NEW DATA ON FERRITUNGSTITE FROM NEVADA*

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Abstract

A yellow ocherous mineral from cavities in a limonitic gossan from the Nevada Scheelite mine, Mineral County, Nevada, is shown by *x*-ray diffraction studies to be identical with ferritungstite from the type locality, the Germania tungsten mine, Deertrail mining district, Stevens County, Washington. New data obtained on the ferritungstite from Nevada show that it is tetragonal, commonly dipyramidal; indices of refraction between 2.09–2.15; specific gravity 5.2; three strongest diffraction lines are 5.94, 2.966, and 3.10 Å; probable unit cell dimensions: $a=10.28$ Å, $c=7.28$ Å, and $c/a=0.708$. The new formula is $Ca_2Fe_2^{++}Fe_2^{+++}(WO_4)_7 \cdot 9H_2O$. In the original material from the Germania mine, jarosite was intimately mixed with the ferritungstite, both minerals being very fine grained.

Introduction

In a recent study of tungsten-bearing gossan collected by D. M. Lemmon and Earl Pampeyan from the Nevada Scheelite mine, near Rawhide, Mineral County, Nevada, a yellow ocherous mineral was observed which at first could not be identified by *x*-ray diffraction data or other means. Spectrochemical analysis showed the presence of Ca, Fe, and W, the latter being the most abundant, and it was suggested by W. T. Schaller that the mineral might be ferritungstite, a mineral described by him many years ago (Schaller, 1911).

Kerr (1946) later reexamined the ferritungstite from the same cavity in the type specimen (U. S. National Museum 86985) from which Schaller obtained his analyzed material. In his report on tungsten mineralization, with reference to his *x*-ray studies of the type material Kerr states on p. 78: "*x*-ray data are inconclusive. Five different *x*-ray diffraction patterns have been taken, all of which show minor variations. One pattern agrees closely with jarosite from several localities and also with synthetic jarosite prepared in the laboratory. Others fail to agree exactly either with normal jarosite or natrojarosite, either natural or artificial. All patterns, on the other hand, bear a resemblance to examples of the jarosite group."

Comparisons and measurements of the *x*-ray powder patterns of our mineral from Nevada and of those taken by Kerr on the Germania material showed complete identity of the pattern of the mineral from Nevada with one of the five films (Graf no. 4) taken by Kerr, which film therefore must have been taken of essentially pure ferritungstite. The

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other four films must have been taken of mixtures of ferritungstite and jarosite.

The hexagonal plates, described by Shaller (1911) and thought by him at that time to be ferritungstite, are now known to have been jarosite, which explains in part Kerr's statement that "All patterns, on the other hand, bear a resemblance to examples of the jarosite group." Most of the samples *x*-rayed by him probably were mixtures of ferritungstite and jarosite. The relatively large hexagonal crystals shown in his Plate 23, facing page 77 (Kerr, 1946), are probably jarosite and not ferritungstite.

That the hexagonal plates of Schaller were jarosite is shown by the following comparison of their determined optical properties.

"Ferritungstite," Larsen (1921), p. 74: $\omega=1.80$, $\epsilon=1.72$. Probably uniaxial
"Ferritungstite," Kerr (1946), p. 77: $\omega=1.815$, $\epsilon=1.716$
Jarosite, Larsen and Berman (1934), p. 90: $\omega=1.820$, $\epsilon=1.715$. Uniaxial
Ferritungstite, this paper. Incides between 2.09–2.15.

Further evidence that much of the original Germania material was a mixture of ferritungstite and jarosite is shown by the results of the partial analysis made by Ledoux and Company on material furnished them by Kerr and given by him (Kerr, 1946, p. 78), namely, $WO_3=20.0$ per cent and $SO_3=17.4$ per cent. These values can be interpreted as representing a mixture of 28 per cent of ferritungstite (new formula) and 55 per cent of jarosite, and if to these figures be added the average value of Schaller's "insoluble," namely 15 per cent, the summation comes to 98 per cent. The sample analyzed by Ledoux and Company then was about $\frac{1}{3}$ ferritungstite and $\frac{2}{3}$ jarosite, after deducting insoluble. This sample probably represents the kind of material on which Schaller observed his hexagonal plates and explains Kerr's statement (p. 78): "One pattern agrees closely with jarosite . . . ," but probably does not represent the kind of material originally analyzed.

Similar conclusions were reached by Graf (1947), who, in an attempt to clarify the problem extended the investigations by Kerr, concentrating chiefly on the synthesis of ferritungstite. His lack of success in this attempt may have been due to the incorrect formula proposed by Schaller. Graf states, after studying the *x*-ray powder pattern of type material (presumably patterns taken by Kerr), that jarosite was more abundant than Kerr had indicated and that the unidentified lines were both consistent in, and common to, all patterns. From these conclusions, Graf reasoned that the SO_3 reported by Kerr (Ledoux's analysis) was due to admixed jarosite, and not, as Kerr intimated, a chemical constituent of ferritungstite. Graf also mentions the possibility that the indices of refraction reported for ferritungstite by Larsen (1921) were actually determined on the associated jarosite.

It is now apparent that the original material on which Schaller based his description of ferritungstite and now nearly all consumed (as only a thin dusty film remains in the quartz cavity) was a mixture of two minerals, both very fine grained, the better crystallized being jarosite and the other being ferritungstite, which apparently was deposited, at least in part, on previously formed jarosite.

Only two other published reports of occurrences of ferritungstite are known and one of these, from the quartz-wolframite deposits at Silvermine, Missouri (Singewald, 1929) has been shown by Graf to be jarosite. The other occurrence has been reported by Kittl (1939) from Cerro Liquinaste, Argentina. Kittl describes "ferritungstite" as a brown mineral with a greasy luster (chestnut brown streak) pseudomorphous after wolframite. The specific gravity was determined as 5.57. These properties are not in accord with those of ferritungstite from either Washington or Nevada.

Occurrence

The Nevada Scheelite mine is in Mineral County, Nevada, approximately 4 miles east of the nearly deserted town of Rawhide. Scheelite was discovered there in 1930, and development of the principal deposit was undertaken in 1936 by Nevada Scheelite, Inc. The mine has been a substantial producer since then, and is now operated as a subsidiary of Kennametal, Inc.

The ore bodies are scheelite-bearing tactite along the contact between granite and limestone of Triassic age (Geehan and Trengove, 1950). The primary ore consists dominantly of andradite garnet accompanied by variable quantities of calcite, quartz, amphibole, epidote, pyroxene, wollastonite, pyrite, chalcopyrite, magnetite, and scheelite. Above the 200-foot level the ore bodies are largely oxidized, and remnants of sulfides are found only in isolated spots. The content of tungsten in the oxidized ore is somewhat greater than in the sulfide ore, due principally to the leaching of gangue minerals during oxidation. The metallurgical recovery from oxidized ore has been unexpectedly difficult and the mill tailings contain nearly a third of the tungsten originally present. It is believed that the tungsten in these tailings occurs largely as very fine grained ferritungstite mixed with limonite.

On the fringes of the oxidized ore bodies, particularly in the footwall against limestone, there are masses of quartzose limonitic gossan that contain very little scheelite but assay several per cent of tungsten trioxide in the form of ferritungstite. The material investigated was collected from two stopes a few feet above the 200-foot level. The gossan contains other secondary minerals in addition to ferritungstite, including bismutite and jarosite, all apparently deposited later than the limonite and possibly contemporaneous with the ferritungstite. No tungstite or hydrotungstite was observed.

The two forms of ferritungstite (crystals and fibers) observed under the microscope seem to be habit modifications and not separate phases. The indices of refraction fall within the same range for the two types, and *x*-ray powder diffraction patterns do not show inconsistent or variable interplanar spacings that might be attributable to two phases.

PROPERTIES

Structural

Probable unit-cell dimensions and contents, as determined from *x*-ray powder patterns, are: $a = 10.28$ Å, $c = 7.28$ Å, $c/a = .708$, cell volume $= 769.3$ Å^3, S.G. (calc.) $= 4.75$ (with $Z = 1$). The *x*-ray powder diffraction data indexed for all interplanar spacings to 1.15 Å are shown in Table 1.

TABLE 1. OBSERVED AND CALCULATED INTERPLANAR SPACINGS FOR FERRITUNGSTITE, INDEXED ON A TETRAGONAL CELL

	CuKα radiation, λ=1.5418 Å* Dia. of camera=114.59 mm. *d*(Å)		
I	*Measured*	*Calculated*	*hkl*
10	5.94	5.92	101
9	3.10	3.10	301
10	2.966	2.968	311
		2.970	202
6	2.572	2.572	222
		2.570	400
3	2.361	2.362	103
		2.359	411
½	2.294	2.298	420
		2.299	331
		2.302	113
1	2.101	2.099	402
5	1.979	1.979	501, 431
		1.980	303
8	1.819	1.820	004
		1.817	440
5	1.738	1.739	413
½	1.625	1.626	442
3	1.569	1.569	433
		1.568	541
7	1.550	1.550	602
		1.551	513
3	1.485	1.485	404
		1.484	622

* CuKα_1 (λ=1.54050 Å) radiation for interplanar spacings below 0.8739 Å.

TABLE 1—(*continued*)

I	*d*(Å)		*hkl*
	Measured	*Calculated*	
4	1.441	1.440	701
		1.442	105
4	1.341	1.340	305
		1.339	543
2	1.286	1.286	444
		1.285	800
½	1.257	1.257	415
		1.256	741, 811
1	1.188	1.188	435, 505
5	1.180	1.180	515
		1.180	733
		1.179	751
		1.179	822
4	1.150	1.149	662
		1.151	226
2	1.129		
2	1.079		
3	1.051		
2	1.035		
½	0.9948		
6	.9902		
1	.9602		
1	.9285		
1	.9105		
3	.8989		
1	.8739		
6	.8700		
4	.8575		
2	.8492		
2	.8270		
4	.8139		
2	.7871		
6	.7850		
6	.7758		

There are still certain features of ferritungstite that, in light of our present knowledge of the mineral, are difficult to explain. The odd number of large $WO_4^=$ groups in a tetragonal mineral and the discrepancy between measured and calculated specific gravity necessitate definition by single-crystal and other studies when suitable material is found. Still another problem is the presence of ferrous iron in a mineral supposedly deposited in an oxidizing environment.

Physical and Optical

Ferritungstite occurs as a bright-yellow ocherous powder lining and filling cavities in the limonitic gossan. Ferritungstite is tetragonal (Fig. 1); crystals dipyramidal, some exhibiting prismatic faces, and also commonly in small fibers. The crystals and fibers are translucent, have

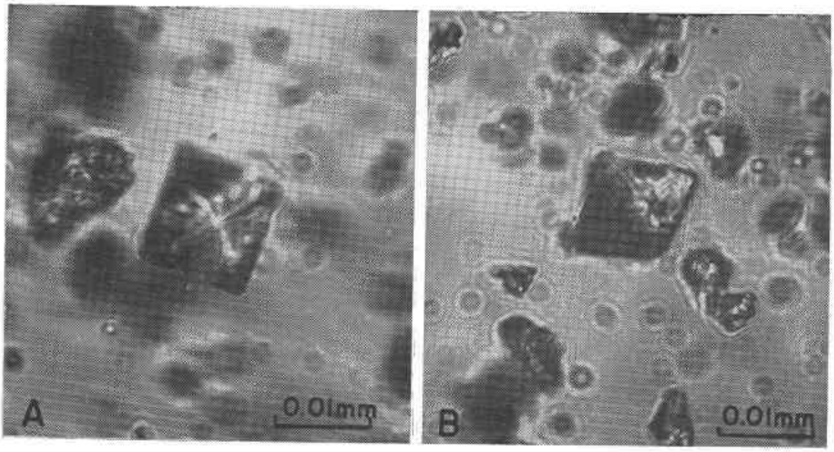

FIG. 1. Ferritungstite crystals. (*A*) Normal to *c*-axis showing four tetragonal pyramid faces. (*B*) Crystal resting on pyramid face showing dipyramidal character.

exceedingly high relief and are very small; the largest single fragment observed was approximately 0.025 mm. in diameter. The optical character and exact indices of refraction were not measurable because of the minute size of the crystals, but n is between 2.09 and 2.15. The specific gravity of the analyzed sample, determined with a fused silica Adams-Johnston pycnometer is 4.89; after correcting for approximately 6.5 per cent quartz as an impurity, the probable specific gravity of ferritungstite is 5.2.

Chemical

A 500 mg. sample was carefully hand-picked for chemical analysis (Table 2). The derived formula is $Ca_2Fe_2^{++}Fe_2^{+++}(WO_4)_7 \cdot 9H_2O$. Ferritungstite is decomposed with difficulty in hot HCl.

Briefly, the analytical procedure used was as follows: water, tungsten, total iron, and calcium were determined on one portion, and ferrous iron and insoluble material on another. Water was determined by measuring the loss of weight when the sample was ignited, with subsequent correction for oxidation of divalent iron. The sample was then fused with sodium carbonate, leached with water, and filtered to separate the tungsten from the iron and calcium. Tungsten was determined polarographically in a supporting electrolyte of 4.5 molar hydrochloric acid and 0.1 molar tartrate. The total iron and calcium were determined according to the usual gravimetric procedures for silicate rocks. The sample for the divalent iron determination was boiled gently for 3 hours in 10 ml. of

TABLE 2. CHEMICAL COMPOSITION OF FERRITUNGSTITE FROM NEVADA

	Chemical analysis	Recalculated to 100%	Moles	Mole ratios	Theoretical chemical composition
WO_3	67.70	72.61	0.3131	6.8	73.75
Fe_2O_3	6.84	7.33	0.0459	1.0	7.26
FeO	6.07	6.51	0.0906	2.0	6.52
CaO	5.62	6.03	0.1075	2.3	5.10
H_2O	7.01	7.52	0.4178	9.0	7.37
SiO_2 (quartz)	6.56				
Total	99.80	100.00			100.00

Formula: $Ca_2Fe_2^{++}Fe_2^{+++}(WO_4)_7\cdot 9H_2O$.

concentrated hydrochloric acid under an atmosphere of carbon dioxide. After cooling, the divalent iron was titrated with permanganate in 5 per cent sulfuric acid containing about 10 per cent Zimmerman-Reinhardt solution. The solution was then filtered through a sintered glass crucible, the tungstic acid dissolved with ammonium hydroxide, and the residue weighed as insoluble material.

Trace amounts of lead, strontium, magnesium, sodium, potassium, copper, manganese, titanium, and uranium are also in the sample.

The chemical formula given in this note is at considerable variance with that originally presented by Schaller. The research reported here shows that Schaller analyzed a mixture of ferritungstite and jarosite.

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