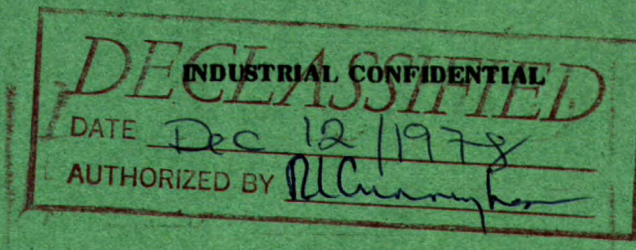


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CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 63-18

**INVESTIGATION OF A SAMPLE OF
MOLYBDENITE ORE FROM OLDEN TOWNSHIP,
FRONTENAC COUNTY, ONTARIO,
SUBMITTED BY K. E. VARRETTE**

by

T. F. BERRY

MINERAL PROCESSING DIVISION

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JANUARY 30, 1963

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SUMMARY OF RESULTS

Flotation tests in which a rougher molybdenite concentrate was reground and given repeated stages of cleaning resulted in a final molybdenite concentrate assaying 67.6% MoS₂ with a recovery of 82.6% of the molybdenum in the ore.

Attempts to produce a concentrate of marketable grade of over 90% MoS₂ were unsuccessful due to the very fine intergrowth of the molybdenite and the serpentine mineral grains. Even ultra-fine regrinding of the above molybdenite concentrate in a Trost jet mill failed to completely liberate the molybdenite-serpentine intergrowth and resulted in a final concentrate, after re-floating, of only 81.3% MoS₂.

*Technical Officer, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

INTRODUCTION

As the result of a meeting and of subsequent correspondence between officers of the Mineral Processing Division, Mines Branch, Ottawa, and Mr. K. E. Varrette, 131 Brock Street, Kingston, Ontario, an investigation was started on August 29, 1962, on a bulk sample of molybdenite ore.

Location of Property

The sample was taken from a deposit located in Lot 2 Concession 7, in Olden Township, Frontenac County, Ontario. Access to the claim, No. E.O. 28376, is by good gravel road, a distance of about 4 miles northwest of the village of Parham, Ontario.

Nature of Investigation Requested

Mr. Varrette asked the Mines Branch to determine if the molybdenite in the sample could be concentrated to a marketable grade by flotation.

Shipment

On August 16, 1962 a bulk sample of the ore weighing about 500 lb was received at the Mines Branch.

Sampling and Analysis

Several representative pieces of the ore were selected for a mineralogical examination. The entire sample was then crushed to $-\frac{1}{2}$ in. with a further reduction of half of this crushed ore to -10 m. A head sample was riffled out of the -10 m material for a semi-quantitative spectrographic and a chemical analysis. In Tables 1 and 2 the results of this analytical work may be seen.

TABLE 1

Semi-Quantitative Spectrographic Analysis*

Range - Per Cent	Elements
Principal Constituent	Mg, Si, Ca,
1.0 to 0.1	Mo, P, Mn, Fe, Pb,
0.1 to 0.01	Al, Sn, In, Sr, Ni,
0.01 to 0.001	Cu, Ti, Ba,
0.001	Be,

*From Internal Report MS-AC-62-1045

TABLE 2

Chemical Analysis* of Head Sample

Element	Per Cent
Molybdenite (MoS ₂)	0.80
Copper (Cu)	0.004
Iron (soluble Fe)	0.52
Sulphur (total S)	0.40
Lead (Pb)	0.07
Bismuth (Bi)	0.08
Magnesium (MgO)	12.98
Lime (CaO)	18.54
Silica (SiO ₂)	25.55
Insoluble	64.10

*From Internal Report MS-AC-62-1186

MINERALOGICAL EXAMINATION

A separate Mines Branch Investigation Report* has been issued outlining the mineralogical work done. As far as the mineralogy on the ore is concerned only a summary of the results of that work will be included here. The mineralogical investigation on the mill products will be outlined in detail later in this report.

Summary of Results

It was found that the molybdenite occurred in mineralized veinlets and pods in a pyroxene-rich rock. These veinlets and pods consisted of complex grains composed of narrow bands in which either molybdenite or serpentine predominated. The bands consisted of still narrower intercolated microbands of molybdenite and serpentine.

A small amount of a highly fluorescent mineral was present on the surfaces of the hand specimens and was identified by X-ray diffraction as powellite (Ca(Mo,W)O₄).

The wall rock consisted of diopside-augite pyroxene and calcite. In some cases the pyroxene was altered to a light green serpentine or antigorite.

The occurrence of the molybdenite showing its relationship with serpentine is illustrated in Figures 1 and 2.

*Mines Branch Investigation Report IR 62-88 by W. Petruk, November 8, 1962.

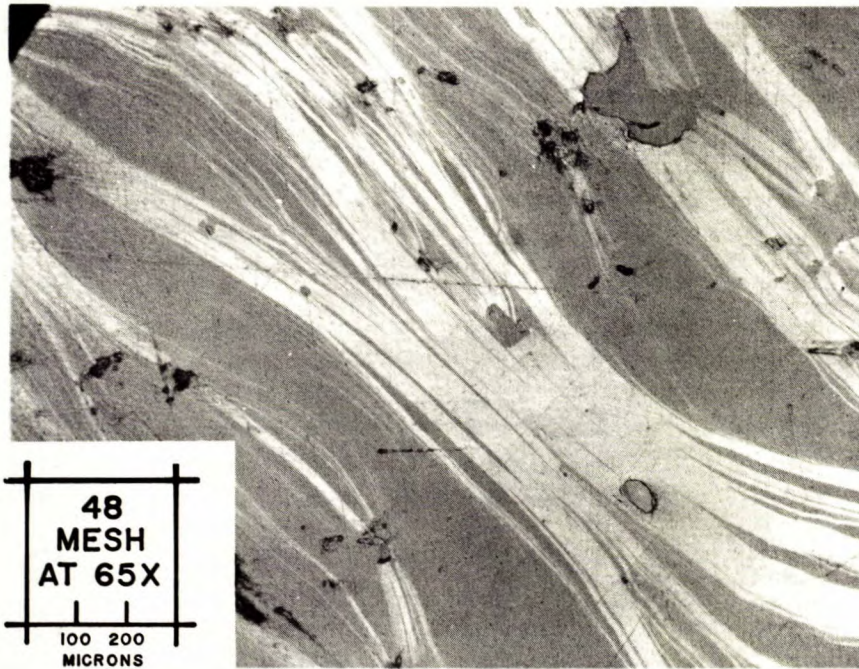


Figure 1 - Photomicrograph of a polished section showing the textural relationship of molybdenite (white) and serpentine (grey).

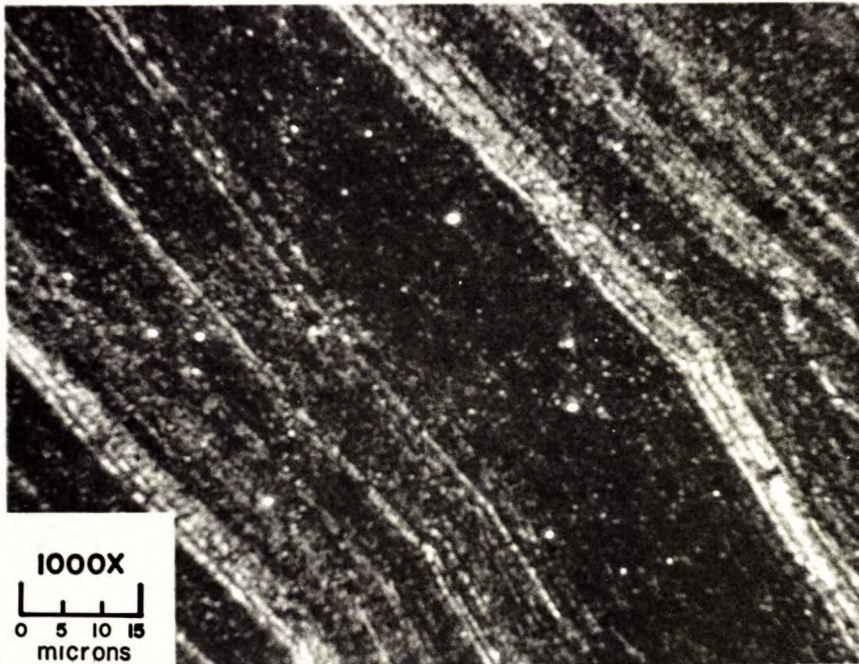


Figure 2 - Photomicrograph of a polished section of a serpentine band showing in detail the numerous "micro-bands" and the extremely fine texture of the molybdenite particles (white).

DETAILS OF INVESTIGATION

A series of three preliminary tests was done to determine the optimum liberation point of the molybdenite in the ore and the amenability of the molybdenite to concentration by flotation using kerosene and pine oil. In each test flotation proceeded until no more molybdenite was visible in the froth. The results of these tests are shown in the following tables.

TABLE 3

Results of Test 1

Product	Weight %	Analysis %* MoS ₂	Distribution % MoS ₂	Remarks
MoS ₂ conc	2.6	30.32	87.1	Grind - 25.8% -200 m
Flotation tail	97.4	0.12	12.9	Kerosene - 0.81 lb/ton
Head (calc'd)	100.0	0.90	100.0	Pine oil - 0.06 " "

TABLE 4

Results of Test 2

Product	Weight %	Analysis %* MoS ₂	Distribution % MoS ₂	Remarks
MoS ₂ conc	3.2	23.94	89.8	Grind - 37.2% -200 m
Flotation tail	96.8	0.09	10.2	Kerosene - 0.63 lb/ton
Head (calc'd)	100.0	0.85	100.0	Pine oil - 0.06 " "

TABLE 5

Results of Test 3

Product	Weight %	Analysis %* MoS ₂	Distribution % MoS ₂	Remarks
MoS ₂ conc	5.3	14.90	90.3	Grind - 70.4% -200 m
Flotation tail	94.7	0.09	9.7	Kerosene - 0.99 lb/ton
Head (calc'd)	100.0	0.87	100.0	Pine oil - 0.08 " "

*From Internal Report MS-AC-62-1143.

Test 4

A 200 g sample of -10 m ore was ground to about 57% -200 m and a molybdenite rougher flotation concentrate was floated for 7 min using staged additions of kerosene (0.63 lb/ton), pine oil (0.03 lb/ton) and Dowfroth 250 (0.045 lb/ton). This concentrate was cleaned three times using sodium cyanide (0.1 lb/ton), sodium silicate (1.0 lb/ton) and small amounts of kerosene, pine oil and Dowfroth 250. The results of this test are shown in the following table.

TABLE 6

Results of Test 4

Product	Weight %	Analyses %* MoS ₂	Distribution % MoS ₂
Final MoS ₂ conc	1.2	55.20	82.7
3rd cl tail	0.2	8.87	2.2
2nd " "	0.8	1.16	1.2
1st " "	2.3	0.27	0.8
Flotation tail	95.5	0.11	13.1
Head (calc'd)	100.0	0.80	100.0

*From Internal Report MS-AC-62-1024.

The results of this test were significant in that even after three stages of cleaning the grade of the final concentrate was only 55.20% MoS₂. Normally one would expect to obtain a concentrate grade of 90% MoS₂ or better under these conditions. Undoubtedly the very fine inclusions of serpentine in the molybdenite grains, shown by the preliminary mineralogical examination, are the reason for the low concentrate grade.

Test 5

To liberate if possible and to remove the serpentine, the conditions which prevailed in this test were considerably more rigorous than those applied in Test 4.

A 10,000 g sample of -10 m ore was ground in lots of 2000 g each to about 37% -200 m. A rougher MoS₂ concentrate was floated in 9 min from each 2000 g of pulp using staged additions of kerosene (0.81 lb/ton), pine oil (0.1 lb/ton) and Dowfroth 250 (0.03 lb/ton). The five rougher concentrates were combined for subsequent treatment.

A scavenger concentrate was floated for 2 min to recover any residual molybdenite using potassium sec-amyl xanthate (0.05 lb/ton) and Dowfroth 250 (0.015 lb/ton). This concentrate was analysed for MoS₂ and MgO.

The combined rougher concentrate was cleaned and recleaned using sodium silicate (1.0 lb/ton) and Guartec (0.01 lb/ton) as serpentine depressants, and small amounts of kerosene and pine oil.

A small portion of this primary recleaner concentrate was retained for a mineralogical examination and the remainder was ground in an Abbe mill using steel balls for 20 min. This ground concentrate was cleaned once using sodium silicate, Guartec and small amounts of kerosene and pine oil. A small portion of this first regrind cleaner concentrate was retained for a mineralogical examination and the remainder was ground in the Abbe mill for 20 min. Two cleaning stages now followed using sodium silicate, Guartec, kerosene and pine oil. The results of this test are shown in Table 7.

TABLE 7

Results of Test 5

Product	Weight %	Analyses %*				Distribution %	
		MoS ₂	MgO	SiO ₂	Fe (total)	MoS ₂	MgO
2nd regrind recl conc	0.90	67.56	12.40	12.40	0.26	82.6	0.9
" " " tail	0.04	23.58	26.91	-	-	1.3	0.1
" " cl "	0.11	10.72	23.77	-	-	1.6	0.2
1st " " "	0.33	3.41	21.35	-	-	1.5	0.6
Primary recl tail	0.65	2.99	20.16	-	-	2.6	1.0
" cl "	2.61	0.55	18.69	-	-	2.0	3.8
Scavenger conc	1.09	0.49	18.55	-	-	0.7	1.6
Flotation tail	94.27	0.06	12.39	-	-	7.7	91.8
Head (calc'd)	100.00	0.74	12.72	-	-	100.0	100.0

*From Internal Report MS-AC-62-1184.

The results of a screen analysis of the second regrind recleaner concentrate are shown in Table 8.

TABLE 8

Screen Analysis on Second Regrind Recleaner Concentrate

Sized Fractions	Weight %	Analysis %*		Distribution %	
		MoS ₂	MgO	MoS ₂	MgO
+ 65 mesh	9.2	77.88	9.77	10.1	7.4
- 65 +100 "	14.6	71.93	12.03	14.8	14.5
-100 +150 "	16.9	69.80	13.04	16.6	18.2
-150 +200 "	16.6	70.22	12.77	16.4	17.5
-200 +325 "	16.9	69.37	10.49	16.5	14.7
-325 "	25.8	70.22	12.94	25.6	27.7
Total	100.0	70.96	12.09	100.0	100.0

*From Internal Report MS-AC-62-1254.

Mineralogical Examination* of Mill Products from Test 5

The mill products from Test 5 were subjected to a thorough mineralogical examination to determine the mode of occurrence of the serpentine in the molybdenite grains. It was necessary to prepare the polished sections of the mill products in such a manner that the molybdenite flakes could be viewed in cross-section. This was achieved by mixing the samples with a liquid cold setting medium in a small glass vial and allowing the molybdenite flakes to preferentially orient themselves by settling in the liquid. The solidified mixture was sawed perpendicular to the direction of the molybdenite flakes. Polished sections of these faces were prepared and examined under a reflecting microscope.

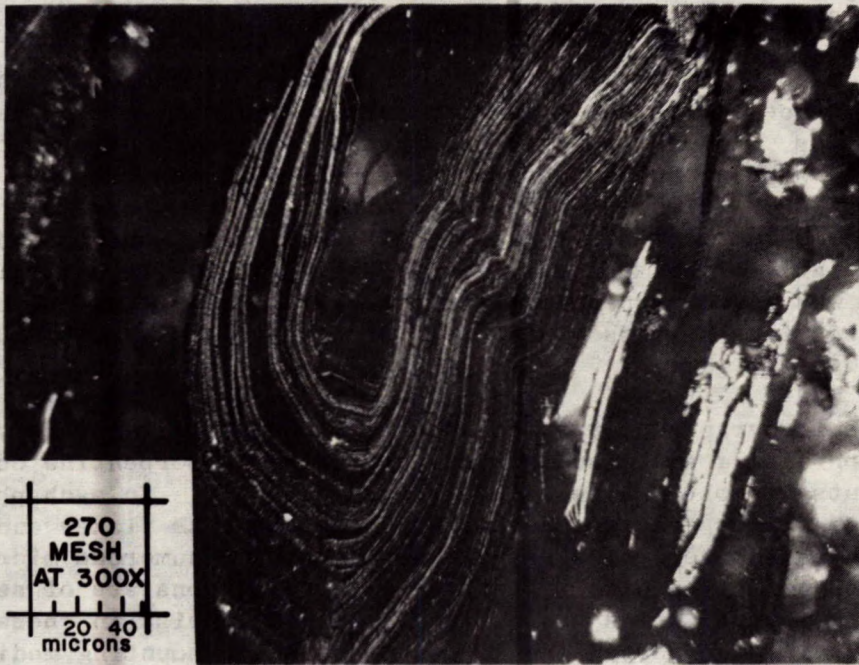
The photomicrographs of the polished sections show grains varying from molybdenite containing small amounts of serpentine to serpentine containing small amounts of molybdenite (see Figures 3 to 11). In each photomicrograph the white grains are cross-sections of molybdenite flakes and the finely banded black and white grains are flakes containing numerous microbands of serpentine and molybdenite. The dark background consists of serpentine and mounting medium which are difficult to distinguish in these photographs, but usually the serpentine is darker than the mounting medium.

The second regrind recleaner concentrate from Test 5 analysing 67.56% MoS₂ was treated on a laboratory scale by heavy liquid separation using tetra-bromoethane (specific gravity 2.96).

This experiment was done to determine whether grains of free serpentine and those of molybdenite containing serpentine could be separated from relatively pure grains of molybdenite and thus determine if it might be possible to obtain a high grade concentrate at a sacrifice in the recovery.

The sink fraction was examined microscopically and found to contain flakes consisting of combined molybdenite and serpentine which were similar in character to those observed in the initial concentrate. No improvement in grade was observed.

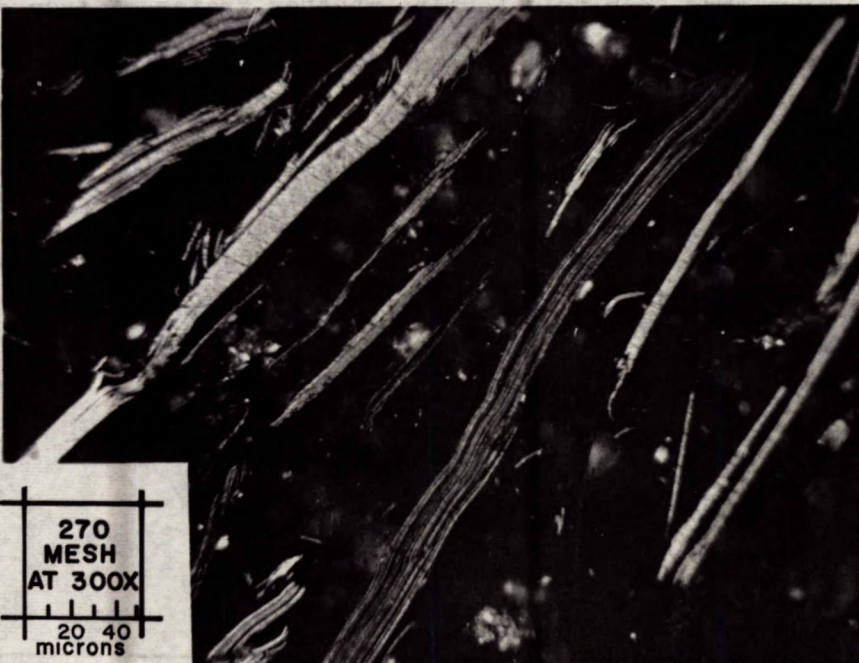
* Mines Branch Investigation Report IR 62-88, by W. Petruk, November 8, 1962.



MoS_2 - 47.13%

MgO - 15.86%

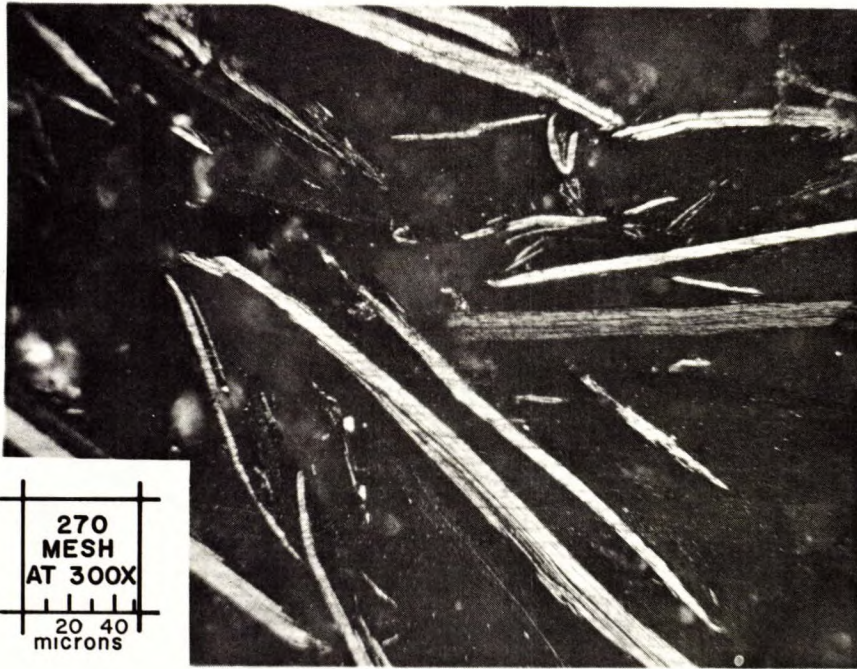
Figure 3 - Primary recleaner concentrate.



MoS_2 - 60.88%

MgO - 14.14%

Figure 4 - First regrind cleaner concentrate.



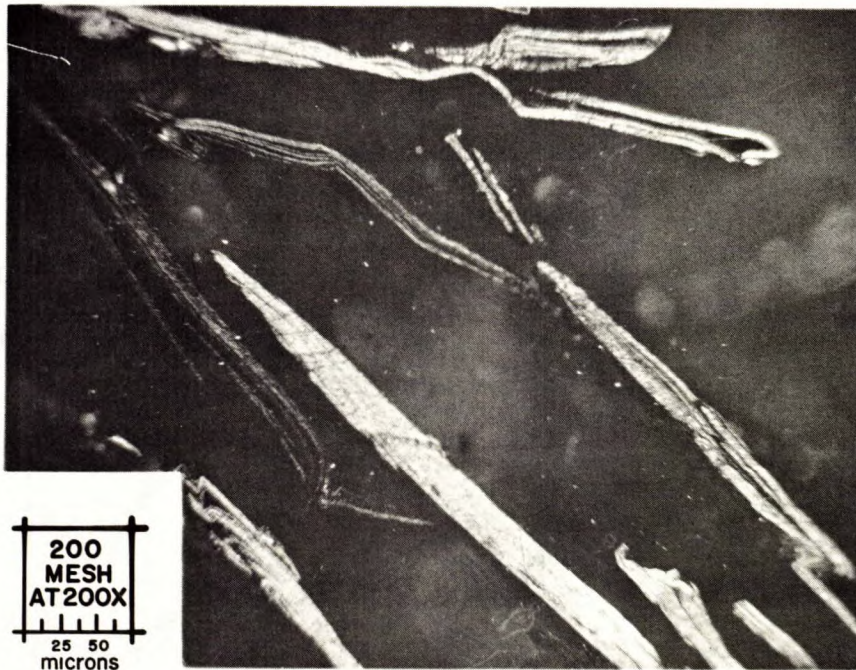
MoS₂ - 67.56%

MgO - 12.40%

SiO₂ - 12.40%

Fe - 0.26%

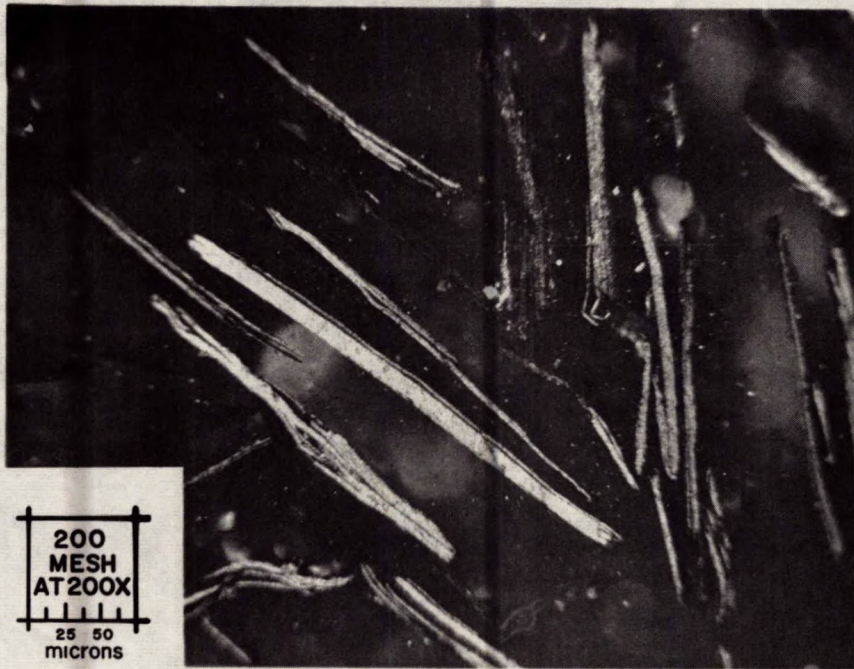
Figure 5 - Second regrind recleaner concentrate.



MoS₂ - 77.88%

MgO - 9.44%

Figure 6 - +65 mesh molybdenite concentrate.



MoS₂ - 71.93%

MgO - 12.03%

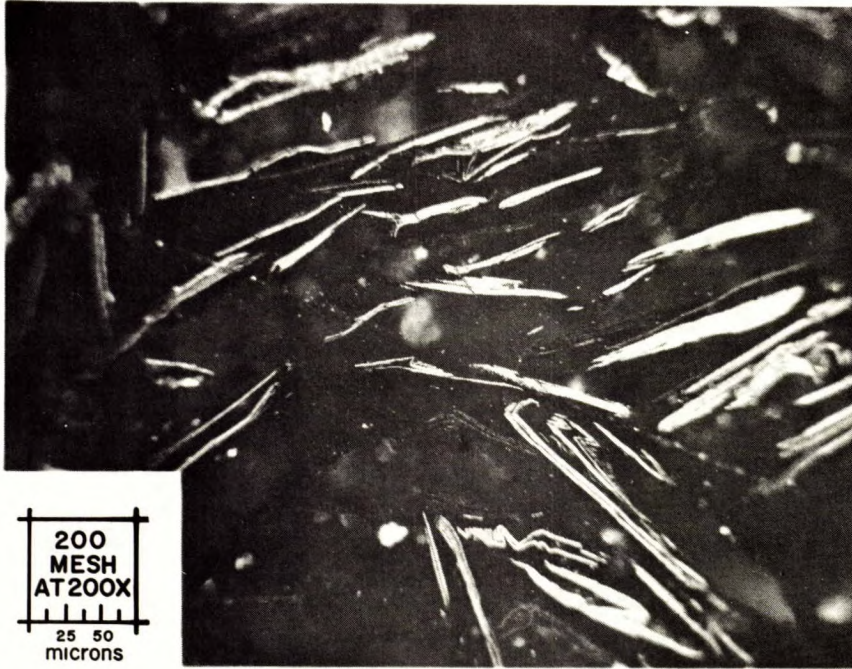
Figure 7 - -65 +100 mesh molybdenite concentrate.



MoS₂ - 69.80%

MgO - 13.04%

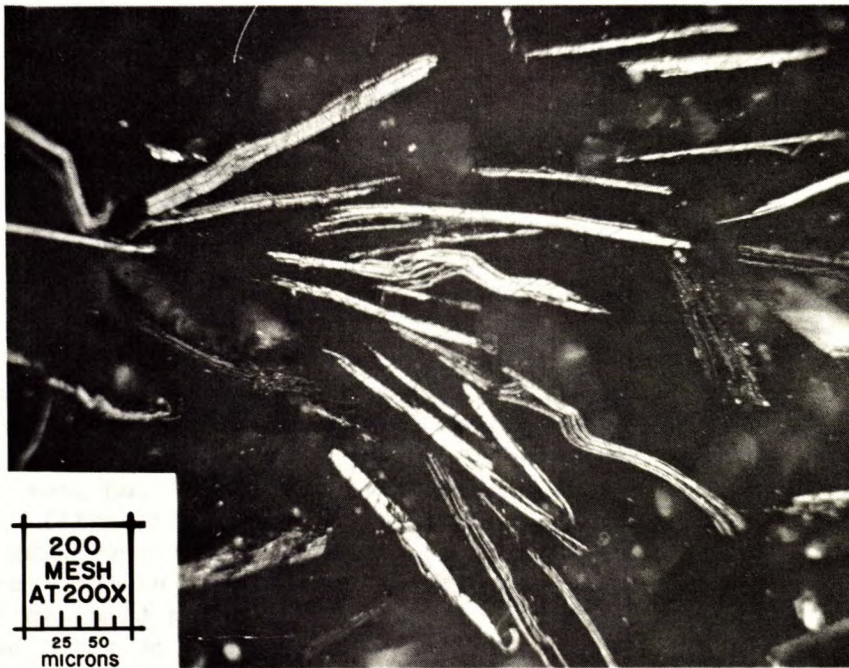
Figure 8 - -100 +150 mesh molybdenite concentrate.



MoS₂ - 70.22%

MgO - 12.77%

Figure 9 - -150 +200 mesh molybdenite concentrate.



MoS₂ - 69.37%

MgO - 10.49%

Figure 10 - -200 +325 mesh molybdenite concentrate.



MoS₂ - 70.22%

MgO - 12.94%

Figure 11 - -325 mesh molybdenite concentrate.

Test 6

Three 2000 g samples of -10 mesh ore were ground to about 37% -200 m. Using the flotation procedure outlined in Test 5 rougher concentrates were floated from each sample and were combined. The combined rougher concentrate was cleaned three times using staged additions of sodium silicate (0.1 lb/ton) Guartec (0.03 lb/ton) and small amounts of kerosene and pine oil. This third cleaner concentrate was ground for 1 hr in an Abbe mill using steel balls and the ground concentrate was cleaned three times using small quantities of the reagents mentioned. The flotation time of the final cleaning stage was reduced to about 1/3 of the previous cleaning times in an attempt to float only serpentine-free molybdenite. The results of this test are shown in Table 9.

TABLE 9

Results of Test 6

Product	Weight %	Analyses*		Distribution % MoS ₂
		MoS ₂	MgO	
Final conc	0.7	74.48	9.83	67.7
Regrind cl tail**	0.6	24.03	-	18.8
Primary cl tail**	3.5	0.80	-	3.6
Flotation tail	95.2	0.08	-	9.9
Head (calc'd)	100.0	0.77	-	100.0

*From Internal Report MS-AC-62-1252.

**All tailings from regrind cleaner stages were combined as were all primary cleaner tailings.

Test 7

The microscopic examination of the mill products from Test 5, showing the extremely fine intergrowth of the molybdenite and serpentine made it impractical to use conventional grinding equipment to achieve the degree of liberation necessary to obtain a high-grade flotation concentrate.

In an attempt to achieve greater liberation, the rejects from the second regrind recleaner concentrate from Test 5 were ground in a Trost jet mill which utilizes a high speed air jet to reduce the particles to micron sizes. Although no particle size determination of the micronized concentrate was made, a microscopic observation indicated that the material was largely minus 10 microns.

The micronized concentrate was floated using small amounts of sodium silicate and Guartec as the serpentine depressants and small amounts of kerosene and pine oil. No problems were encountered in the flotation of this ultra-fine material. The results of this flotation test may be seen in Table 10.

TABLE 10

Results of Test 7

Product	Weight %	Analyses*		Distribution % MoS ₂
		MoS ₂	MgO	
Final conc	63.4	81.34	8.02	70.2**
Cleaner tail	24.1	68.11	13.11	22.4
Flotation tail	12.5	43.37	21.76	7.4
Head (calc'd)	100.0	73.40	10.96	100.0

*From Internal Report MS-AC-63-161.

**This recovery represents about 58% of the MoS₂ in the ore.

CONCLUSIONS

All attempts to produce a high-grade molybdenite concentrate were unsuccessful. The extremely fine intergrowth of the molybdenite and the serpentine grains, which was positively identified by the thorough mineralogical examination of the ore and mill products accounts for the low grade of the concentrates. In the results of Test 7, shown in Table 10, it may be seen that when a relatively high grade molybdenite concentrate (73.40% MoS₂) was "micronized" in a Trost jet mill, the resulting final flotation concentrate analysed considerably below the 90% plus MoS₂ analysis considered by the industry to be a minimum saleable concentrate.

The investigation shows that a 90% grade of molybdenite concentrate cannot be made with this ore using standard mineral dressing methods. However, the impurity in the molybdenite concentrate appears to be solely magnesium silicate, so there might be a possibility of utilizing the molybdenite concentrate for making ferromolybdenum.

ACKNOWLEDGEMENT

The writer wishes to acknowledge the work of the staff of the Analytical Chemistry Sub-division of the Mineral Science Division for the chemical analyses done in this investigation.