

Cobalt-, nickel-, and iron-bearing sulpharsenides from the North of England

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SUMMARY. Within the Alston Orefield of the North Pennines, glaucodot and gersdorffite have been found in samples from Tynebottom Mine, Garrigill, and zoned gersdorffite has been found from Nenthead and the Great Sulphur Vein. At Scar Crag in the English Lake District, glaucodot and alloclaste (the first reported occurrence in the United Kingdom) occur associated with arsenopyrite and minor cobaltite and skutterudite. The mineralogy and parageneses of these associations have been studied by ore microscopy, X-ray powder photography, and electron probe microanalysis.

Electron probe microanalysis shows a considerable range in nickel content in the sulpharsenides from the Alston Orefield with a relatively constant Co:Fe ratio. Samples from Scar Crag contain no nickel but exhibit almost a complete range of Co:Fe ratios from FeAsS to CoAsS. The compositions of the Alston Orefield sulpharsenides, in particular, show them to be metastable phases when compared with data from synthetic studies. At Tynebottom Mine, glaucodot and gersdorffite overgrow arsenical marcasite, and at Nenthead and the Great Sulphur Vein, early pyrite framboids or euhedra act as cores to zoned gersdorffite crystals. The Scar Crag sulpharsenides occur in a quartz-chlorite-apatite vein with the glaucodot and alloclaste as overgrowths on arsenopyrite.

In the case of the Scar Crag association, consideration of the compositions of coexisting phases, together with precise determinations of the arsenic content of the arsenopyrites, has permitted speculation regarding temperatures and sulphur activities during ore formation. Estimated ranges are T c. 400 °C–300 °C and $a_{S_2} \approx 10^{-9}$ – 10^{-11} bar. The occurrence of the sulpharsenides in the Alston Orefield correlates with further geochemical differences compared to other Pennine ores, differences that have been linked to higher temperatures of formation and a magmatic contribution to the ore-forming fluid. The Scar Crag mineralization may be related to a postulated stock intrusion beneath Causey Pike and the geographical proximity of the Alston and Scar Crag occurrences does suggest the possibility of a genetic link.

SULPHARSENIDE minerals of cobalt, nickel, and iron have been found in two distinct associations within the Alston Block area of the North Pennines; cobalt and iron-bearing sulpharsenides have also been found in ore samples from Scar Crag in the English Lake District. The com-

positions, mineral properties, associations, and parageneses of these newly reported phases are presented here, together with some speculations as to the conditions of formation of the assemblages.

At Tynebottom Mine, Garrigill [NY 739 418], glaucodot and gersdorffite have been found in samples collected from dump materials. The occurrence of cobalt-bearing minerals from Tynebottom Mine confirms the suggestion by Dunham (1948) of the presence of an 'unknown cobalt arsenide or cobalt sulphide', which was made on the basis of abundant erythrite around the mine. A second association, which contains zoned crystals of gersdorffite, occurs both in material collected from Nenthead [NY 790 436] and from the Great Sulphur Vein [NY 740 390]. The mineralization at Scar Crag [NY 2060 2070] in the Lake District contains glaucodot and alloclaste associated with arsenopyrite and minor amounts of cobaltite and skutterudite. This is the first authenticated occurrence of alloclaste in the United Kingdom. Samples collected from surface exposures and obtained from the Kingsbury Collection at the British Museum have been studied in this work.

The geological setting of the mineral veins of the Alston Block and their general mineralogy has been discussed in detail by Dunham (1948). The Scar Crag Vein (also known as the 'Cobalt Vein') outcrops above the footpath from Braithwaite village to Sail and Scar Crag in Long Comb, about one kilometre to the west of Causey Pike. Strens (1962) considered that the vein, which trends a few degrees east of north, was emplaced along a pre-Bala tear fault in Mosser-Kirkstle slates of the Skiddaw Slates Group. Although minor amounts of cobalt- and nickel-bearing phases have been found at Coniston (Russell, 1925) and at Dale Head (by C. J. S.), the over-all assemblages are different from those at Scar Crag, which appears to be a unique association for the Lake District. Unsuccessful attempts to extract cobalt at Scar Crag have been recorded by Postlethwaite (1913).

Experimental methods. Samples have been studied using reflected light microscopy and

selected areas analysed by electron microprobe. The instrument used was a Cambridge Microscan V employing an accelerating voltage of 15 kV. Standards used were synthetic sulphides and sulpharsenides and the results were corrected by standard procedures. Certain samples were also examined by X-ray powder (Debye-Scherrer) photography using Co radiation. Vein material from Scar Crag was also examined in thin section.

Mineral data. At Tynebottom Mine, glaucodot and gersdorffite overgrowths on arsenical marcasite occur within a quartz, calcite, and ankerite gangue. The arsenical marcasite forms 1–2 cm by 0.2 cm laths, which are randomly oriented or collected into sheaf-like aggregates or rarely into large (2–5 cm) anhedral (see fig. 1). In oil immersion, it has a distinct green-white to pink-brown reflection pleochroism and a strong, green to brown anisotropy. The laths show a fine diamond-shaped

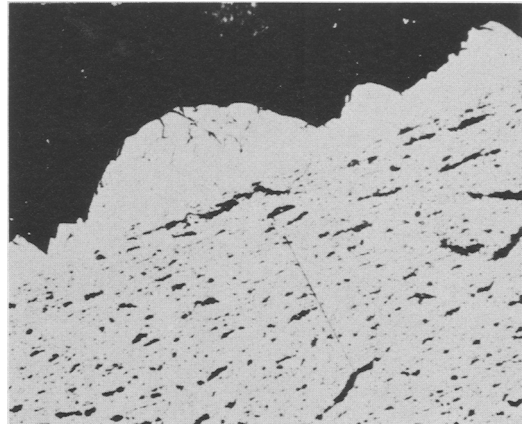


FIG. 1. Photomicrograph of a polished section showing euhedral-subhedral sulpharsenide overgrowths on lath crystal of arsenical marcasite, Tynebottom Mine, Garrigill.

TABLE I. Representative electron microprobe analyses of cobalt, nickel, and iron-bearing sulpharsenides (All data in wt. %.)

Locality/Mineral Species	Fe	Co	Ni	As	S	Total
<i>Alston Block (Tynebottom Mine)</i>						
Arsenical marcasite	39.83	3.34	1.24	8.60	46.67	99.68
	39.77	3.75	1.14	8.68	46.44	99.79
	44.19	0.88	0.12	4.13	50.55	99.87
Gersdorffite	4.86	11.45	19.28	46.70	20.02	102.30
	3.35	11.88	21.39	44.84	19.57	101.04
	2.36	7.03	24.49	45.95	18.82	98.65
Glaucodot	12.04	22.03	2.22	45.69	20.64	102.62
	9.86	21.06	5.80	43.98	21.93	102.62
	11.29	23.68	1.71	42.27	21.59	100.54
	11.24	24.27	1.56	40.41	23.92	101.41
<i>Lake District (Scar Crag)</i>						
Arsenopyrite	34.7	0.3	—	44.6	19.7	99.3
	35.0	0.3	—	45.4	20.1	100.8
	34.2	0.4	—	46.7	19.1	100.4
Alloclase	4.8	32.0	—	46.6	18.0	101.5
	4.9	32.0	—	46.4	17.3	100.6
	4.8	30.9	—	46.3	17.4	99.4
	5.9	29.5	—	47.3	16.9	99.6
	6.4	29.8	—	45.7	17.8	99.7
	6.1	29.5	—	47.4	16.9	99.9
	6.8	29.4	—	45.9	18.9	101.0
	5.6	30.1	—	47.3	18.4	101.5
	5.6	30.4	—	47.5	17.7	101.2
	6.7	28.2	—	48.1	16.9	99.9
8.0	27.1	—	48.3	16.8	100.2	
Glaucodot	11.9	23.61	—	46.2	19.4	101.1
	15.8	19.4	—	46.3	19.1	100.6
	13.4	22.3	—	45.8	18.8	100.3
	12.8	23.2	—	46.2	19.1	101.3
	24.0	10.6	—	45.4	19.1	99.1
	24.5	10.7	—	46.1	17.9	99.2
	22.4	12.8	—	46.7	17.2	99.1
Cobaltian Arsenopyrite	28.2	5.9	—	47.0	18.7	99.8
Cobaltite	2.0	33.6	—	44.7	19.0	99.3
Skutterudite	2.7	19.5	—	77.2	1.7	101.1

cleavage in addition to later fracturing and individual marcasite rhombs are composed of a complex intergrowth of small ($< 5 \mu\text{m}$) domains. Compared to normal marcasite it has a lower reflectivity and the yellow component of the reflection pleochroism is absent. Electron microprobe analysis (Table I) shows the marcasite commonly contains in excess of 8 wt. % arsenic as well as 3–4 wt. % cobalt and c. 1 wt. % nickel. Along the width of the marcasite laths, oriented tabular aggregates of pyrite or pyritohedra ($5\text{--}20 \mu\text{m}$ diameter) occur. The pyrite itself encloses small pyrrotine ($< 5 \mu\text{m}$) inclusions.

The cobalt-, nickel-, and iron-bearing sulpharsenides at Tynebottom form subhedral to euhedral overgrowths (up to $40 \mu\text{m}$ in width) on the edges of fractured marcasite or heal internal fractures. The overgrowths have two, or sometimes three, optically distinct phases, which have been identified by electron microprobe analysis. The relationships between these phases and the distribution of Co, Ni, Fe, As, and S between them are illustrated in fig. 2. The outermost phase has been identified as gersdorffite by electron microprobe analysis (see Table I). It displays a good cubic cleavage, cubic habit and is pink-yellow in colour under the ore microscope. It is isotropic to weakly anisotropic. The analytical data (Table I) show dominant nickel (c. 17–24 wt. %) with lesser cobalt (c. 7–12 wt. %) and minor iron (c. 2–6 wt. %). The phase within this has been identified as glaucodot by electron microprobe analysis (and X-ray powder diffraction). It contains dominant cobalt (c. 19–24 wt. %) with lesser iron (c. 9–12 wt. %) and minor amounts of nickel (c. 1–6 wt. %). This glaucodot exhibits slight pink to yellow-pink reflection pleochroism and moderate anisotropy. Both phases take a good polish and have reflectivities a little below that of pyrite on qualitative observation.

A thin zone, adjacent to the enclosed arsenical marcasite, appears an optically distinct third phase in some parts of the sections as it has a lower

reflectivity. The electron microprobe data, however, show that this phase has the composition of gersdorffite (fig. 2, Table I). Frequently, the sulpharsenide overgrowths display optical continuity with the enclosed marcasite. Minor amounts of euhedral gersdorffite (c. $10 \mu\text{m}$ diameter) also occur associated with anhedral chalcopyrite grains (c. $20 \mu\text{m}$ diameter).

In the material collected from the Great Sulphur Vein and from workings at Nenthead, euhedral zoned gersdorffite crystals have been found in quartz in association with anhedral chalcopyrite and pyrite. The gersdorffite was identified initially on the basis of its optical properties in comparison with the Tynebottom Mine material. Electron microprobe analysis showed the presence of major nickel, sulphur, and arsenic with some cobalt and iron, but it was not possible to obtain quantitative data for these small grains. Gersdorffite occurs as small ($5\text{--}10 \mu\text{m}$) discrete, lilac-white euhedral crystals within vein quartz and calcite, and within mineralized shaley limestones. Associated opaque minerals are chalcopyrite, galena, pyrite, and marcasite. The gersdorffite euhedra display cubic habit and cleavage (triangular polishing pits), and exhibit faint zoning characterized by colour differences (lilac to pink) and differences in reflectivity and hardness. The zoning is difficult to see in freshly polished material but the colour differences between zones gradually become enhanced. The zone boundaries follow the crystal outlines and frequently more than one (c. $2\text{--}3 \mu\text{m}$ wide) zone is present. The zoning is too fine in scale to enable compositional differences between zones to be determined by microprobe analysis. Small pyrite framboids or euhedra act as cores to many gersdorffite crystals and the gersdorffite itself is commonly enclosed and replaced by galena and chalcopyrite.

At Scar Crag, cobalt-iron sulpharsenide minerals occur with arsenopyrite in a quartz-chlorite-apatite vein. Quartz is by far the most dominant

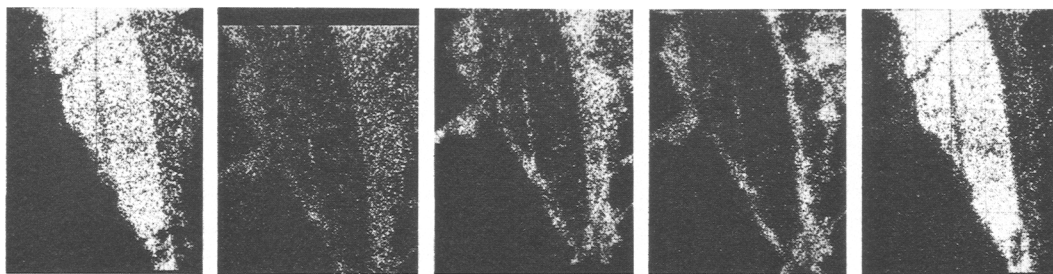


FIG. 2. Electron microprobe X-ray scanning micrographs of a bladed crystal of arsenical marcasite with sulpharsenide overgrowths (*bottom*), Tynebottom Mine, Garrigill: (a) sulphur K_{α} ; (b) arsenic K_{α} ; (c) cobalt K_{α} ; (d) nickel K_{α} ; (e) iron K_{α} .

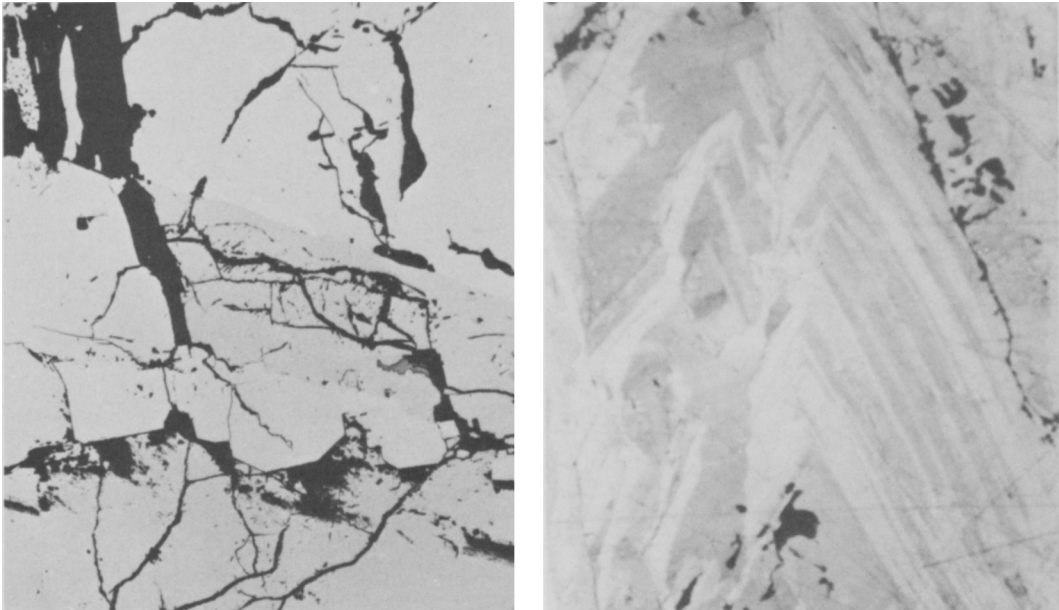


FIG. 3. Photomicrographs of polished sections of material from Scar Crag: (a, left) Arsenopyrite (bottom) overgrown by allose with cobaltite euhedra developed along a fracture in allose. (b, right) Zoning in glaucodot shown by etching with saturated ferric chloride solution (high-cobalt glaucodot is less etched than low-cobalt glaucodot and cobaltian arsenopyrite).

vein mineral; early quartz is often euhedral and with a zoned arrangement of inclusions. Rutile occurs within quartz either as radiating bunches of fine needles ($< 100 \mu\text{m}$ in length) or as subhedral grains ($< 30 \mu\text{m}$). Small amounts of tourmaline showing blue-brown pleochroism occur as subhedral to euhedral grains ($< 400 \mu\text{m}$) associated with early quartz. Abundant apatite occurs as large euhedra ($< 5 \text{ cm}$ in some specimens) growing inwards from the vein margins, and chlorite is also common. Arsenopyrite is the most abundant opaque mineral present, occurring as large euhedral and subhedral grains and aggregates ($< 3 \text{ mm}$). Electron microprobe analysis (Table I) shows that the arsenopyrite always contains some cobalt (usually $< 1\%$) and slight zoning is evident in polished section. This zonation could be related to the variations in S:As ratio but this was not observed in electron microprobe data, and Kretschmar and Scott (1976) have pointed out that 'optically zoned arsenopyrites are not necessarily compositionally zoned'. The arsenopyrite is overgrown by glaucodot or, more commonly, allose. Electron microprobe data for both phases are presented in Table I. In contrast to the Alston area sulpharsenides, no nickel is present in these minerals and there is a much greater variation in

the Fe:Co ratio, which was relatively constant in the former samples (Table I, fig. 4).

The identification of glaucodot and allose at Scar Crag has been substantiated by X-ray powder

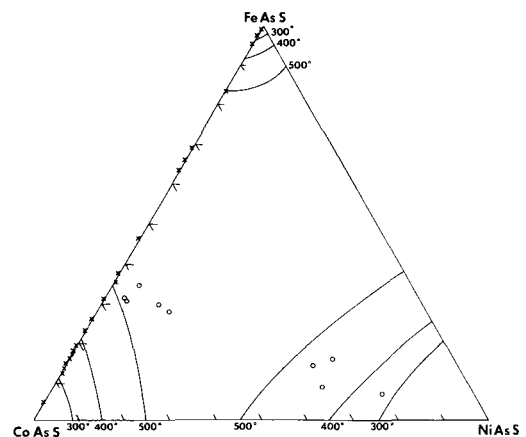


FIG. 4. Compositions of cobalt-, nickel-, and iron-bearing sulpharsenides from northern England plotted (in atomic percent) on the ternary diagram FeAsS-CoAsS-NiAsS (x Scar Crag phases; o Tynebottom phases). Also shown are the solid solution limits experimentally determined by Klemm (1965).

photography. The X-ray powder data obtained for glaucodot are closely comparable to published data and distinctly different to data obtained for the more cobalt-rich alloclose. Comparison of the data for Scar Crag alloclose with that published by Kingston (1971) and by Petruk *et al.* (1971) for the type material and samples from Cobalt, Ontario, respectively, support the identification. Although, as illustrated by our analysis, alloclose is a generally more cobalt-rich sulpharsenide than glaucodot, there is no evidence of a clear break in composition in the CoAsS–FeAsS solid solution series (fig. 4). The distinction between glaucodot and alloclose must be made on the basis of crystal structure, glaucodot being of orthorhombic space group *Cmmm* (Ferguson, 1946), whereas alloclose from the type locality is now known to be monoclinic, of space group *P2₁* (Scott and Nowacki, 1976). Although alloclose is not easily distinguished in polished section, it does have slightly lower polishing hardness and reflectivity than arsenopyrite and exhibits purple/mauve-greenish olive anisotropic tints under partly crossed nicols; arsenopyrite exhibits blue and orange tints whereas the colours exhibited by glaucodot are much less intense greys and browns.

As well as anhedral overgrowths (< 500 μm) on arsenopyrite, alloclose occasionally forms (< 100 μm) subhedra. It also sometimes encloses grains of skutterudite (< 20 μm) as confirmed by microprobe analysis (Table I). Cobaltite, also confirmed by electron microprobe (Table I) occurs as small euhedra (< 50 μm) at the outer margin of the alloclose, at the boundary between alloclose and arsenopyrite or along fractures in these two minerals. Other minerals observed are: marcasite, which rarely overgrows alloclose; native bismuth, which occurs as inclusions in arsenopyrite and early quartz; bismuthinite, which occurs along fractures in arsenopyrite and alloclose and at grain boundaries; and a few small (< 20 μm) molybdenite fibres.

Certain of the Scar Crag specimens exhibit a fine zonal texture with individual zones having compositions of cobaltian arsenopyrite, glaucodot, and alloclose. This faint zonation is best seen under partly crossed nicols and is enhanced by etching. Although previous studies of the mineralization at Scar Crag have resulted in reports of scheelite (Kingsbury and Hartley, 1957) and löllingite (Strens, 1962), neither of these minerals was found in the present study, despite considerable searching.

Mineral parageneses. At the Tynebottom Mine, the earliest phases are pyrite with very minor associated pyrrhotite. These are succeeded by the arsenical marcasite, which is in turn overgrown by the sulpharsenides—gersdorffite and glaucodot.

Some euhedral gersdorffite is also associated with later chalcopyrite and very minor arsenopyrite occurs within the carbonate gangue. Oxidation of the pyrite to limonite and of the gersdorffite to erythrite has occurred as a result of weathering, especially along cleavages.

The paragenesis at Nenthead and the Great Sulphur Vein is of early pyrite framboids or euhedra acting as cores to gersdorffite crystals, which have in turn been enclosed and replaced by galena and chalcopyrite. Weathering has resulted in the alteration of chalcopyrite to covelline, blaubleibender covelline, and limonite along cleavages and crystal edges; of galena to anglesite and covelline or cerussite and chalcosine; and of gersdorffite to limonite.

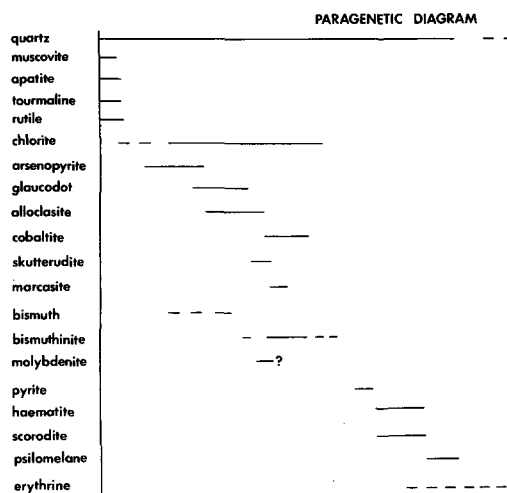


FIG. 5. The mineral paragenesis at Scar Crag, Lake District.

The Scar Crag paragenesis is illustrated in fig. 5. Quartz, rutile, tourmaline, and apatite appear to have been the first minerals introduced into the vein with minor muscovite, now mostly replaced by chlorite. Quartz was probably deposited throughout the main period of ore mineralization. The major ore minerals were deposited at the same time or shortly after these early minerals and arsenopyrite was the first of these phases. Arsenopyrite is overgrown (but not replaced) by later glaucodot and alloclose. Skutterudite occasionally is found enclosed by alloclose; cobaltite and marcasite formed later than alloclose. Native bismuth occurs within arsenopyrite and early quartz, bismuthinite is a slightly later phase. Minor molybdenite and pyrite occur as late minerals in the primary assemblage. There has been extensive later replacement of arsenopyrite by scorodite and

some replacement by hematite. Psilomelane is found as a coating on joint planes and erythrite has resulted from weathering of the cobaltiferous sulpharsenides.

Discussion. Very little data are available on phase relations in the ternary system FeAsS–CoAsS–NiAsS (or its constituent binary systems). Klemm (1965) has investigated equilibrium solid solution limits in the dry FeAsS–CoAsS–NiAsS system as a function of temperature and his results are shown in fig. 4, together with the sulpharsenide compositions determined in this work. According to these data, most of the sulpharsenides found in this work should form only above 300 °C and many only above 500°. A few analyses represent compositions that cannot be synthesized in the dry system. Estimates of the temperatures of mineralization in the Alston orefield (from fluid inclusion and isotope studies) indicate appreciably lower temperatures than 300 °C (c. 100–200 °C) (Sawkins, 1966; Smith, 1974). The sulpharsenide assemblages from the Alston orefield are certainly metastable, like the bravoites that are a significant minor feature of the mineralization of the South Pennines (Ixer, 1978). Klemm (1965) suggested widespread metastability of glaucodot by comparison of his experimental work with analyses from the literature.

The arsenopyrite in the mineral assemblage at Scar Crag can be employed as a geothermometer through determination of S:As ratios provided an independent estimate can be made of sulphur activity during ore formation. This technique, based on the work of Kretschmar and Scott (1976), also requires that the arsenopyrite be from an equilibrium a_{S_2} -buffered assemblage and contains a combined minor element content < 1%. Since the Scar Crag arsenopyrite generally only contains < 0.5% Co, the latter criterion is fulfilled. The question of equilibrium is much more difficult to assess, but it would seem worth while to apply the technique and then critically evaluate the results. Accurate determination of the wt.% As in the Scar Crag arsenopyrite samples is also essential and two methods have been employed here. Careful measurement of the position of the d_{131} line in X-ray powder diffraction data enables the determinative curve of Kretschmar and Scott (1976) to be used (the equation Atomic% As = $866.67 d_{131} - 1381.2$ defines this curve). The measured d_{131} values gave a range of 1.630–1.631 Å which leads to a range in at.% As = 32.42–31.55. These results are in good agreement with those obtained by the second method, electron microprobe analysis using synthetic arsenopyrite as a standard (if pure arsenic is used as a standard, results are up to 2% different due to differences in As coordination and bonding between element and sulpharsenide). The a_{S_2} during

the main phase of sulpharsenide formation can be approximately estimated by the formation of native bismuth and bismuthinite at this time (fig. 5). In fig. 6, the bismuth–bismuthinite sulphidation curve (Barton and Skinner, 1967) is plotted on a diagram showing variation of $\log a_{S_2}$ with temperature; also plotted are the limits of temperature and sulphur activity defined by the range of As content in Scar Crag arsenopyrite. Sulphidation curves for certain Fe–As–S phases are also shown along with the curve for the sulphidation of pyrrhotite to pyrite.

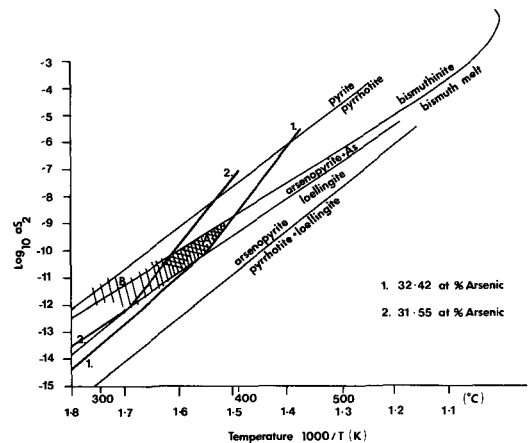


FIG. 6. Plot of sulphur activity ($\log_{10} a_{S_2}$) against temperature ($1000/T$ K) for sulphidation equilibria relevant to the Scar Crag assemblages. Also shown are sulphidation curves for arsenopyrite of limiting As:S ratios observed in those assemblages.

The early sulpharsenide assemblage appears to have been in equilibrium with bismuth and to have formed under the temperature (c. 400 °C) and a_{S_2} (c. 10^{-9} atm) conditions defined by the field 'A' in fig. 6. Later assemblages see the appearance of bismuthinite and cobaltite (which Maurel and Picot, 1973, relate to increasing S:As ratio). This could correspond to an increase in a_{S_2} or, more likely, just a drop in temperature. The eventual appearance of pyrite ties in with falling temperature and conditions closer to those shown by field 'B' (T c. 300 °C, a_{S_2} c. 10^{-12} atm). Although arsenopyrites of the composition range determined could coexist with bismuth at much lower values of a_{S_2} and somewhat lower temperature, this seems unlikely in view of the subsequent appearance of bismuthinite, cobaltite, and pyrite. The conditions of ore formation indicated here are well within the 'main-line' ore fluid environment outlined by Holland (1965) and other workers. The only comparable vein in the Lake District occurs in the Carrock Fell Tungsten deposit where Shepherd *et al.* (1973) obtained

temperatures of 295 °C (assuming 800 bar pressure) from fluid inclusions in mineralized quartz.

A factor of importance in the occurrence of these sulpharsenides in the Alston orefield is their complete absence from samples studied from the Derbyshire orefield of the South Pennines (Ixer and Townley, 1978). Other notable mineralogical differences between the two orefields include the presence of pyrrhotine in the Alston ores as a characteristic and early sulphide but its rarity in the South Pennines, and the occurrence within only the Alston galenas of a number of small (< 2 µm) crystallographically orientated inclusions, which probably account for the high silver and arsenic values reported by Small (1977). This correlates with other geochemical differences shown by the Alston orefield, such as a marked enrichment of rare-earth elements in fluorite (Smith, 1974), which has been suggested to indicate a magmatic contribution to the ore-forming fluid (Rogers, 1978). Fluid inclusion data from the Alston ores also indicate higher temperatures of formation and greater potassium content than the other Pennine orefields (Smith, 1974; Sawkins, 1966; Rogers, 1978).

The origin of the Scar Crag mineralization could be linked to the possible existence of a stock-like intrusion beneath Causey Pike (Rose, 1955). The slates in this area have been altered to a lighter and harder rock with minute dark spots and this lithology, termed Blakefell Mudstone (Eastwood *et al.*, 1931), has been interpreted as the metamorphic aureole of the supposed intrusion. Although there are certainly differences in the temperatures of sulpharsenide mineralization in the Alston orefield and at Scar Crag, and probably differences in age, their geographical proximity does suggest a possible genetic link.

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