

KOBEITE FROM PARINGA RIVER, SOUTH WESTLAND, NEW ZEALAND

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ABSTRACT

A tantalum-niobium-titanium-bearing mineral has been found as slender prismatic crystals in a cobble of graphic granite in gravels of the Paringa River, South Westland, New Zealand. The mineral is dark brown in color and metamict with the characteristic resinous luster and strikingly conchoidal fracture of a mineral in that physical state. The refractive index is 2.205 but after being heated to 1200° C. in vacuo a value of 2.35 was recorded; specific gravity is close to 5. X-ray diffraction patterns secured from unheated and carefully heated fragments are either identical to or exhibit insignificant differences from powder photographs obtained with similarly treated kobeite from the type locality of Shiraiishi, Kyoto Prefecture, Japan. On the basis of these details but in the absence of quantitative analytical data, the South Westland mineral is considered to be identical, or very closely related to, kobeite. The source of the pebble is uncertain but it has probably been derived from granitic stocks or pegmatites that are known to outcrop in this general area.

OCCURRENCE

The kobeite-bearing cobble, 75 mm. in diameter, was collected while panning sands and gravels in the Paringa River, South Westland, New Zealand, at a point approximately $\frac{3}{4}$ of a mile upstream from the bridge on the Weheka-Paringa highway. The mineral occurs as very dark brown prismatic crystals that extend intermittently over distances of 30–45 mm. with a sheaf-like or radiating arrangement (Figs. 1 and 2) that rather strikingly resembles Figs. 1*a* and 1*b* in Takubo, Ukai, and Minato's (1950) paper on kobeite. The matrix consists of a coarse aggregate of oligoclase and microcline with a rude micrographic relationship; very rare quartz, epidote, muscovite, and chlorite complete the mineral assemblage.

MINERALOGY

In transmitted light the mineral is deep coffee brown, sometimes semi-opaque, and by reflected light a bright resinous luster and strongly conchoidal fracture surfaces are evident. Crystals are either long prismatic when they are clear and transparent, or stumpy and irregular in form with a tendency to exhibit irregularly distributed areas of much darker brown color; these zones may be shown to be much more radioactive than the more translucent portions of the crystals (Fig. 3). This situation is not dissimilar from that found for manganoan allanite from Yosemite National Park (Hutton, 1951, p. 242), and several other localities (Marble, 1948, pp. 80–85). Crystals are coarsely striated parallel to their lengths and pronounced cross-fracture is evident in thin section and crushed material.

The mineral is metamict and under strong illumination an almost imperceptible birefringence may be detected. The refractive index, measured several times in sulfur-selenium glasses, has an average of 2.205 ± 0.005 . After the mineral has been heated at 1200° C. for $2\frac{1}{2}$ hours in vacuo the refractive index increases to 2.35 ± 0.01 , and at the same time a strong aggregate birefringence becomes evident. The mineral exhibits

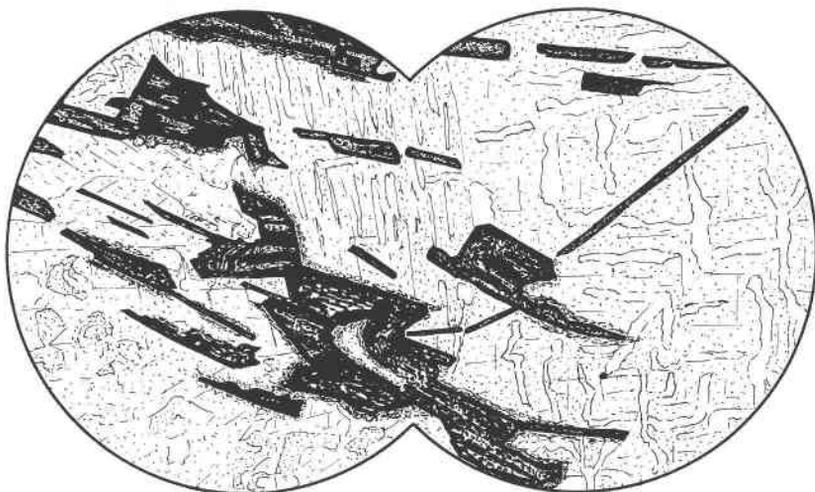


FIG. 1. Regularly arranged prismatic crystals of kobeite in a matrix of micrographically related microcline and oligoclase from Paringa River, South Westland, New Zealand. Magnification $\times 65$.

no superficial alteration¹ as does Japanese kobeite, but boundary zones between crystals and enclosing feldspar are very often distinctly stained brown, probably as a result of oxidation of iron freed from the mineral during metamictization. Owing to the limited amount of material available, specific gravity determinations have not been successfully carried out beyond noting that particles appeared to sink after twelve hours suspension in a melt of thallos formate and thallos malonate; accordingly one may only report that the specific gravity of the Paringa mineral is probably slightly in excess of 5.

¹ An x -ray powder film of the yellow alteration material that surrounds crystals of Japanese kobeite indicates that anatase is the chief constituent with line diffuseness that suggests extremely fine particle size; slightly larger d -spacings than those recorded for pure anatase (Swanson and Tatge, 1953, p. 46) may be attributed to ionic substitution of Ti^{4+} ions by the larger Ta^{5+} and Nb^{5+} ions. A few additional rather strong lines present are not attributable to associated minerals.

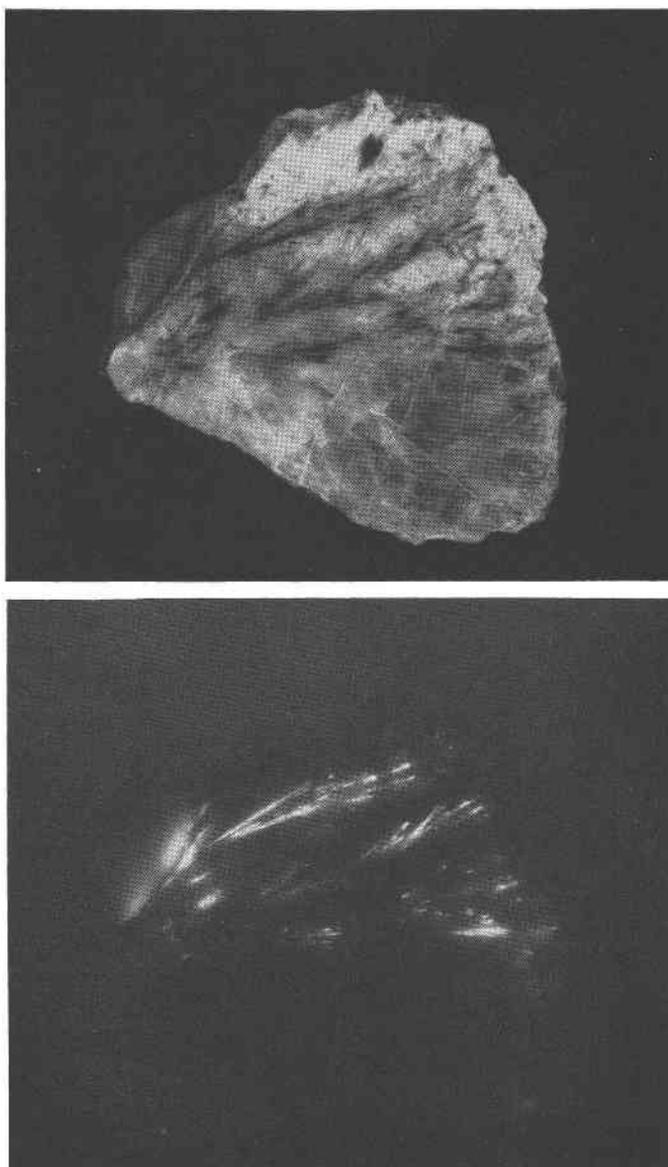


FIG. 2 (top). Polished surface of micrographic granite that exhibits radiating aggregates of prismatic crystals of kobeite from Paringa River, South Westland, New Zealand. Dimension from top to bottom of the rock surface is 70 mm.

FIG. 3 (bottom). Autoradiograph of Paringa River kobeite as exposed on polished surface illustrated in Fig. 2. Exposure period was 72 hours.

CHEMICAL COMPOSITION

It is unfortunate that scarcity of material permitted only qualitative chemical tests to be made. Rare earths, tantalum, niobium, and titanium were found to be present in considerable amounts, and the color of the flocculent tannin-complex precipitate devoid of titanium obtained by leaching potassium bisulphate fusions with 1 per cent tannin solution in 5% H_2SO_4 was reddish-orange in color, a condition that points to dominance of niobium over tantalum. The presence of iron, oxidation state unknown, aluminum, uranium, thorium, and water was verified.

The Paringa River mineral is apparently insoluble in hot concentrated HCl after long treatment, but some reaction is evident with hot concentrated H_2SO_4 . It is readily soluble in fused potassium bisulphate. With a concentrated oxalic acid solution at about 100° C. no noticeable effect was evident with either the Paringa River mineral or with Japanese kobeite. On the other hand, 5 minutes treatment with 1:1 HF in the cold caused slight iridescent films to develop on the bright conchoidal fracture surfaces of the Paringa River mineral, and thin greyish-white opalescent crusts appeared rather patchily developed on Japanese kobeite. With cold concentrated HF the effect was only very slightly more evident in each instance. In their reactivity towards oxalic acid and HF these two minerals differ from what is usually found for polycrase-euxenite, eschynite-priorite, fergusonite, or samarskite, since considerable reaction occurs with the latter minerals.

X-RAY STUDY

Powder cameras of 114.59 mm. diameter and nickel-filtered copper radiation were employed exclusively in this work; and in addition it should be understood that a single fragment, 0.1–0.2 mm. in diameter, mounted on a glass fiber was used in each instance as the diffraction material rather than powdered specimens.

Pure fragments of the Paringa River mineral and kobeite from Japan have been subjected to heat-treatment as follows: (1) Samples, in platinum microcrucibles, were placed in an already-heated furnace at 620° C. and at 1200° C.; accordingly these samples were heated in contact with air. (2) Samples were sealed in Vycor or silica capillaries in vacuo and then heated at the same temperatures as before, and in addition at 900° C. and 1000° C. All samples were *x*-rayed after having cooled to room temperature.

Paringa River material untreated in any way, gives weak, usually diffuse powder lines (Table 1, column A) after an exposure of 24 hours; this diffraction pattern, which was found to be quite reproducible, appears indicative of remnants of a primitive cubic structure, cell edge

about 14.3 Å, that may have existed before metamictization, and at the same time the utterly disoriented nature of the mineral is clear. With material heated in air and in vacuo at 620° C. new patterns are obtained that are closely similar to one another, and except for two or three exceedingly faint lines that are not permitted by the space-group, the spacings may be compared to those of a cubic face-centered uraninite-type of pattern with cell edges of 5.029 Å and 5.021 Å respectively

TABLE 1. X-RAY DIFFRACTION PATTERNS YIELDED BY UNHEATED FRAGMENTS OF THE PARINGA RIVER MINERAL (A) AND KOBEITE, JAPAN (B)
Radiation $\text{CuK}\alpha=1.5418 \text{ \AA}$. Intensities determined visually

A		B	
<i>d.</i> meas. Å	<i>I</i>	<i>d.</i> meas. Å	<i>I</i>
4.50	<1	4.50	<1
4.16	3	4.16	3
3.75	1	3.74	1
3.50	<1	3.50	<1
3.32	<1	3.35	<1
3.20	1	3.13	1
—	—	2.55	<1
—	—	2.18	<1
—	—	2.10	<1
—	—	1.74	<1

(Tables 2 and 3). Careful comparison of these films with powder patterns of reverted microlite reveals an absence of several fairly strong lines of the latter in the former; consequently the patterns of the Paringa River mineral are considered to be more comparable to a uraninite-type rather than to that of microlite-pyrochlore with cell edges of 10.058 Å and 10.042 Å in which the strongest lines only are developed. It is interesting to note that heating in contact with air has permitted a slightly larger cell to develop; this increase in size is probably due to oxygen intake during the heating process.

After being heated in air or in vacuo at 1200° C. fragments of the Paringa River material give distinctive x-ray diffraction patterns that are closely similar to one another (Table 4); particle size has increased because most of the lines are now quite sharp. In each case the three strongest lines of the uraninite-type pattern, viz. (111), (200), and (311), persist in the new patterns, although not at the same relative intensities, whereas the strong reflection (220) appears to coincide with or be very

close to a line that is usually almost imperceptible in the new patterns. Attempts to index these films systematically were unsuccessful and this fact leads one to the conclusion that two or more phases may be present, although the degree of simplicity in the patterns may be an argument

TABLE 2. X-RAY DIFFRACTION POWDER PATTERNS OF PARINGA RIVER MATERIAL AND KOBEBITE, JAPAN

All samples heated for one hour at 620° C. Radiation $\text{CuK}\alpha = 1.5418 \text{ \AA}$

A		B		C		D	
<i>d.</i> meas. \AA	<i>I</i>						
4.15	1	4.15	1	4.15	<1	4.14	1
3.76	<1	3.74	1	—	—	3.76	<1
2.89	10	2.89	10	2.90	10	2.89	10
2.505	4	2.508	5	2.514	5	2.506	4
1.777	8	1.772	8	1.778	8	1.776	8
1.516	7	1.513	7	1.517	7	1.514	7
1.453	1	1.447	1	1.451	1	1.452	1
1.255	1	1.254	1	1.256	1	1.257	1
1.154	2#	1.153	3#	1.156	3#	1.154	3#
1.125	2#	1.124	3#	1.126	3#	1.126	3#
1.028	1#	1.026	2#	1.028	2#	1.027	2#
.969	1#	.967	2#	.968	2#	.969	2#
—	—	.888	<1	.890	<1	—	—
.851	<1#	.850	2#	.850	3#	.851	2#
.838	2#	.839	3#	.838	1#	.838	2#

Diffuse lines.

- A. Kobeite, Japan, heated in vacuo.
- B. Paringa River material, heated in vacuo.
- C. Paringa River material, heated in air.
- D. Kobeite, Japan, heated in air.

against this suggestion. Certainly there was no indication of any lines due to rutile, brookite, or pseudobrookite.

The *x*-ray films obtained with the Paringa River mineral after being heated to 1200° C. have been compared with those obtained from a large number of multiple oxides of tantalum, niobium, titanium, and rare earths, but this provided no clue as to the true identity of the mineral in question, although it may be pointed out that a number of minerals belonging to this general group do give *x*-ray powder patterns of the uraninite-type after being heated to 620° C. in vacuo or in air for $\frac{3}{4}$ –1 hour. Among these are yttriotantalite, eschynite, brannerite, and some

euxenites, including a specimen of Simpson's (1951, pp. 257-259) tant-euxenite from Cooglegong, Pilbara Goldfield, Western Australia.²

However, kobeite from the type locality—Shiraishi, Kobe-mura, Kyoto Prefecture, Japan—gives x -ray patterns without being heated,

TABLE 3. INDEXING OF POWDER PATTERNS OF PARINGA RIVER MINERAL

<i>hkl</i>	A		B	
	Heated in air		Heated in vacuo	
	<i>d</i> , Å	<i>a</i> ₀	<i>d</i> , Å	<i>a</i> ₀
(111)	2.90	5.027	2.89	5.012
(200)	2.514	5.030	2.508	5.015
(220)	1.778	5.027	1.772	5.011
(311)	1.517	5.030	1.513	5.018
(222)	1.451	5.025	1.447	5.010
(400)	1.256	5.025	1.254	5.014
(331)	1.156	5.036	1.153	5.027
(420)	1.126	5.036	1.124	5.024
(422)	1.028	5.034	1.026	5.026
(511)	.968	5.029	.967	5.025
(440)	.890	5.032	.888	5.027
(531)	.850	5.028	.850	5.032
(600)	.838	5.027	.839	5.034

Exceedingly faint lines, one in A and two in B, that are not permitted by the space-group are omitted.

A. Average $a_0 = 5.029$ Å.

B. Average $a_0 = 5.021$ Å.

and after being heated in air or in vacuo, that are almost identical with those yielded by the Paringa River mineral under corresponding circumstances. This is doubly interesting since Takubo, Ukai, and Minato (1950, p. 511) have reported that after kobeite is heated to 900° C. for 30 minutes hardly any signs of recrystallization were evident when the mineral was subsequently examined optically and by x -ray diffraction methods. Unheated kobeite gives a weak pattern (Table 1, column B) that is identical with that yielded by the Paringa River mineral except for the presence of a few additional exceedingly faint, diffuse lines at

² Arnott (1950, pp. 389, 399) obtained a similar pattern for tanteuxenite from Eleys, Western Australia, but he does not record the temperature at which the specimen was heated, nor the length of time of heating. The present writer has determined that if tanteuxenite is heated in vacuo at 1200° C. for 1 hour a typical euxenite-type pattern (Arnott, 1950, pp. 396-397; Berman, 1955, pp. 817-818) is obtained by x -ray diffraction.

TABLE 4. X-RAY DIFFRACTION POWDER PATTERNS FOR PARINGA RIVER MATERIAL AND KOBEITE, JAPAN

All specimens heated for 1 hour at 1200° C. Radiation $\text{CuK}\alpha=1.5418 \text{ \AA}$

A		B		C		D	
<i>d.</i> meas. \AA	<i>I</i>						
5.55	1	5.55	<1	5.46	<1	5.46	<1
4.15	1	4.13	<1	4.13	<1	4.13	<1
—	—	3.74	<1	3.71	<1	3.71	<1
3.59	1	—	—	3.61	<1	3.61	1
3.29	1	—	—	—	—	—	—
3.06	1	3.06	<1	3.07	<1	3.07	1
2.92	10	2.92	10	2.91	10	2.91	10
—	—	2.844 ¹	3	—	—	—	—
2.781	3	2.789	3	2.781	3	2.781	3
2.564	<1	—	—	—	—	—	—
2.501	4	2.500	4	2.500	4	2.495	4
2.417	<1	—	—	—	—	2.413	<1 [#]
—	—	—	—	—	—	2.355	<1 [#]
2.286	2	2.286	2	2.281	2	2.274	2
2.089	<1	—	—	2.084	<1	—	—
1.884	<1	—	—	—	—	1.880	<1
1.809	7	1.811	6	1.806	6	1.806	7
1.770	<1	1.770	<1	1.768	<1	1.767	<1
1.742	3	1.737	4	1.734	4	1.739	3
1.721	1	1.722	<1	1.715	<1	1.720	1
1.630	<1	—	—	—	—	1.630	<1
1.604	<1	—	—	—	—	1.606	<1
1.538	2	1.542	3	1.541	3	1.538	2
1.518	3	1.518	4	1.516	4	1.518	3
—	—	1.4955	<1	—	—	—	—
1.479	2 [#]	1.472	2 [#]	1.471	2 [#]	1.476	2 [#]
1.466	1 [#]	—	—	—	—	1.467	1 [#]
1.422	<1 [#]	1.426	<1	—	—	—	—
1.330	<1	—	—	1.325	<1	—	—
1.255	2	1.256	2	1.254	2	1.254	2
1.174	2	1.175	2 [#]	1.175	2 [#]	1.174	2
—	—	1.159	<1	—	—	—	—
1.141	1	1.142	2 [#]	1.141	2 [#]	1.140	2
1.118	1	1.119	1 [#]	1.117	1 [#]	1.117	1 [#]
1.109	1	1.105	1 [#]	1.104	1 [#]	1.106	1 [#]
1.046	1	1.048	1 [#]	1.046	1 [#]	1.045	1 [#]
1.031	1 [#]	1.032	1 [#]	1.033	1 [#]	1.032	1 [#]
.982	1	.981	1 [#]	.981	1 [#]	.980	1 [#]
.970	1	.969	1 [#]	.969	1 [#]	.969	1 [#]
.906	1	.906	<1 [#]	.905	<1 [#]	.905	1 [#]
.865	1 [#]	.864	1 [#]	.865	1 [#]	.865	1 [#]
.852	<1 [#]	.852	<1 [#]	.852	<1 [#]	.851	<1 [#]
.840	<1 [#]	.840	2 [#]	.839	2 [#]	.839	2 [#]

¹ Not present in most films of Paringa River Material.

Diffuse line.

A. Kobeite, Japan, heated in vacuo.

B. Paringa River mineral heated in vacuo.

C. Paringa River mineral heated in air.

D. Kobeite, Japan, heated in air.

Additional lines are present in all films but it is not possible to measure them accurately owing to faintness and diffuseness.

higher 2θ values in the film of Japanese kobeite. At the same time another film of Japanese kobeite exhibits a few elongated spots that indicate a very slight degree of order; this is almost certainly due to remnants of the original structure.

Specimens of Japanese kobeite after being heated to 620°C . for 1 hour in air or in vacuo, on most occasions yield powder patterns that are identical to one another, and to the patterns found for the Paringa River mineral after being heated in air; a few spots were observed in one film. The heating of kobeite in air did not produce an increase in d -spacings over those found for this mineral after being heated in vacuo; in this it differs from the Paringa River mineral. Comparisons of the d -spacings of kobeite and of the Paringa River mineral, both heated to 620°C . are set out in Table 2.

X-ray diffraction powder patterns secured with both kobeite and the Paringa River mineral heated to 1200°C . in air or in vacuo are distinct from those yielded at the lower temperature and a glance at Table 4 makes it clear that there are no significant differences in these patterns of the high temperature modification. Although otherwise similar to the patterns found to be constant (Table 4) one fragment of the Paringa River material, after being heated in vacuo for 1 hour at 1200°C ., yielded an additional weak reflection at 2.844 \AA and the relative intensity of the line at 1.770 \AA is of the order of 2; in one other instance it is about 5, although the 2.844 \AA reflection is missing in this case. Furthermore, two fragments of Japanese kobeite after being heated in vacuo gave X-ray films with intensities of 2 for the reflection at or close to 1.770 \AA . This line appears to correspond with and result from the persistence of the relatively strong (220) reflection found in all films of these minerals that have been heated to the lower temperature of 620°C ., but it is not clear why it should persist more distinctly in some instances and be almost absent in most, unless this situation is connected with the apparent localization of radioactive material in the mineral, a condition that is clearly observed in the radiographs of the Paringa River mineral at least. In this connection it is of interest to compare the patterns obtained for these two minerals that had undergone heating at 900°C . for 30 minutes, the conditions under which Takubo *et al.* (1950, p. 511) found no significant recrystallization had resulted in kobeite. In these films (Table 5) the uraninite-type pattern is dominant but with the strongest lines of the unique higher temperature phase or phases also present, so that the reflection close to 1.770 \AA is much more intense than those immediately on either side of it. It is interesting to observe that the reflection at approximately 4.15 \AA is the only line that persists in films yielded by

TABLE 5. COMPARISON OF X-RAY DIFFRACTION PATTERNS YIELDED BY KOBEITE, JAPAN, AFTER FRAGMENTS HAD BEEN SUBJECTED TO TEMPERATURES OF 620° C. (A), 900° C., (B), AND 1200° C. (C) IN VACUO

Radiation $\text{CuK}\alpha=1.5418 \text{ \AA}$. Intensities determined visually

A		B		C	
<i>d.</i> meas. \AA	<i>I</i>	<i>d.</i> meas. \AA	<i>I</i>	<i>d.</i> meas. \AA	<i>I</i>
—	—	5.94	<1	—	—
—	—	—	—	5.55	1
—	—	4.45	1	—	—
4.15	1	4.15	<1	4.15	1
3.76	<1	3.76	<1	—	—
—	—	3.56	<1	3.59	1
—	—	3.34	1	3.29	1
—	—	3.25	<1	—	—
—	—	—	—	3.06	1
2.89	10	2.92	10	2.92	10
—	—	2.82	1	2.781	3
—	—	—	—	2.564	<1
2.505	4	2.515	7	2.501	4
—	—	—	—	2.417	<1
—	—	2.284	<1	2.286	2
—	—	2.073	<1	2.089	<1
—	—	1.912	1	—	—
—	—	—	—	1.884	<1
—	—	1.808	3	1.809	7
1.777	8	1.782	5	1.770	<1
—	—	1.744	1	1.742	3
—	—	1.720	<1	1.721	1
—	—	1.627	<1	1.630	<1
—	—	—	—	1.604	<1
—	—	1.538	<1	1.538	2
1.516	7	1.520	6	1.518	3
—	—	—	—	1.479	2#
—	—	—	—	1.466	1#
1.453	1	—	—	—	—
—	—	—	—	1.422	<1#
—	—	1.368	<1	—	—
—	—	—	—	1.330	<1
1.255	1	1.255	2	1.255	2
—	—	1.176	1	1.174	2
1.154	2#	1.141	<1	1.141	1
1.125	2#	1.129	1	1.118	1
—	—	—	—	1.109	1
—	—	1.046	<1	1.046	1
1.028	1#	1.032	2#	1.031	1#
—	—	.979	1#	.982	1
.969	1#	.970	1#	.970	1
—	—	—	—	.906	1
—	—	.864	2#	.865	1#
.851	<1#	.852	2#	.852	<1#
.838	2#	.839	3#	.840	<1#

Diffuse lines.

unheated material and material heated at 620° C., 900° C., 1000° C., and 1200° C.; and furthermore it remains very weak throughout. After being heated to this intermediate temperature of 900° C. particle size is still quite minute as evidenced by broadness or diffuseness of most lines.

The temperature of 1200° C. was chosen because work in progress in connection with a wide variety of multiple oxides of tantalum, niobium, and titanium has shown that this temperature is a convenient one in most instances if reproducible *x*-ray diffraction patterns are to be obtained. However, subsequent experiments with kobeite and the Paringa River mineral have shown that heating to 1000° C. for 40 minutes will give identical and reproducible results.

NOMENCLATURE

The *x*-ray diffraction study of the Paringa River material before and after heat-treatment clearly suggests that this mineral is identical, or at least very closely related to, kobeite from Japan described by Takubo *et al.* (1950), but apparently distinct from a number of other multiple oxides of tantalum, niobium, and titanium. The actual appearance of the two minerals in their host rocks is strikingly similar, although the Paringa River mineral exhibits no pronounced alteration to an anatase-rich product; this may be due to a difference in age. Finally the chemical data, although less complete than the physical, do not detract from this similarity. Therefore it is believed that the Paringa River mineral is kobeite, and it is hoped that it will be found *in situ* and more and better material obtained that will allow a more thorough study than that reported here.

Study of the Japanese report at first leads one to the conclusion that kobeite might be a high-titanium member of the euxenite-polycrase or eschynite-priorite groups, but eight specimens that undoubtedly belong to one or other of these groups yielded *x*-ray patterns after being heated to 1200° C. that exhibited little if any resemblance to patterns obtained with kobeite similarly treated; the same observation may also be made for fergusonite, including risörite, brannerite, samarskite, and yttrio-tantalite. These data rather definitely support the suggestion that kobeite is a distinct and valid mineral species, and it is hoped that the results of work in progress in connection with a number of multiple oxides of tantalum, niobium, and titanium, may provide further data on this question.

SOURCE

Granitic intrusions and pegmatites are known to outcrop at many localities in the general area from which the kobeite-bearing rock was obtained, but so far as the writer is aware no tantalum-niobium bearing

minerals have been found in these rocks. However, it might be pointed out that tantalian cassiterite is not a rarity in concentrates from sands and gravels in many localities in Westland (Hutton, 1950). Again radioactive minerals such as uranoan thorite, huttonite, monazite, xenotime, and others (Hutton, 1950) have been found in South Westland sands and gravels, although the actual source for these minerals has not been determined as yet. The problem of the source of kobeite is complicated further by the existence of large quantities of glacial debris, much of which has been resorted by rivers and streams.

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