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A MICROPROBE-HOMOGENEOUS INTERGROWTH OF GALENA AND MATILDITE FROM THE NIPISSING MINE, COBALT, ONTARIO

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Abstract

Material from the Nipissing mine, Cobalt, Ontario, identified originally as schirmerite on the basis of its *X*-ray powder pattern, was proven to be a microprobe-homogeneous intergrowth of galena and matildite. The intergrowth could not be etched by standard methods. The etch technique which resolved the problem consisted of exposing a polished section, without immersion, to calcite vigorously effervescing in concentrated nitric acid. This caused the galena to blacken, but left the matildite unaffected.

The powder pattern given for schirmerite as #175 in the Peacock Atlas should be discarded in favour of the data given by Karup-Møller (1973).

Sommaire

Un spécimen provenant de la mine Nipissing, à Cobalt, Ontario, identifié d'abord comme schirmérite, au vu de son diagramme de poudre aux rayons X, s'est avéré être un agrégat intime de galène et de matildite homogène à la microsonde. Cet édifice résiste à toute attaque à l'acide faite par les méthodes habituelles. La technique qui résoud le problème consiste à exposer une section polie, sans immersion, à de la calcite en forte effervescence dans de l'acide nitrique concentré. Sous cette action, la galène noirçit et la matildite reste intacte.

Le diagramme de poudre donné pour la schirmérite au No. 175 de l'Atlas Peacock doit être abandonné et remplacé par les données de Karup-Møller (1973).

(Traduit par le journal)

Introduction

Sufficient rare mineral species from the Cobalt camp have been reported in the literature to warrant the undertaking of a program of routine examination of any specimen which appears in the least unusual. This paper is based on the investigation of a 2 to 3 mm thick layer of a compact grey sulfide, adhering to one side of a superb specimen of native bismuth, collected about 1907 from the early workings of the Nipissing mine. The sulfide was initially assumed to be matildite; however, since the powder pattern of a "pure" sample closely matched the pattern given for schirmerite (#175 in the Peacock Atlas of Berry & Thompson 1962) further work was undertaken. The recent study of schirmerite by Karup-Møller (1973) had cast serious doubt on the validity of the pattern given for schirmerite in the Peacock Atlas. It therefore seemed desirable to characterize the mineral to which this pattern actually belonged.

Description

The bulk of the specimen is composed of native bismuth in the form of an ellipsoidal pod some 9×4×2 cm in size. The surface of the bismuth is coated with a thin layer of granular calcite containing a few large (3 mm) subhedral quartz crystals. The grey sulfide occurs as a surface deposit in only one area, some 5×3 cm in size, and is in direct contact with the bismuth. No other minerals have been found on the specimen. Since it appears obvious that the bismuth formed as a pod in a granular white calcite vein, and further that there is a complete absence of native silver, argentite and galena, it can be assumed that the specimen is probably from the Cobalt Hill vein on the Nipissing property.

In hand specimen the material forms anhedral compact masses, having a subconchoidal to somewhat uneven fracture, with no visible cleavage or parting. On a fresh break it is dark steel-grey in colour, but rapidly tarnishes to iron black. The average Vickers microhardness, measured at a 10g load, is 47.2, with a range of 45.5 to 50.1; this is equivalent to a Mohs hardness of 2.5 to 3. The average density of seven fragments, measured by flotation in toluene using a Berman balance, was 7.02 with a range of 6.99 to 7.03.

The sulfide has formed in direct contact with the surface of the bismuth mass, frequently on the prominent {001} cleavages of the bismuth. In polished section this contact is sharply defined, though somewhat undulatory. Both the bismuth and the sulfide phase are optically homogeneous in polished section, and contain neither inclusions nor *visible* intergrowths of other minerals. The sulfide is of completely uniform texture and very rarely shows evidence of fracturing. Such fractures as are present are fresh and without alteration rims; this distinguishes the material quite clearly from that described by Karup-Møller (1973).

Analytical

Electron microprobe analyses of the sulfide and the bismuth were obtained at 20 kV using pure metallic elements as standards for Bi, Ag and Sb; galena was used for Pb and synthetic pyrrhotite (39.6% S) for S. Other elements searched for but not detected were Cu, Fe and Zn. The only impurity detected in the bismuth was 0.02% antimony (corrected microprobe sum before normalization = 99.63).

Five sulfide grains from various portions of the specimen were analyzed. As the observed counts were virtually identical in all cases, they were averaged to form a composite analysis (Table 1). Numerous scans made across each of the grains confirmed that the material was homogeneous to the microprobe.

The powder pattern for the sulfide material is given in Table 2, together with the pattern given for schirmerite in the Peacock Atlas and the patterns of galena and matildite. The pattern given by Karup-Møller (1973) for schirmerite bears little resemblance to any of these. The powder pattern for the sulfide material, and the fact that Ag/Bi determined by microprobe was essentially exactly that of matildite, strongly suggested that the material was an intergrowth. A minute, somewhat more coarsely-crystalline fragment was selected and mounted on the precession camera. The resulting photographs were consistent with two species diffracting simultaneously. Comparison with the original precession photographs of matildite (kindly loaned by Dr. D. C. Harris; Harris & Thorpe 1967) indicated that approximately half of the fragment was indeed matildite (dial axis $\simeq a$ axis) and that the remainder was galena (dial axis $\simeq$ [111]).

This left unexplained both the microprobe homogeneity and the total lack of reaction (except for a uniform blackening of the specimen) to any of the standard etch tests, including KCN, which is highly recommended by Ramdohr (1969). A number of experimental etching solutions were tested on additional polished sections. The results were unpromising except in one case, in which the nitric acid used continued to react

TABLE 1. MICROPROBE DATA

Element	Nipissing Mine Material[1]	Theoretical ½ $PbS.AgBiS_2$
Pb	21.98	20.69
Ag	20.47	21.55
Bi	41.20	41.75
Sb	0.24	-
S	16.11	16.01

[1] Corrected sum = 99.40.

TABLE 2. COMPARATIVE POWDER PATTERNS (Ni-FILTERED Cu RADIATION)

Nipissing mine intergrowth		Peacock Atlas #175		Matildite (Harris & Thorpe 1967)		Galena (Peacock Atlas)	
I_{est}	d_{meas}Å	I	d(Å)	I	d(Å)	I	d(Å)
1	6.30			3	6.311		
		2	6.11				
3	3.419	3	3.39	2	3.453	9	3.44
6	3.305	7	3.27	8	3.302		
4	3.164	3	3.13	2	3.166		
				1	3.074		
4	2.960	4	2.93			10	2.98
10	2.828	10	2.82	10	2.827		
3	2.803						
1 b	2.581	1	2.60	2	2.582		
				1	2.356		
1	2.152			1	2.149		
3	2.093	3	2.08			8	2.10
3	2.031			5	2.029		
5	1.966	6	1.961	6	1.966		
2	1.784	2	1.781			5	1.791
1 b	1.731	½	1.722	2	1.729		
3	1.710	6	1.710	3	1.709	3	1.710
2	1.670	1	1.672	1	1.671		
1 b	1.651	1	1.650	1	1.650		
				½	1.600		
1 b	1.581	2	1.585	2	1.580		
½	1.482	1	1.478	1 b	1.468	2	1.486
1	1.414	3	1.412	2	1.412		
½	1.358	1	1.356			3	1.361
2	1.324	4	1.323	2	1.333		
2	1.320					4	1.321

cessful technique resulting from this was to hold the polished section over a beaker in which calcite was vigorously effervescing in concentrated nitric acid. This rapidly blackened the galena with a small calcite vein cutting the sulfide after the specimen had been removed from the solution. This revealed faint traces of an intergrowth in the area adjacent to the calcite vein. The succompletely, but left the matildite unaffected, as can be seen in Figure 1, which also shows the sub-hexagonal net of matildite blades. Photographs of similar intergrowths are given by Ramdohr (1969, Figs. 450 and 452). The area occupied by matildite in an enlargment of Figure 1 was measured by planimeter; on this basis, the galena content is approximately 45%. An analysis subsequently calculated for ½ $PbS{\cdot}AgBiS_2$ is given in Table 1, and is very similar to that obtained for the intergrowth from the microprobe.

Conclusions

The powder pattern (#175) given for schirmerite in the Peacock Atlas can now be considered

FIG. 1. Oil-immersion photograph (1000×) of an etched grain. The black areas are galena and the light-grey blades are matildite.

discredited as representing a mixture of galena and matildite, and should be replaced by that given by Karup-Møller (1973). In view of the well-known painstaking care exercised by the late R. M. Thompson, it is almost certain that this pattern represents an example of an "unetchable" intergrowth such as that described above, and as such may be taken as a clear warning to all ore mineralogists.

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