Methane in sulphides from gold-bearing deposits, Britain and Ireland

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Abstract

The direct measurement of gases trapped in sulphide minerals shows that samples from gold-bearing deposits in Britain and Ireland are anomalously rich in methane. Chalcopyrite samples in deposits of Palaeozoic age sited in Neoproterozoic basement (Caledonides greenschist), Munster Basin (Variscides greenschist) and the Carboniferous cover (diagenetic) were compared using mass spectrometry of cold-crushed gases. All host sequences contain sources of organic matter. The content of non-aqueous gas is greater in both sets of greenschist-hosted deposits than in the diagenetic-hosted deposits. However, chalcopyrite accompanying gold in the Neoproterozoic is methane-rich, but in the low-gold Munster Basin it is methane-poor. These gas data from opaque minerals complement fluid inclusion data from gangue minerals, and add support to models for the involvement of organic species in orogenic gold mineralization.

Introduction

Models for mineralization in (meta-)sedimentary rocks commonly include gases within the mineralizing fluids, especially carbon dioxide, hydrogen sulphide and methane, which can influence solute transport and deposition (e.g. Landis & Hofstra 1991; Wilkinson 2001). Some supporting evidence is provided by fluid inclusion compositional data, but this is often limited to the dominant gas (usually CO_2) and to translucent minerals. An alternative approach is the analysis of gases released from fluid inclusions by crushing samples into a mass spectrometer at ambient temperature (Blamey 2012), which can measure multiple gases and from opaque minerals. The gases entrained in minerals include species liberated from the thermal alteration of organic matter, which could include variable mixtures of methane, higher hydrocarbons, hydrogen and carbon dioxide (Blamey 2012, Suzuki et al 2017).

One model for orogenic gold deposits involves the role of methane for complexing and precipitating gold (Naden & Shepherd 1989; Gaboury 2013; Goldfarb & Groves 2015; Jébrak et al. 2018). This was tested using deposits from Britain and Ireland (Fig. 1) by assessing if the compositions of gases in gold deposits are distinct among samples in three distinct settings. ~~As far as possible a~~All~~ll~~ samples are chalcopyrite of Palaeozoic age, to minimize variables, from the Neoproterozoic of Scotland and Northern Ireland, the Munster Basin of southwest Ireland, and widely distributed deposits attributed to Mississippi Valley Type (MVT). Chalcopyrite was used because it occurs in a wide range of host lithologies and redox environments, but lacks the prevalent cleavage found in galena and sphalerite that is conducive to the loss of entrained gases. We avoid pyrite because it is commonly zoned, and thus a single crystal can represent a range of entrapment conditions. The abundance of samples from the three categories reflects their relative distribution: The most widely distributed style of mineralization in Britain and Ireland is limestone-hosted vein deposits in the Carboniferous, considered to be ~~Mississippi Valley Type (~~MVT~~:~~ (Ixer 1986; Ixer & Vaughan 1993). The Carboniferous rocks are in the oil window. In addition to the deposits within the Carboniferous, sulphide veins in the sub-Carboniferous basement at Ballygrant, Leadhills, Conlig and Caldbeck are attributed to the lower part of the same mineralizing system (Anderson et al. 1989; Lowry et al. 1991; Moles & Nawaz 1996; Baron & Parnell 2005). These deposits occur particularly in dolostone as fillings of open space, in collapse breccias and as replacements of the carbonate host rock. They are epigenetic, emplaced after the lithification of the host rock. Fluid inclusion data indicate an origin from saline basinal brines at temperatures in the range 75 to 200 ˚C. Sulphur isotope data typically indicate thermogenic sulphate reduction (Thom & Anderson 2008). ~~The Carboniferous-hosted set includes a sample from Navan, which is of exhalative rather than MVT origin.~~ In the southwest, the Devonian-Carboniferous of the Munster Basin, adjacent to the Variscan orogenic front, experienced limited metamorphism to greenschist facies and contains copper ores (Wen et al. 1996; Spinks et al. 2016). Sediment-hosted copper mineralization occurs within reduced grey-green-purple sandstone, representing diagenetic reduced zones. Disseminated chalcopyrite, chalcocite and bornite occur interstitially between pores. Sulphur isotope compositions indicate mineralization by microbial sulphate reduction (Wen et al. 1996). Copper mineralization also occurs as veinlets within mineralized red beds, and in E-W trending quartz-barite veins up to 2 m wide, stratigraphically above the diagenetic sediment-hosted copper deposits. The minor and major vein mineralization is thought to be the result of remobilization from the diagenetic mineralization. In the northwest, the Neoproterozoic (Dalradian Supergroup) basement is similarly metamorphosed to greenschist facies, and contains chalcopyrite in gold mines at Cononish (Scotland) and Curraghinalt (Northern Ireland), and in a former gold prospect at Stronchullin (Pattrick et al. 1988; Gunn et al. 1996; Parnell et al. 2000). These are orogenic gold deposits, focussed on vein structures. At Cononish, sulphur isotope data indicate that the fluid that deposited most of the gold and sulphides could have been sourced from other metasedimentary units in the succession or was possibly of magmatic origin Spence-Jones et al. (2018). The age of the mineralization is the same as the last stage of crystallization in nearby granites, and the gold-bearing Rhynie Chert (Rice et al. 2012).

Paragenetic sequences for each sample type (Fig. 2) show that chalcopyrite is typically hosted in quartz in the Neoproterozoic and Munster Basin deposits, and in dolomite or other carbonates in the MVT deposits, and samples were selected from such veins (Table 1). Chalcopyrite was sampled from specimens with a simple mineralogy, so that it was not contaminated by other phases (Fig. 3). Gold in the Neoproterozoic, which occurs as tellurides or electrum, is coeval with chalcopyrite (Fig. 2). Chalcopyrite is coeval with other sulphides in the other deposit types, but was sampled where it is monomineralic.

Methodology

The cold crush method involves analysis by mass spectrometry conducted in high vacuum (Blamey 2012; Blamey et al. 2015). Each session was preceded and followed by analysis of 1 microlitre capillary tubes for calibration. Atmosphere was also introduced to verify the calibration using 100 to 200 acquisitions for both the sample and atmosphere standard. A match head sized sample (about 250 microns) is crushed incrementally under a vacuum of ~10^{-8} Torr, producing 4 to 10 successive bursts, which remained in the vacuum chamber for 8-10 analyser scans (~2 s) before removal by the vacuum pump. This method does not require a carrier gas and volatiles are not separated from each other but released simultaneously into the chamber. The act of incremental crushing may open a single inclusion or multiple fluid inclusions. The data acquisition is performed by means of two Pfeiffer Prisma quadrupole mass spectrometers operating in fast-scan, peak-hopping mode. Routinely the system analyses for the following gaseous species including H_2, He, CH_4, H_2O, N_2, O_2, Ar, and CO_2. The volatiles are reported in mol%. The instrument is calibrated using Scott Gas Mini-mix gas mixtures (with 2% uncertainty), capillary tubes filled with gas mixtures (with 1% uncertainty), and three in-house fluid inclusion gas standards. The 2-sigma detection limit for most inorganic species is about 0.2 ppm for aqueous fluid inclusions. Instrumental blanks were also analyzed routinely to assess if gases were produced during the crushing process. The mass spectra remained at background during crushing of blanks indicating that gases released are not sourced from the crushers or hardware. The amount of each species was calculated by matrix methods to provide a quantitative analysis, which is corrected for the instrumental background. Nine capillary tubes with encapsulated atmosphere were analyzed and yielded N_2/Ar ratios of 83.2 with a standard deviation of 1.4, within error of the atmospheric N_2/Ar ratio of 83.6. This translates into 0.5% accuracy for artificial inclusions made under laboratory conditions. Precision using natural inclusions for the major gas species measured is generally 2-5%, these being dependent on summed errors derived from instrument noise, linearity of the mass spectrometer, uncertainty of standards, blanks, interferences, and measurement of sensitivity factors. Before analysis, the crushing area and the bellows of the crusher were cleaned using potassium hydroxide. The apparatus is also routinely cleaned with isopropanol. Thereafter, the crushing chamber is baked at about 150-200 °C for 72 h before loading and analysing the samples at room temperature the next day.

Results

Data were successfully measured in ~~27~~ 25 sulphide samples (Fig. 1, Table 1). The gases in the Carboniferous set is consistently almost exclusively (>99 %) water, and this contain very low contents of carbon dioxide or methane. The data show no relationship to the Iapetus suture. The two greenschist-hosted sequences yield gas with a higher non-aqueous content (Fig. ~~2~~4), which is dominated by carbon dioxide (Table 1). The samples from the three gold-bearing localities in the Neoproterozoic additionally contain variable contents of methane. In contrast, the samples from the Munster Basin do not contain significant methane (Fig. ~~2~~4). The total gas yield for each sample is a weighted sum of incremental crushes: an example of the chemistry of successive crushes is given in Fig. ~~3~~5.

Discussion

Fluid types

The Carboniferous-hosted samples yield almost exclusively water, typical of inclusion fluids in sedimentary basins. This includes the samples at Tynagh and Gortdrum, where the host sediments have vitrinite reflectance values of about 4 % (Clayton et al. 1989), indicative of the gas window of hydrocarbon generation. The host rocks in the Munster Basin to the south have higher reflectance values of >5 % (Clayton et al. 1989) and are categorized as greenschist facies. The higher non-aqueous component in both of the greenschist-hosted sample sets is typical of metamorphosed sediments, in which carbon dioxide in particular becomes prevalent. The data for successive crushes in a single sample show that the relative proportions of gas are relatively constant (Fig. ~~3~~5). This is a pattern characteristic of a single assemblage of fluid inclusions, as multiple assemblages (such as a combination of primary and secondary inclusions) tend to exhibit progressive changes in chemistry as the assemblages split open with different ease (Blamey 2012). Thus the data indicate that carbon dioxide and methane occur together (Giggenbach 1980).

The inclusion of non-aqueous gases in both greenschist-hosted sequences is consistent with the observation of carbonic fluid inclusions in all three gold-bearing deposits in the Neoproterozoic (Pattrick et al. 1988; Baron & Parnell 2005; Parnell et al 2000) and in the Munster Basin (Wen et al. 1996). However, while carbon dioxide was inferred from thermometric data, the additional presence of methane was not recognised. The methane entrapped in the Neoproterozoic-hosted localities is newly recognized here.

Organic species

Each of the three sample sets occurs in a sequence containing black shales. However in the Munster Basin, the shales are limited and have not yielded carbon contents of oil-generating capacity (> 1%), whereas the Carboniferous further north (Cornford 1998) and the Neoproterozoic (Bata & Parnell 2014) have both sourced large volumes of hydrocarbons and so could yield methane. This may explain the lack of methane in the Munster Basin. There is a difference in the occurrence of organic species detected geochemically and petrographically. MVT deposits, to which the Carboniferous-hosted deposits are attributed, commonly contain solid oil residues (bitumen), and such residues occur in several of the deposits analysed in this study including ~~Laghy,~~ Great Orme and Breedon. However the Carboniferous-hosted deposits do not yield high methane values. Conversely, the gold-bearing deposits contain methane, but do not contain solid organic residues, although the host Neoproterozoic contains a large fossil oil reservoir in the same region (Bata & Parnell 2014).

These observations add weight to previous suggestions that methane plays a role in orogenic gold mineralization (Naden & Shepherd 1989; Gaboury 2013; Goldfarb & Groves 2015; Jébrak et al. 2018). The data could be viewed as showing that the most methanic of the sample set are a guide to gold-bearing deposits. The methodology therefore has potential in support of gold exploration.

Other studies

Data for gases are available for a limited number of gold-bearing deposits elsewhere. In particular gas data from minerals in Carlin-type gold deposits in Nevada have also shown them to contain significant methane (Blamey & Norman 2000; Blamey et al. 2017). The data from Britain and Ireland are, in the case of opaque sulphides, from minerals which do not yield conventional fluid inclusion microthermometric data, and so extend the range of minerals that can be investigated for direct measurement of fluid chemistry.

Previous measurements of hydrocarbon gases extracted from limestones (Carter & Cazalet 1984; Ferguson 1988; Mulshaw 1996) in the Carboniferous of Ireland and England show that anomalous concentrations of gases may be detected in the vicinity of sulphide ore deposits, but that observation is not matched by anomalies in the sulphides. The anomalies in the limestones may reflect hydrocarbon generation due to thermal alteration of the host rock around the hydrothermal systems. In contrast, the previously published fluid inclusion data for the greenschist-hosted deposits show that carbon-bearing species were components of the mineralizing fluid.

Conclusions

The measurement of gases released by the crushing of chalcopyrite samples from Britain and Ireland, at ambient temperature, shows that the resultant data may contain evidence for the nature of the mineralizing fluids. In particular:

(i) The samples not heated to greenschist facies almost exclusively yield water.
(ii) The greenschist-hosted samples yield non-aqueous species, complementing observations of CO_2 in fluid inclusions in accompanying minerals.
(iii) The Neoproterozoic samples, associated with gold mineralization, consistently contain anomalous quantities of methane.
(iv) The data from opaque sulphides extend the range of phases that can be used to characterize the mineralizing fluid.

These data add to previous studies of gas content in minerals from ore deposits, which have been used for example to infer models for MVT deposits (Blamey 2012) and unconformity-related uranium deposits (Rabiei et al. 2017), and the role of magmatic fluids (Blamey et al. 2017). Such measurements contribute to an understanding of the origin of ore deposits, and thus can be valuable in discriminating potential ore prospects during exploration.

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Figure captions

Fig. 1. Distribution of chalcopyrite samples in Britain and Ireland analysed for entrapped gases. Sample localities: AL, Allihies; BC, Ballycummisk; BD, Breedon, BG, Ballygrant; BR, Brennand; CB, Caldbeck; CL, Conlig; CN, Cononish; CT, Cloontoo; CT, Curraghinalt; GD, Gortdrum; GO, Great Orme's Head; ~~LY, Laghy;~~ NH, Nenthead; ~~NV, Navan;~~ SC, Stronchullin; TY, Tynagh; WH, Wanlockhead.

Fig. 2. Paragenetic sequences for representative deposits of each type, at Conlig (MVT), Curraghinalt (Neoproterozoic) and Ballycummisk (Munster Basin). Data modified from Moles & Nawaz (1996), Parnell et al. (2000) and Wen et al. (1999).

Fig. 3. Photomicrographs of selected samples. A, Chalcopyrite (bright) in quartz, Cononish; B, Chalcopyrite (bright) and specular haematite (grey) in quartz, Ballycummisk; C, Chalcopyrite (bright) in dolomite, Tynagh.

Fig. ~~2~~4. Cross-plot of non-aqueous gas content and methane content for sulphide samples. Two sets of greenschist samples are distinguished by high and low methane contents.

Fig. ~~3~~5. Chemistry of successive incremental crushes of a single sample of chalcopyrite from Cononish. Similarity of increments shows that the chemistry reflects a single assemblage of fluid inclusions.

Table 1. Compositional data for gases released from sulphide samples, Britain and Ireland

Lab no.	Locality	Mineral	Type	H_2O (%)	non-H_2O (%)	CH_4(%)	CO_2 (%)	Total mols	%CH_4 (non-H_2O)	%CO_2 (non-H_2O)
296	Cononish	Chalcopyrite in quartz	Neoproterozoic psammite	98.36	1.64	0.68	0.97	2.52E-09	0.41	0.59
294	Cononish	Chalcopyrite in quartz	Neoproterozoic psammite	99.44	0.56	0.22	0.34	3.49E-09	0.39	0.61
320	Cononish	Chalcopyrite in quartz	Neoproterozoic psammite	87.43	12.57	5.09	7.46	4.20E-09	0.40	0.59
304	Stronchullin	Chalcopyrite in quartz	Neoproterozoic psammite	95.68	4.32	0.07	4.18	1.15E-10	0.02	0.97
305	Stronchullin	Chalcopyrite in quartz	Neoproterozoic psammite	93.78	6.22	0.1	6.1	9.44E-10	0.02	0.98
315	Curraghinalt	Chalcopyrite in quartz	Neoproterozoic psammite	55.9	44.1	10.44	33.54	1.53E-09	0.24	0.76
316	Curraghinalt	Chalcopyrite in quartz	Neoproterozoic psammite	65.93	34.07	8.16	25.79	4.87E-09	0.24	0.76
314	Ballygrant	Chalcopyrite in dolomite	MVT in Neoproterozoic Lst	99.73	0.27	0.02	0.13	1.81E-11	0.07	0.48
302	Great Ormes Head	Chalcopyrite in dolomite	MVT in Carboniferous Lst	99.91	0.09	0.004	0.016	1.80E-11	0.04	0.18
303	Brennand	Chalcopyrite in calcite	MVT in Carboniferous Sst	99.92	0.08	0.003	0.04	4.71E-12	0.04	0.50
309	Langness	Chalcopyrite in calcite	MVT in Carboniferous Sst	99.43	0.57	0.03	0.5	2.44E-10	0.05	0.88
321	Nenthead	Chalcopyrite in siderite-ankerite	MVT in Carboniferous Lst	99.7	0.3	0.06	0.03	4.18E-11	0.20	0.10
323	Breedon	Chalcopyrite in dolomite	MVT in Carboniferous Lst	99.87	0.13	0.01	0.08	1.61E-11	0.08	0.62
~~9799~~	~~Laghy~~	~~Sphalerite~~	~~MVT~~	~~99.66~~	~~0.34~~	~~0.23~~	~~0.08~~	~~1.95E-09~~	~~0.68~~	~~0.24~~

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306	Leadhills	Chalcopyrite in dolomite-calcite	MVT in Ordovician pelite	98.35	1.65	0.02	1.58	1.75E-10	0.01	0.96
307	Leadhills	Chalcopyrite in dolomite-calcite	MVT in Ordovician pelite	99.95	0.05	0.01	0.01	7.09E-12	0.20	0.20
312	Caldbeck	Chalcopyrite in dolomite	MVT in Ordovician volcanics	99.96	0.04	0.01	0.01	2.38E-11	0.25	0.25
318	Conlig	Chalcopyrite in dolomite-barite	MVT in Silurian pelites	99.27	0.73	0.01	0.22	2.89E-10	0.01	0.30
319	Conlig	Chalcopyrite in dolomite-barite	MVT in Silurian pelites	99.51	0.49	0.014	0.2	3.95E-10	0.03	0.41
~~327~~	~~Navan~~	~~Galena~~	~~MVT~~	~~98.45~~	~~1.55~~	~~0.47~~	~~0.9~~	~~6.39E-10~~	~~0.30~~	~~0.58~~
300	Tynagh	Chalcopyrite in dolomite	MVT in Carboniferous Lst	99.05	0.95	0.01	0.86	1.61E-09	0.01	0.91
301	Tynagh	Chalcopyrite in dolomite	MVT in Carboniferous Lst	99.26	0.74	0.01	0.65	1.35E-09	0.01	0.88
293	Gortdrum	Chalcopyrite in dolomite-quartz	MVT in Carboniferous Lst	99.9	0.1	0.001	0.04	3.68E-10	0.01	0.40
					~~100~~				~~0.00~~	~~0.00~~
297	Allihies	Chalcopyrite in quartz	Munster Basin Devonian Sst	86.86	13.14	0.01	12.64	2.79E-09	0.00	0.96
325	Allihies	Chalcopyrite in quartz	Munster Basin Devonian Sst	98.59	1.41	0.002	0.43	6.76E-12	0.00	0.30
299	Ballycummisk	Chalcopyrite in quartz-dolomite	Munster Basin Devonian Sst	89.93	10.07	0.004	6.26	3.06E-10	0.00	0.62
311	Ballycummisk	Chalcopyrite in quartz-dolomite	Munster Basin Devonian Sst	94.57	5.43	0.0002	0.88	3.12E-11	0.00	0.16
317	Cloontoo	Chalcopyrite in quartz	Munster Basin Devonian Sst	95.51	4.49	0.06	4.18	4.74E-11	0.01	0.93