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MINERALOGICAL NOTES

MOLYBDENITE $2H_1$, MOLYBDENITE $3R$, AND JORDISITE FROM CARRIZAL ALTO, ATACAMA, CHILE

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ABSTRACT

Three molybdenum disulfide modifications have been confirmed by X-ray powder and electron microprobe analysis of specimens from Carrizal Alto, Chile. Textural relations indicate the crystallization sequence, molybdenite $2H_1$-molybdenite $3R$-jordisite. The jordisite and, less probably, rhombohedral molybdenite, may have formed under supergene conditions.

OBSERVATIONS

Crudely botryoidal encrustations, 5–10 mm in thickness, on the walls of cavities in a 15-25 mm quartz vein exposed in two specimens collected from the dumps of the Carrizal Alto copper mines, Atacama Province, Chile (at. 28°5.5′S.; Long. 70°55″W.; Ruiz, 1965, pp. 211–214), have been found to consist of three distinct modifications of molybdenum disulfide.

On binocular examination of freshly broken surfaces, the rounded aggregates are seen to have developed over rosettes of coarse-grained, flaky molybdenite. The cores are mantled by 3–4 mm-thick layers, with a finer grain size, a poorly-defined radiate habit, and a markedly reduced luster. This second phase is itself coated and veined by a black, extremely fine-grained, sectile material, which everywhere constitutes the surface of the aggregates. In polished sections of impregnated specimens, the two inner zones are confirmed to be molybdenite, while the outer constituent exhibits only slight and confused anisotropic effects and a considerably lower reflectance.

X-ray powder patterns of the core and intermediate phases, taken in a Debye-Scherrer 114.6 mm.-diameter diffraction camera (Ni-filtered Cu-Kα radiation) are characteristic of the $2H_1$ and $3R$ polytypes of molybdenite, respectively (Traille, 1963; Graeser, 1964; Takéuchi and Nowacki, 1964; Wickman and Smith, 1970). Careful examination of the relative intensities of X-ray reflections yielded by several samples drilled serially from the cores to the outer margin of the intermediate zone shows that the boundary between the zones represents an abrupt change in molybdenite structure; no patterns indicative of intergrowths of the two polytypes were obtained (*cf.* Frondel and Wickman, 1970).

In contrast to the two inner phases, the sectile veinlets and coatings yield only very poor powder patterns, exhibiting only very faint traces of the stronger lines of molybdenite $3R$. These may reasonably be ascribed to contamination by the enclosing molybdenite, and it is concluded that this third constituent is probably amorphous.

X-ray scanning photographs for Mo-Lα and S-Kα taken on an electron microprobe reveal that these elements occur in sensibly homogeneous and identical concentrations in all three phases in the encrustations, in amounts equal to those found for a coarse-grained molybdenite-$2H_1$ from Knaben, Norway. No additional elements could be detected by microprobe analysis, and only traces of Re and Fe were revealed by optical spectrography. All three phases appear to approach MoS_2 in composition.

DISCUSSION

Molybdenite-$3R$ has been shown (Vorma, Kallio, and Meriläinen, 1966; Mandarino and Gait, 1970; Frondel and Wickman, 1970) to be distinctly less abundant than the $2H_1$ modification, although it appears to predominate in certain ore deposits. The coexistence of the two forms has been confirmed at several localities, and evidence for the conversion, during the hypogene development of an ore, of one polytype to the other has been presented (Clark, 1970). The textural relations observed in the specimens from Carrizal Alto are not diagnostic of the *replacement* of hexagonal molybdenite by the rhombohedral modification, and could merely reflect overgrowth of one polytype on the other. However, the occurrence of malachite and goethite as thin coatings on the specimens testifies to the local activity of supergene, oxidizing solutions. Further, molybdenite in several specimens of unaltered, hypogene ore from this and other vein deposits in the Copiapó mining district consists solely of the hexagonal modification, suggesting that the molybdenite—$3R$ may here be a product of supergene alteration.

The author (1970) has deduced that the rhombohedral molybdenite polytype is slightly enriched in molybdenum relative to the more common hexagonal form, a conclusion supported by the experimental data recently presented by Zelikman *et al.* (1969), and has suggested that this is the major factor determining the geological occurrence of the two modifications (*cf.* Frondel and Wickman, 1970). If this observation is generally valid, the molybdenite overgrowths in the Carrizal Alto ores may be inferred to reflect a lowering of sulfur fugacity, either at a late stage in the hypogene development of the deposit, or during supergene alteration.

The black, apparently amorphous phase which clearly veins molybdenite-$3R$ in these specimens is a molybdenum disulfide, and may probably be equated with the jordisite of Cornu (1909). Although Hess (1925), among others, has questioned the identification of this phase as a molybdenum disulfide, Staples (1951) and, less definitively, Granger and Ingram (1966) have subsequently presented analytical evidence that jordisite is indeed black, amorphous MoS_2. The occurrence of this mineraloid at Carrizal Alto differs from those previously described in that ilsemannite is apparently absent, and jordisite itself has the appearance of a secondary product. Wulfenite and tungstenian wulfenite, the oxidation products of molybdenite characteristic of this district (Clark and Sillitoe, 1970), have not been observed in specimens from the Carrizal Alto mine dumps, suggesting either that a distinct oxidation regime prevailed locally, or that the jordisite formed during an episode of supergene sulfide *enrichment*. It is not, however, certain that the jordisite formed as a supergene phase, or, if that were the case, whether its restriction to the molybdenite-3*R* zones indicates that it developed through selective replacement of that mineral, or that the assemblage, molybdenite$-3R+$jordisite, represents a breakdown product of hexagonal molybdenite.

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A RAPID SAMPLE PREPARATION METHOD FOR POWDER DIFFRACTION CAMERAS

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ABSTRACT

A fiber pulled from a mixture of sample and liquid collodion is a rapid and satisfactory mount for X-ray powder cameras.

The following method is based on a modification of the well known collodion mount.

A few milligrams of powered sample (<200 mesh) are placed on a glass slide. A drop or two of collodion is added to the powder. The mixture is then rapidly stirred with a sharp needle. After 20–30 seconds, as the collodion begins to set, the needle is lifted from the mixture. As the needle is withdrawn a thin fiber of collodion-bound sample will form. If a fiber fails to form, the viscosity of the sample-collodion mixture is still too low. The mixture is continuously stirred until a fiber of from 0.05–0.2 mm in diameter and from 1–2 inches in length is formed. A fiber of these dimensions can be produced in less than one minute.

The end of the fiber is held between the thumb and forefinger of the free hand and pulled slightly in order to keep it under tension. Tension is applied for about one minute to allow the collodion to set. The fiber, still attached at one end to the stirring needle, should then be put aside for about 10 minutes to allow the collodion to set more completely. The earlier the needle is withdrawn from the sample-collodion mixture, the thinner the resulting fiber. However, if the fiber becomes excessively thin, it will have a tendency to curl after drying. Two or three samples of 10–15 mm in length can then be cut from the fiber with a sharp safety razor. The simplicity of the method is such that any one should be able to prepare samples of proper dimensions after a few trial runs.

The advantages of this method are: 1) Rapidity of sample preparation.