite and the various phases/minerals associated with it. The composition of these various phases/minerals and their possible origins or source areas are also discussed.

METHOD

Samples were picked out by the EPMA from the study of polished sections of *amang* from the various localities in the Kinta Valley. Strüverite for this study was also obtained from BEH Minerals Sdn. Bhd., an *amang* retreatment factory in Menglembu, Ipoh. The BEH strüverite samples are believed to be a mixture of strüverite from various areas in the Peninsula.

Initial observations were made on the binocular and petrographic microscopes before further study was made using the XRD and EPMA techniques.

The overall composition of strüverite concentrates were determined from prepared fusion discs on the EPMA. Besides full elemental composition of the various associated phases/minerals, X-ray mapping was also performed to give a better picture of the distribution of the main elements, namely, Ta, Nb, Ti, W, Fe, in these strüverite associated phases/minerals (Teh and Idawati, 2002). The EPMA analysis results were normalised as it was found that the normalised values are more appropriate in the characterisation of the samples studied.

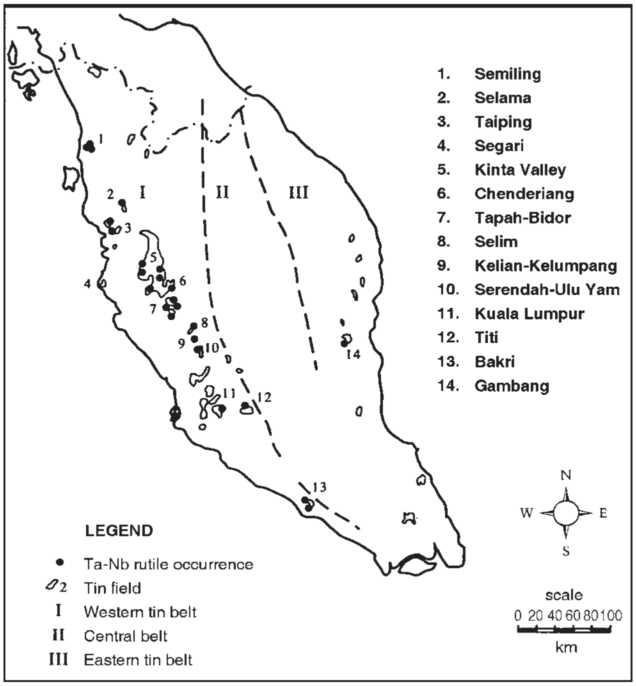

Figure 1: Map of Peninsular Malaysia showing the struverite (Ta-Nb rutile) occurrences (Wan Fuad, 1994). Samples for this study are from Bidor, while theBEH samples are from various areas in the Peninsula.

The EPMA is in essence a Scanning Electron Microscope (SEM) with precise analytical capabilities. The EPMA available at the Geology Department, University of Malaya is a highly-automated Cameca SX100, which is workstation-based with full instrument control and qualitative, quantitative software via windows and multi-task user environment. The EPMA utilises a fully integrated energy dispersive spectrometer (EDS) for fast full spectrum scans of elemental compositions and 4 conventional wavelength dispersive spectrometers (WDS) with 12 analysing crystals for accurate composition determinations.

RESULTS

Under the binocular microscope, strüverite is a greyish to yellowish black, sometimes black mineral (Figure 2). Strüverite crystallizes in the tetragonal system and common habits are prisms and bipyramids. In fact, strüverite is physically not much different from rutile except for differences in their chemical properties.

EPMA BSE (backscattered electron) images of strüverite grains show a complex intergrowth of rutile (TiO_2), columbite (Nb_2O_5) and tantalite (Ta_2O_5) (Figure 3). EPMA data shows that only a few grains of strüverite were devoid of any inclusions or exsolved phases. Three of such "pure" strüverite grains gave quite consistent ratios of Ti (19.57–29.08 wt%), Nb (5.45–7.99 wt%), Ta (25.43–33.46 wt%) and Fe (6.89–8.11 wt%)(Table 1 and Figure 4). They also contain significant amounts of Sn

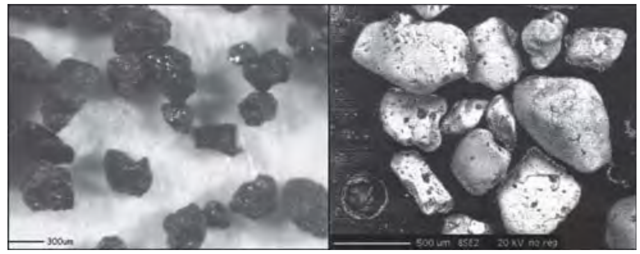

Figure 2: Under the binocular microscope (left), strüverite is a greyish to dark grey, sometimes black mineral. EPMA BSE image (right) show strüverite grains crystallizing in the tetragonal system and common habits are prismatic and bipyramidal tetragons. Scale bar is 0.3 mm.

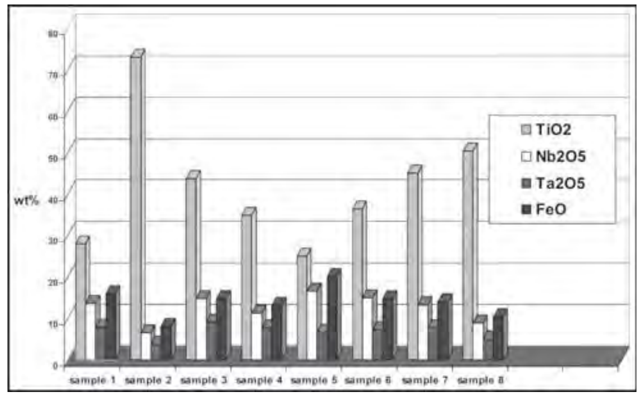

Figure 3: EPMA BSE images showing the complex nature of strüverite with numerous inclusions, exsolutions, intergrowths and even zonations. The top row shows rather 'pure', clean strüverite grains (white).

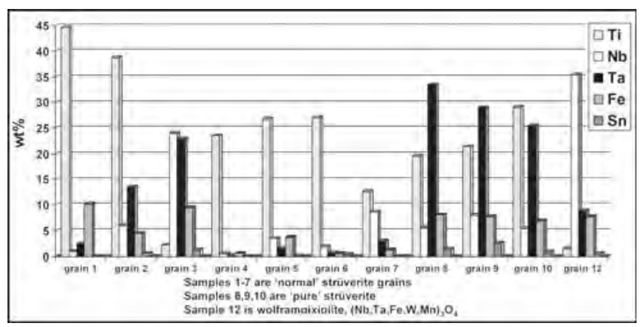

Figure 4: Chart showing variable contents of Ti, Nb, Ta and Fe in struverite concentrates.

(0.79–2.62 wt%) and Th (3.25–6.03 wt%) but very low W (0.10–0.47 wt%) and Mn (0.01–0.12 wt%) and no Y.

Other strüverite grains which have inclusions or exsolved phases (Figure 6) all show very variable contents of Ti, Nb, Ta, Fe, W, Mn, Th and Y.

In addition, the Bidor strüverite sample has exsolution bodies of wolframoixiolite $(Nb,W,Ta,Fe,Mn)_3O_4$ a black, opaque, monoclinic mineral, occurring together with columbite/tantalite (Figure 7).

BSE images on the EPMA also picked up zoned strüverite crystals where great variations in compositions are clearly discernible in X-ray maps of these zoned crystals (Figures 8 and 9).

EPMA analysis of fusion discs made of the strüverite concentrates also show more variable contents for Ti

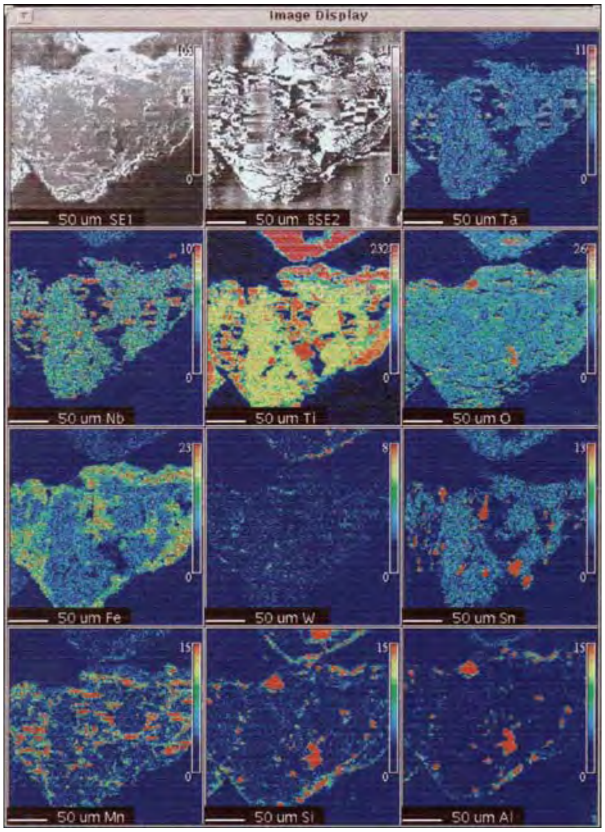

Figure 5: Chart showing the variation in Ti, Nb, Ta, Fe, and Sn contents of struverite grains.

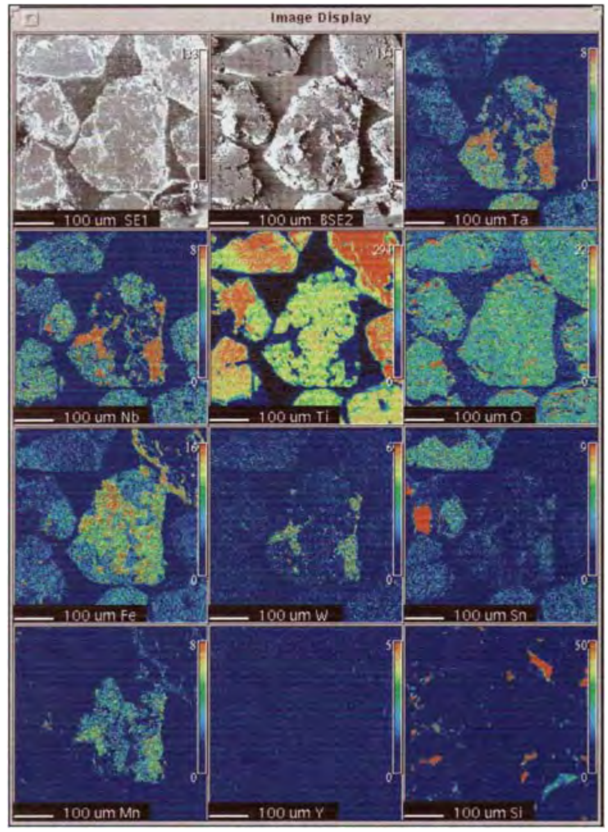

Figure 6: EPMA X-ray map of strüverite grain showing exsolved bodies rich in Ta, Nb, Ti, Sn, Mn and Fe with a rim of ilmenite ($FeTiO_3$). Sample BEH10.

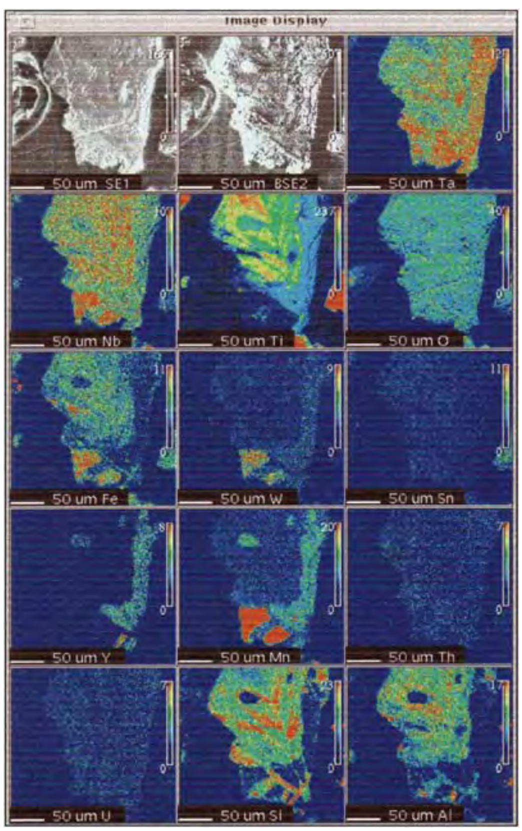

Figure 7: X-ray map showing strüverite grain with exsolved columbite (Nb_2O_5), tantalite (Ta_2O_5) and wolframoixiolite $(Ta,Nb,Fe,W,Mn)_3O_4$. Sample Bidor.

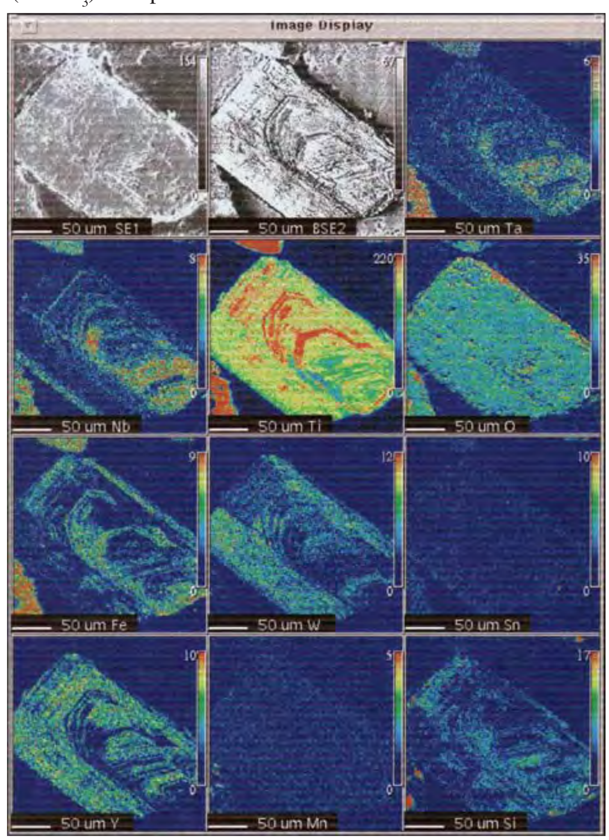

Figure 8: EPMA X-ray map of zoned strüverite showing the distribution of Ta, Nb, Ti, Fe, W, Sn, Mn and Y within zones in the grain. Sample BEH struv2.

Table 1: Results of EPMA analysis of strüverite concentrates prepared as fusion discs.

Element	Norm. Conc (wt%)										
	1	2	3	4	5	6	7	8	9	12	13
Ti	16.82	43.76	26.26	20.92	15.01	21.83	27.07	30.23	12.61	29.68	16.66
Nb	10.85	5.27	11.80	9.01	13.12	11.93	10.61	7.04	9.40	8.84	12.35
Ta	6.55	3.07	7.40	6.32	5.54	6.01	6.30	4.17	4.22	2.88	5.94
Si	1.93	0.73	0.85	1.05	2.23	0.76	0.89	0.76	1.28	1.52	2.72
Fe	12.46	6.39	11.57	10.34	15.65	11.47	10.95	8.11	10.21	12.53	12.76
Sn	7.48	3.60	6.39	5.66	7.40	5.82	5.93	4.36	6.43	7.46	12.11
W	1.88	0.31	2.44	0.81	2.52	1.71	1.88	0.59	0.00	2.86	3.48
Al	1.50	0.84	0.93	0.85	1.48	1.03	1.71	1.00	1.53	1.31	1.50
Cr	0.00	0.26	0.00	0.00	0.00	0.10	0.00	0.06	0.06	0.00	0.02
Zr	0.27	0.00	0.73	0.29	0.22	0.44	0.03	0.35	0.05	0.00	0.00
Mn	1.11	0.31	1.09	1.03	0.38	1.08	1.17	0.65	0.61	1.51	1.16
Zn	0.81	0.00	0.50	0.00	0.00	0.44	0.22	0.00	0.52	0.00	0.30
As	0.00	0.00	0.00	0.48	0.00	0.00	0.00	0.05	0.34	0.00	0.00
Th	11.99	0.00	0.44	16.78	10.20	10.08	3.18	12.72	29.76	0.00	3.77
O	26.36	35.46	29.60	26.47	26.24	27.30	30.04	29.88	22.96	31.42	27.21
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Oxide	Norm. Conc (wt%)										
	1	2	3	4	5	6	7	8	9	12	13
TiO_2	28.08	72.99	43.81	34.91	25.06	36.41	45.16	50.43	21.03	49.51	27.79
Nb_2O_3	13.65	6.64	14.85	11.33	16.51	15.02	13.36	8.86	11.83	11.12	15.55
Ta_2O_5	8.00	3.75	9.04	7.71	6.77	7.33	7.69	5.09	5.14	3.52	7.25
SiO_2	4.11	1.56	1.82	2.26	4.78	1.62	1.91	1.63	2.74	3.25	5.83
FeO	16.02	8.22	14.88	13.30	20.13	14.76	14.09	10.43	13.14	0.00	16.41
SnO	8.49	4.09	7.25	6.42	8.39	6.61	6.73	4.95	7.30	16.12	13.75
WO_3	2.37	0.39	3.08	1.01	3.17	2.16	2.38	0.75	0.00	8.47	4.39
Al_2O_3	2.84	1.59	1.77	1.61	2.80	1.95	3.24	1.89	2.89	3.61	2.84
Cr_2O_3	0.00	0.37	0.00	0.00	0.00	0.15	0.00	0.09	0.09	2.47	0.02
ZrO_2	0.36	0.00	0.98	0.39	0.29	0.59	0.05	0.48	0.08	0.00	0.00
MnO	1.42	0.40	1.41	1.33	0.49	1.40	1.51	0.84	0.79	0.00	1.50
ZnO	1.02	0.00	0.62	0.00	0.00	0.54	0.28	0.00	0.65	1.94	0.38
As_2O_3	0.00	0.00	0.00	0.63	0.00	0.00	0.00	0.07	0.46	0.00	0.00
ThO_2	13.64	0.00	0.49	19.09	11.61	11.47	3.61	14.48	33.87	0.00	4.30
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Table 2: Results of EPMA analysis of various strüverite grains.

Element	Norm. Conc (wt%)										
	1	2	3	4	5	6	7	8	9	10	12
Ti	44.60	38.74	2.17	23.49	26.79	27.00	12.68	19.58	21.34	29.08	1.53
Nb	0.88	6.02	24.04	0.49	3.46	1.80	8.69	5.61	7.99	5.45	35.38
Ta	2.40	13.50	22.84	0.12	1.53	0.65	2.99	33.46	29.00	25.43	8.85
Si	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe	10.19	4.49	9.45	0.62	3.72	0.57	1.26	8.11	7.73	6.89	7.77
Sn	0.00	0.60	1.26	0.04	0.08	0.35	0.03	1.36	2.62	0.79	0.50
W	0.14	0.17	10.38	15.62	1.44	10.69	0.60	0.38	0.47	0.11	14.29
Al	0.51	0.01	0.01	0.00	0.58	0.00	0.34	0.00	0.06	0.00	0.01
Mn	1.96	0.01	4.49	0.00	0.01	0.00	0.01	0.07	0.12	0.01	8.97
Th	4.19	4.06	5.05	14.35	36.29	10.22	53.48	6.03	4.54	3.25	1.69
Y	0.00	0.00	0.00	18.26	0.00	20.21	0.27	0.00	0.00	0.00	0.00
O	35.11	32.40	20.31	27.01	26.11	28.51	19.65	25.39	26.13	29.01	21.00
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

(12.61–54.38 wt%), Nb (1.71–13.12 wt%), Ta (2.88–7.40 wt%), Fe (1.43–15.65 wt%), W (0–3.48 wt%), Mn (0.04–1.51 wt%) and Th (0–29.76 wt%) (Table 2 and Figure 5). This further confirms the complex nature of strüverite.

The occurrence of strüverite with the heavy minerals normally associated with tin mineralisation suggests that the origin of strüverite is in the same quartz veins and pegmatite lenses that are associated with cassiterite, wolframite, tantalite, columbite, rutile and ilmenite.

CONCLUSION

The rutile-columbite-tantalite ore, strüverite, has very variable contents of Ti, Nb, Ta, as well as Fe, W, Sn, Mn and Th. This inhomogeneity is due largely to its very complex nature of intergrowths, including zonation, of a number of minerals, inclusions and exsolved phases.

Even the various exsolved phases and associated minerals have very variable Ti, Ta, Nb, Fe and W contents. This shows that besides strüverite the *amang* concentrates also contain other associated minerals like ilmenorutile, rutile, columbite, tantalite, ilmenite and wolframoixiolite.

The presence of Th with or without Y in some strüverite grains would be useful criteria to look into greater detail for determining their specific source areas other than the tin-bearing quartz veins and pegmatite lenses in mineralised granites in particular in the Kinta Valley and Klang Valley.

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Funds from the University of Malaya for this research in the form of Peruntukan Penyelidikan Fundamental (FP051/2005D) and Project Science Fund (14-02-03-4007) are greatly appreciated. Strüverite samples from BEH Minerals Sdn. Bhd. are highly appreciated.

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