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UPGRADING ALABAMA TANTALUM CONCENTRATES

A. E. Petersen

D. V. Behunin

US Bureau of Mines
Salt Lake City, Utah

W. O. O'Dell, Jr.

O'Dell Construction Company, Incorporated
Prattville, Alabama

LINDA HALL LIBRARY
5109 CHERRY STREET
KANSAS CITY, MO. 64110
U. S. A.

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Abstract. The United States Bureau of Mines conducted research on high-intensity magnetic separation methods for upgrading tantalum concentrates from Alabama. Process flow sheets were studied to maximize tin removal and minimize tantalum loss. Using laboratory-scale equipment, concentrates were produced which contained over 65% combined $Ta_2O_5-Nb_2O_5$. Up to 78% of the tin was recovered as a byproduct, containing about 70% SnO_2 . Only 1 to 5% of the tantalum reported with the tin byproduct. The producer subsequently incorporated high-intensity magnetic separation into its process flow sheet to provide concentrates for the National Defense Stockpile.

Introduction

The McAllister Ta-Sn deposit, located in Coosa County, Alabama, consists of a series of complex pegmatite dikes and pipes which intrude a granite pluton formation. Wodginite $((Ta,Nb,Sn,Mn,Fe)_{16}O_{32})$ is the primary tantalum mineral. The deposit was discovered in 1982 by Callahan Mining Corporation, of Phoenix, Arizona, through stream-sediment reconnaissance and soil geochemical survey methods. Foord and Cook described the deposit and summarized much of the available geological information (1989).

The property was acquired in 1985 by O'Dell Construction Co. Inc., of Prattville, Alabama. The firm established a mine and concentrator, which began producing tantalum mineral concentrates in 1989. The mineral concentrates are recovered by gravity beneficiation methods. The firm is currently the Nation's only supplier of domestic tantalum minerals.

The United States Bureau of Mines acquired samples of the Alabama concentrates as part of a Bureau investigation into potential domestic tantalum resources. A previous report (Wouden and Chatman, 1991) described Bureau evaluations on the geology and beneficiation of tantalum-bearing pegmatites in Wyoming. The Wyoming deposits contained little tin and were unweathered.

The concentrates received from Alabama had been prepared by wet gravity separation methods, including the use of spirals and tabling. These concentrates contained considerable tin and low amounts of niobium. Analyses revealed that much of the tin was in the form of free cassiterite (SnO_2). Tests conducted by the Bureau at the Salt Lake City Research Center showed that Alabama tantalum concentrates could be upgraded by high-intensity cross-belt magnetic separation, and a tin byproduct could also be recovered.

The results of this work helped establish methods for commercially reprocessing the Alabama concentrates using high-intensity induced-roll magnetic separation equipment. Incorporating magnetic separation into the process flow sheet helped the producer meet Defense Logistics Agency specifications in providing concentrates for the National

Defense Stockpile.

Sample Description

The samples used for evaluation were supplied by O'Dell Construction Company from stocks of previously prepared concentrate. Sample evaluations included chemical analysis, x-ray diffraction (XRD), and scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS).

Sample A represented 2 mt of concentrate material. It was noted that some of the tantalum and tin minerals in the sample were bound together into aggregates by rust, which had formed when metallic iron, contained in the concentrate, was oxidized by air drying after beneficiation. Simple attrition milling proved insufficient for breaking up these aggregates. Sample B had been oven dried with no evidence of formation of mineral aggregations. Sample C, taken from 9 mt of concentrate, was also oven dried to avoid mineral aggregations. Sample C contained more cassiterite than the other samples. The material in all three samples was finer than 1.18 mm.

Chemical analyses of the samples are provided in table 1. The tantalum and niobium assays were attractively high. The tin content, however, far exceeded 6.0% SnO_2 allowed by Defense Logistics Agency specifications for National Defense Stockpile grade 1 concentrates.

Evaluation by XRD and SEM-EDS revealed that the samples consisted primarily of wodginite $((Ta,Nb,Sn,Mn,Fe)_{16}O_{32})$, manganotapiolite $((Mn,Fe)(Ta,Nb)_2O_6)$, manganocolumbite $((Fe,Mn)(Nb,Ta)_2O_6)$, and cassiterite (SnO_2), with minor amounts of zircon, pollucite (a hydrated cesium-aluminum silicate), quartz, mica, iron oxides, and iron-manganese oxides. While part of the tin was in the wodginite, evaluation indicated 60 to 80% of the tin was in cassiterite form. In general, the minerals were well liberated with only a few cassiterite grains locked with tantalum minerals.

Cassiterite and tantalum minerals appeared similar at low magnification. At higher magnification cassiterite grains exhibited brown translucence, especially when finely ground, moistened, and observed against a white background. In contrast, the tantalum minerals were shiny, opaque, and black.

Processing Evaluations

When using cross-belt high-intensity magnetic separation equipment it was found that the cassiterite reported to the non-magnetic fraction and the tantalum minerals reported to the magnetic fraction. The effects of several variables were then studied: field strength, preliminary grinding, and multiple passes through the separator.

Field Strength

The effect of field strength was evaluated by repeatedly passing a quantity of Sample A through the cross-belt separator at successively increasing field strength. The non-magnetic fraction from using lower strength fields was reprocessed at higher field strengths. Figure 1 shows the cumulative amounts of tantalum, niobium, and tin recovered in the magnetic fraction. The spacing between the magnet head and the feed belt was kept constant at about 5 mm.

Most of the rust bound aggregates were recovered with low field strength. Because more niobium than tantalum was recovered at low field strength, it appears that the manganocolumbite was slightly more magnetic than most of the tantalum bearing minerals. At high field strength nearly all the niobium and tantalum were recovered. Evaluations using SEM-EDS indicated that most of the tin that was recovered in magnetic products at low field strength was in wadginite form.

The magnetic product obtained at the highest field strength (0.72 Wb/m^2) had a relatively higher level of tin contamination than the low field strength products. This material contained considerably more cassiterite than those products obtained at lower field strength. Much of this cassiterite was locked with magnetic minerals. A plot of the Sn/Ta weight ratio for individual products at each field strength emphasizes this jump in tin contamination (figure 2). The non-magnetic cassiterite byproduct, which passed through the separator at 0.72 Wb/m^2 , had a Sn/Ta weight ratio of 44.

Multiple Pass Separation

Tests were performed to determine how many times the magnetic material should be passed through the separator to remove non-magnetic minerals. Quantities of sample A were dry ground in a rod mill to $-589 \mu\text{m}$ and a portion was passed through the cross-belt separator at 0.38 Wb/m^2 . The magnetic tantalum product was sampled by splitting, and the remaining magnetic product was reprocessed three additional times through the separator and sampled by splitting after each pass. The work was repeated using other portions of ground sample A at 0.52 and 0.75 Wb/m^2 . The SnO_2 content of each magnetic product is shown in figure 3.

These results show that two passes through the separator removed most of the cassiterite from the tantalum concentrate. Use of low field strength yielded tantalum concentrates with less tin contamination. However, high field strength was required to achieve high overall tantalum recovery.

Effect of Particle Size

A test was performed to determine the effect of particle size on separation and recovery. Quantities of sample A were sized by screening at $-1180 +589$, $-589 +300$, -300

$+147$, and $-147 \mu\text{m}$. Each sample was passed through the cross-belt separator at 0.65 Wb/m^2 . The non-magnetic cassiterite portion was again passed through the separator to maximize tantalum recovery. The products were assayed to determine tantalum and tin distributions. The test showed that coarse tantalum minerals were easily recovered, but fine sized tantalum minerals were more easily lost to the non-magnetic product (table 2). Tin removal was somewhat higher when finer mesh material was used. This was at least partly related to wadginite, containing both tantalum and tin, reporting to the nonmagnetic fraction.

These results suggested that serious tantalum loss could occur if concentrates were ground too fine prior to the magnetic separation step. For the concentrate samples evaluated there seemed to be little need for grinding prior to magnetic separation. The cassiterite was quite well liberated from the tantalum minerals, with the exception of sample A where aggregates had formed during the air-drying step.

Methods of Separation

Evaluations were conducted to compare magnetic separation methods involving no grinding, preliminary grinding, and grinding of a middling product. A description of each process flow sheet follows.

The first separation method (figure 4) involved the following steps:

- 1) The sample was passed through a cross-belt separator operated at 0.72 Wb/m^2 to obtain a magnetic tantalum concentrate. A scalper on this separator removed tramp iron and highly-magnetic minerals at about 0.08 Wb/m^2 .
- 2) Magnetic material from step 1 was reprocessed and cleaned on a second separator at 0.72 Wb/m^2 .
- 3) The non-magnetic material from steps 1 and 2 was reprocessed to clean the cassiterite and recover fugitive tantalum. The magnetic tantalum concentrates recovered from each of the three steps were combined into a final tantalum product.

Preliminary removal of highly magnetic minerals and tramp iron is necessary in many magnetic separation systems because such materials interfere with normal equipment operation. The scalper product obtained during testing consisted primarily of iron and iron oxide minerals. It contained small amounts of tantalum, niobium, and traces of tin. To avoid unnecessary loss of tantalum, the scalper product was added back to the magnetic tantalum product.

The second flow sheet was identical to the first except that feed material was ground to about $-589 \mu\text{m}$ in a rod mill prior to magnetic separation.

The third flow sheet (figure 5) involved grinding of a middling product. Feed material was first passed through a cross-belt magnetic separator at 0.38 Wb/m^2 to recover a magnetic tantalum concentrate. As with the earlier flow sheets, a scalper on this cross-belt separator removed tramp iron

and other highly magnetic minerals at about 0.08 Wb/m². The magnetic tantalum product was cleaned on a second cross-belt separator at 0.38 Wb/m². Non-magnetic material from these two separators was reprocessed at 0.73 Wb/m² to recover locked and less magnetic tantalum minerals. Magnetic tantalum product from this third separator was ground to about -589 μ m to improve liberation. This ground product was reprocessed on a fourth separator at 0.73 Wb/m² to recover cleaned magnetic tantalum concentrate, which was combined with the magnetic products from the first two separation steps. The non-magnetic materials from the third and fourth separators were combined to form a cassiterite byproduct.

Results obtained by testing each separation flow sheet are provided in table 3. Evaluation of the three flow sheets indicate that, where possible, grinding should be avoided because the resulting fine tantalum minerals are more difficult to recover during the magnetic separation step. Discussion of specific test results follow.

For tests involving sample A, tin separation was improved when the aggregates were broken down by brief grinding prior to magnetic separation. Only a small amount of tantalum was lost to the tin byproduct. This preliminary grinding approach yielded the best results in terms of product recovery and grade. Grinding only the middling product lowered grinding requirements but an additional magnetic separation step was needed.

Only two flow sheets were evaluated using sample B. Preliminary grinding to -589 μ m followed by magnetic separation produced results nearly identical to those obtained when no preliminary grinding was used.

Sample C was tested using only the method that involved grinding of a middling product. While the amount of tin removed was quite high (78%), tantalum loss to the tin byproduct was somewhat more than desired (4.5%). The loss of tantalum to the tin byproduct was proportional to the amount of tin byproduct obtained. Tin byproducts using samples A and C assayed 4.6 and 5.0% Ta₂O₅, respectively.

The cassiterite byproducts could be upgraded by eliminating quartz, mica, and other gangue minerals through gravity separation methods.

Application of Results

Tests showed that Alabama tantalum concentrates with 22 to 29% SnO₂ can be magnetically upgraded. As much as 78% of the tin was removed during magnetic separation. The resulting cassiterite byproduct contained 70 to 74% SnO₂. The magnetic tantalum product contained 54 to 60% Ta₂O₅, 9 to 11% Nb₂O₅, and 10 to 12% SnO₂. Meeting Defense Logistics Agency tin specifications would require blending these magnetic tantalum concentrates with low-tin tantalite or columbite concentrates from other zones within the deposit. To aid in this dilution, other low-tin materials, like

magnetite or sand, could also be used.

This information was used by O'Dell Construction Co. Inc. in preparing grade 1 concentrate with 45 400 kg contained Ta₂O₅ for the National Defense Stockpile. Some blending of the magnetic tantalum product with material containing little or no tin was required in order to meet tin specifications.

References

Foord, E. E. and Cook R. B., 1989, "Mineralogy and Paragenesis of the McAllister Sn-Ta-Bearing Pegmatite, Coosa County, Alabama," Canadian Mineralogist, Vol. 27, pp. 93-105.

Wouden, M. L. and Chatman, M. L., 1991, "Tantalum and Niobium From Wyoming Pegmatites," 91-43, Society for Mining, Metallurgy, and Exploration, Inc., Littleton, Colorado.

Table 1. Chemical analysis of dried concentrates, in percent

| Constituent | Sample A | Sample B | Sample C |
|--------------------------------|----------|----------|----------|
| Ta ₂ O ₅ | 48.2 | 46.0 | 37.0 |
| Nb ₂ O ₅ | 8.5 | 8.4 | 7.4 |
| SnO ₂ | 21.6 | 21.6 | 29.2 |
| TiO ₂ | 1.2 | NA | 1.4 |
| Sb | <.006 | NA | <.006 |
| As | <.01 | NA | <.01 |
| P | .14 | NA | .17 |

NA Not analyzed

Table 2. Tantalum and tin distributions to non-magnetic products as a function of particle size

| Screen size, μm | Distribution to non-magnetic, % | |
|----------------------------|---------------------------------|----|
| | Ta | Sn |
| -1180 +589..... | 0.6 | 57 |
| -589 +300..... | 2.5 | 61 |
| -300 +147..... | 3.6 | 62 |
| -147..... | 6.7 | 64 |

Table 3. Comparison of separation methods

| Sample | Grinding method | Product type | Distribution, % | | | Analysis, % | | |
|----------|-----------------|----------------|-----------------|------|------|--------------------------------|--------------------------------|------------------|
| | | | Ta | Nb | Sn | Ta ₂ O ₅ | Nb ₂ O ₅ | SnO ₂ |
| A..... | None | Ta concentrate | 99.2 | 99.2 | 50.4 | 56.8 | 9.4 | 12.2 |
| | | Sn byproduct | .8 | .8 | 49.6 | 2.7 | .5 | 72.1 |
| | Preliminary | Ta concentrate | 98.7 | 98.7 | 41.0 | 59.3 | 9.9 | 10.4 |
| | | Sn byproduct | 1.3 | 1.3 | 59.0 | 3.7 | .6 | 70.4 |
| Middling | Ta concentrate | 98.2 | 98.2 | 40.4 | 55.3 | 8.9 | 10.7 | |
| | Sn byproduct | 1.8 | 1.8 | 59.6 | 4.6 | .7 | 73.8 | |
| B..... | None | Ta concentrate | 98.7 | 98.6 | 39.0 | 59.7 | 11.1 | 10.8 |
| | | Sn byproduct | 1.3 | 1.4 | 61.0 | 3.5 | .7 | 73.9 |
| | Preliminary | Ta concentrate | 98.3 | 98.4 | 38.0 | 60.1 | 11.0 | 10.5 |
| | | Sn byproduct | 1.7 | 1.6 | 62.0 | 4.4 | .8 | 73.4 |
| C..... | Middling | Ta concentrate | 95.5 | 95.5 | 21.6 | 54.0 | 10.9 | 9.9 |
| | | Sn byproduct | 4.5 | 4.5 | 78.4 | 5.0 | 1.0 | 69.7 |

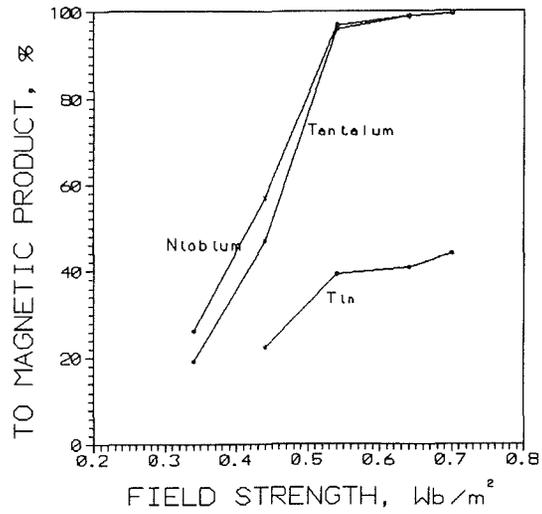


Figure 1. Effect of field strength on metal recovery

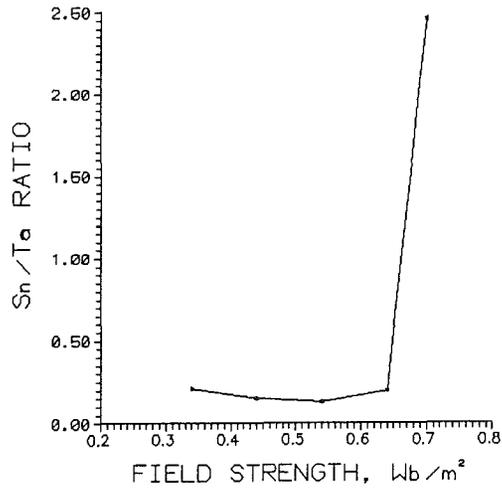


Figure 2. Effect of increasing field strength on the Sn/Ta weight ratio in magnetic products

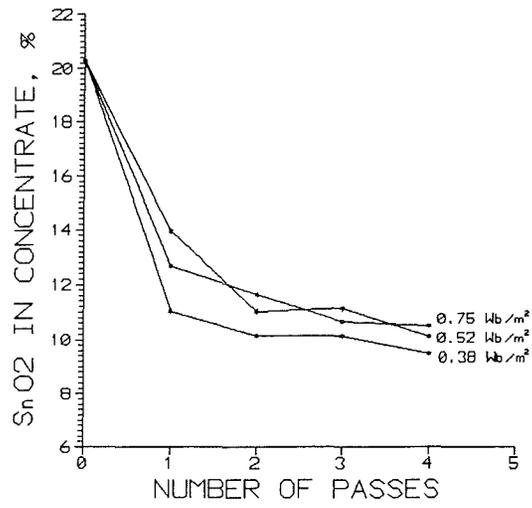


Figure 3. Tin assays of magnetic concentrates after multiple passes through the magnetic separator at constant field strength

Figure 4. - Basic separation flow sheet.

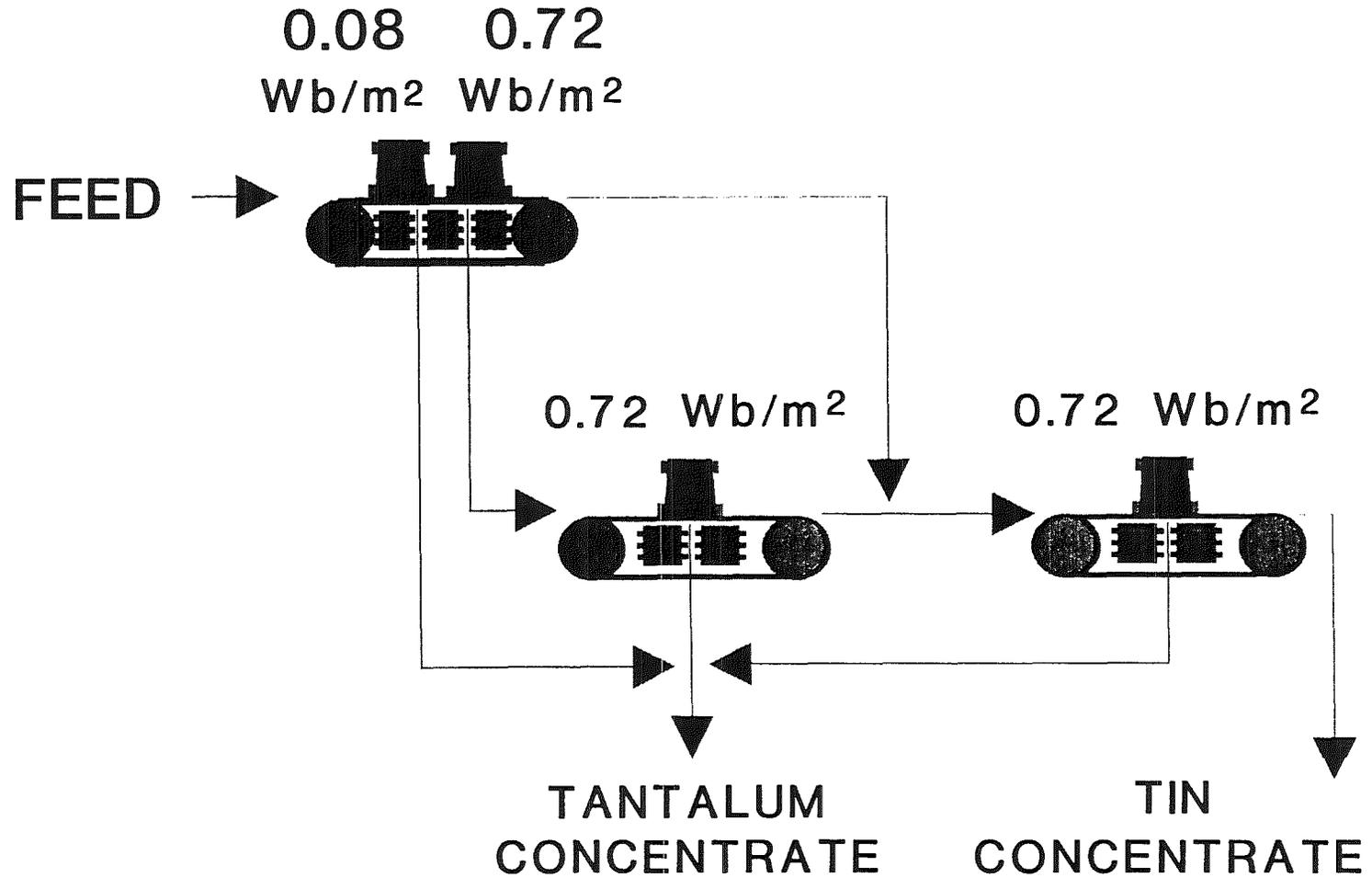


Figure 5. - Flowsheet with grind of middling.

