Rhombohedral molybdenite from the Minas da Panasqueira, Beira Baixa, Portugal

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Summary. Rhombohedral (3R) molybdenite has hitherto been reported from only one locality. Its mode of occurrence at a new locality is reported in this paper. At Panasqueira it occurs as a rare accessory in greisenized granite with which wolframite–cassiterite–quartz veins are associated. In the veins themselves only normal hexagonal (2H) molybdenite has been detected.

AN X-ray diffraction study of accessory molybdenite in a greisenized granite at the Minas da Panasqueira, near Fundão, Beira Baixa, east-central Portugal, has confirmed the occurrence of the rhombohedral polytype of this mineral, molybdenite-3R. Until Traill (1963) found rhombohedral molybdenite in quartz–feldspar porphyry at the Con mine, Yellowknife, Northwest Territories, Canada, it was thought that natural molybdenite was invariably hexagonal (Morimoto and Kullerud, 1962). The structure and synthesis of rhombohedral MoS₂ have been discussed by Bell and Herfert (1957), Jellinek *et al.* (1960), Zelikman *et al.* (1961), and Semiletov (1962). Traill (1963) lists indexed powder data for natural hexagonal and rhombohedral molybdenite. Further polytypes may be found to occur naturally, and Traill recommends the adoption of the classification proposed for SiC by Ramsdell (1947).

The geology and mineralogy of the Panasqueira wolframite–cassiterite mineralization have been described by Thadeu (1951) and by Bloot and de Wolf (1954). The author spent ten weeks on the mine in 1959, and has studied selected aspects of the mineralization.

Towards the close of the Hercynian orogeny a system of flat-lying joints in phyllites of the Infracambrian Beira series controlled the emplacement of a post-tectonic granite cupola and of the associated quartz veins. The granite shows an unbroken crystallization sequence from magmatic, through endoblastic (formation of K-feldspar, muscovite, and quartz), to hydrothermal conditions (Clark, 1960, 1964). The

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economic mineralization is considered to have been the immediate result of the greisenization and silicification of the parent granite. The composition of arsenopyrite, blende, and pyrrhotine in the apical zone of the granite suggest that intrusion took place at $\sim 700°$ C under a confining pressure of only 500–1000 bars.

Molybdenite is a rare constituent of the mineralized veins (Fonseca, 1943; Thadeu, 1951), and occurs sporadically in the Barroca Grande and Rebordões sections of the mine. Deposition of this mineral apparently preceded that of the other sulphides, blende and chalcopyrite, and, in part, of the arsenopyrite, although diagnostic textural relationships are lacking. Small (< 2 mm), highly contorted flakes of molybdenite infill sinuous fractures in quartz and wolframite. X-ray examination of four molybdenites from the quartz veins has shown that the normal hexagonal polytype is alone present.[1]

Megascopically similar flakes (< 1 mm) of molybdenite occurring sparsely disseminated in irregular veinlets of sugary quartz in the greisenized granite (Level 1, Panel 4, northern crosscut, Barroca Grande section) have, however, given powder patterns identical to that of molybdenite-3R (Traill, 1963, table 1).[2] Blende (16·3 mol. % FeS) and pyrite occur in the greisen in the vicinity of the veinlets and, together with the molybdenite, were probably deposited towards the close of the endoblastic stage of crystallization of the granite. Coarse muscovite and minute needles of rutile (perhaps resulting from the replacement of Ti-bearing biotite by muscovite and chlorite) form discontinuous selvedges around the veinlets. The rhombohedral molydenite is apparently indistinguishable in hand specimen and in reflected light from the hexagonal form, but no quantitative measurements of optical properties have been made. Qualitative X-ray fluorescence analysis of the 2H and 3R polytypes showed that no minor elements exceeded 50 ppm, with the exception of iron (~ 150 ppm). Estimation of tungsten was not possible with the fluorescence equipment available; little is known of the extent of solid solution between molybdenite and tungstenite in natural assemblages. In an experimental study of the Fe–Mo–S system,

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[1] Examination of some 55 specimens from the Ylöjärvi and Mätäsvaara deposits, Finland, and from Knaben, Norway, has also revealed only hexagonal molybdenite.

[2] The specimens were prepared for diffractometer examination by grinding between sheets of sand paper. Prolonged grinding of hexagonal and rhombohedral molybdenite was found to have no effect on the angular position of the reflections. The finely ground mineral was pressed gently, with silicon internal standard, into glass cavity mounts; preferred orientation of the particles does not appear to have been serious. Ni-filtered Cu-$K\alpha$ radiation was used for all runs.

Kullerud and Buseck (1962) have demonstrated that solid solution between FeS_2 and MoS_2 is negligible at 724° C; the minor amounts of Fe reported in natural molybdenites are probably due to mechanical impurities.

Morimoto and Kullerud (1962) have studied phase relations in the Mo–S system. They found that MoS_2 synthesized below 900° C showed a series of diffuse reflections conforming to neither the hexagonal nor the rhombohedral structures and simulating diffraction effects ascribed to stacking faults in layered structures. The broadness of the peaks increased with decreasing temperature. Above 900° C normal hexagonal MoS_2 was formed. On this rather meagre evidence it was suggested that rhombohedral molybdenite was metastable throughout the entire temperature range (to ~1350° C). Apparently it was not possible to synthesize molybdenite-3R from the elements. The occurrence of molybdenite-3R at the Con mine and at Panasqueira[1] suggests that further investigation of the conditions of formation and possible stability field of rhombohedral molybdenite as well as a systematic study of the structure of naturally occurring molybdenite are needed.

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[1] Traill (written communication) has recently identified molybdenite-3R in a hand specimen from an unknown locality.