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THE USE OF FLUID INCLUSION DECREPITOMETRY TO DISTINGUISH MINERALIZED AND BARREN QUARTZ VEINS IN THE ABERFOYLE TIN-TUNGSTEN MINE AREA, TASMANIA

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ABSTRACT

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In the Aberfoyle Sn/W district of N.E. Tasmania, mineralization is in quartz veins associated with Devonian granite. The host rocks to the mineralization are folded Silurian quartzites, greywackes and shales and these also contain abundant pre-mineralization quartz veins which can be difficult to distinguish from irregularly mineralized ore veins on geological criteria, especially in drill core. It was found that the decrepitation characteristics of the quartz, chiefly the intensity ratio of high and low temperature peaks, which are developed in all decrepigrams, enable a distinction between the two generations of veins to be readily made. The differences between the fluid inclusions in the two generations of veins are relatively subtle, however it seems clear that "CO_2-rich" inclusions having a wide range of composition and density are the main source of decrepitation events and that the major differences in decrepitation behaviour can be correlated with differences in average homogenization temperature of these inclusions. Even those ore veins which have undergone moderate ductile deformation have the typical signature of their origin. The decrepitation results are supported by analyses of inclusion gases by Raman microprobe. These analyses differentiate a third group of veins which are possibly unmineralized veins belonging to a separate hydrothermal system.

INTRODUCTION

Decrepitometry is a method of characterizing mineral samples by sounds produced by leaking or bursting of fluid inclusions during heating. It was introduced by H.S. Scott in 1948, and the instrumentation was refined by Peach (1949) and Smith and Peach (1949). The decrepitometer output or "decrepigram" is a histogram of the number of decrepitation events versus temperature.

Initial interest in the technique originated in the claim that it could be used to determine the homogenization temperatures of generations of primary and secondary inclusions in minerals. This claim was challenged on a number of theoretical and experimental grounds (Kennedy, 1950; Stephenson, 1952). Despite vigorous rebuttal of these criticisms (Smith et al., 1950; Smith and Little, 1953) the technique rapidly fell into disuse in the West, and no program of applications research has been reported for more than 25 years. In the USSR, however, decrepitometry continues to be a widely used technique of fluid inclusion research and this work has recently been reviewed by Roedder (1977).

At the present time no adequate theory is available for the method and it is probably best suited to screening applications preceding more intensive microscopic studies, or used as a semi-empirical tool for certain mineral exploration applications. One of these, which we consider in this paper, is to use decrepitometry to distinguish quartz veins of different generations. In particular we deal with the problem of distinguishing the locally barren parts of a mineralized hydrothermal vein system from pre-mineralization quartz veins within a geographically restricted area. The aim of the study was to determine those factors resulting in critical differences between decrepigrams of veins of different generations in the Aberfoyle Sn/W district, so that the likely usefulness of the technique in other areas and in other applications can be better evaluated.

THE ABERFOYLE Sn-W DISTRICT VEINS

Mineralization at the Aberfoyle mine in northeastern Tasmania (Blissett, 1959; Kingsbury, 1965) occurs in quartz veins within tightly folded Silurian quartzite, greywacke and shale, immediately adjoining a Devonian granitoid intrusion and rarely extending into it (Fig. 1). The ore veins often have a selvedge of muscovite which is a useful indicator of vein type when it is present. Cassiterite and wolframite are often associated with the muscovite on the margins of the ore veins. There are a number of minor mineral components of these veins and their paragenesis has been determined by Edwards and Lyon (1957).

Barren quartz veins of the same or related hydrothermal systems are found within the mine and the surrounding area and barren pre-mineralization quartz veins are extremely common. Whereas the pre-mineralization veins are invariably strongly deformed and partly recrystallized, the hydrothermal veins are typically little deformed, but they may show evidence of ductile deformation to a degree which makes them difficult to distinguish from the pre-mineralization veins even in thin section.

PROCEDURE

Quartz samples from seven barren and ten ore veins from the Aberfoyle mine and surrounding area (Fig. 2) were characterized by decrepitation,

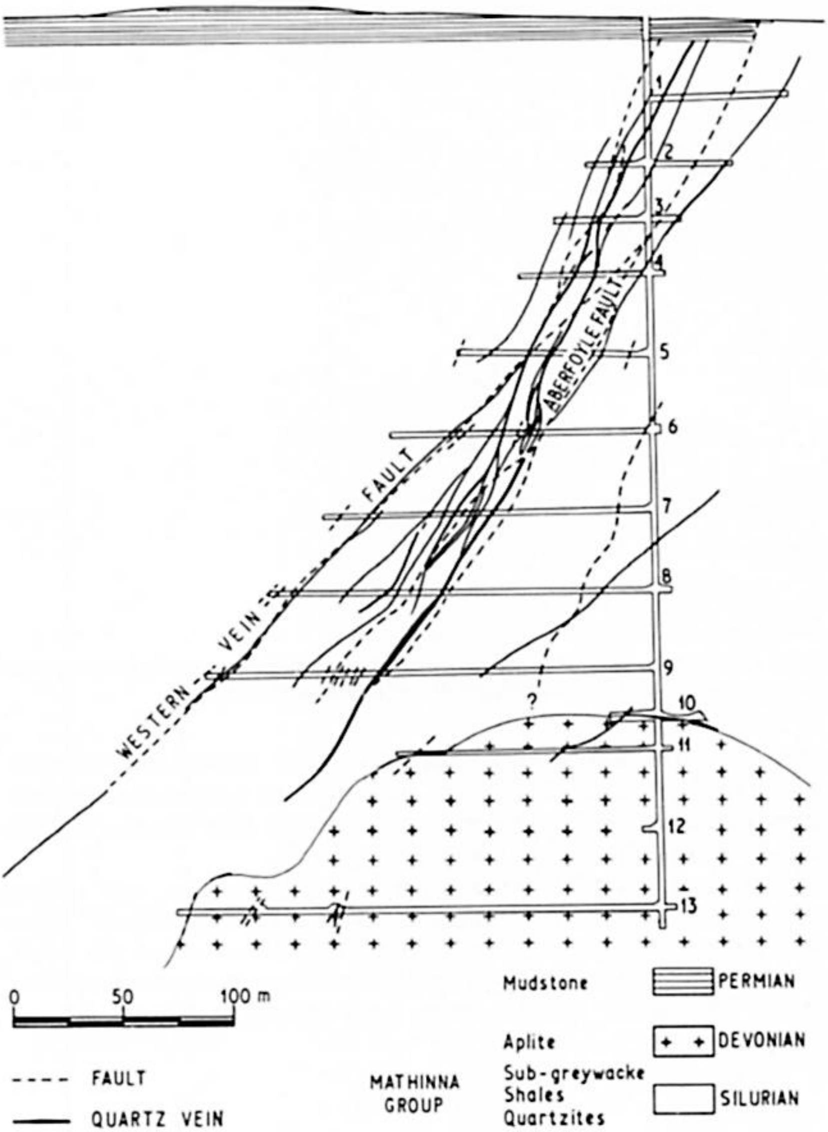

Fig. 1. Cross-section of the Aberfoyle mine looking north, showing the relationship of faults and veins to the aplitic cupola. After Kingsbury, 1965.

using a newly constructed microprocessor-controlled apparatus of the basic type described by Peach (1949), in which a crushed mineral sample is heated at a constant rate with decrepitation events sensed by a microphone and recorded as a histogram of counts versus temperature. One gram samples of −30 to +40 mesh (>420 to <600 μm diameter) crushed quartz were used for each determination. The programmed heating rate was 20°C/min over a range 100—550°C.

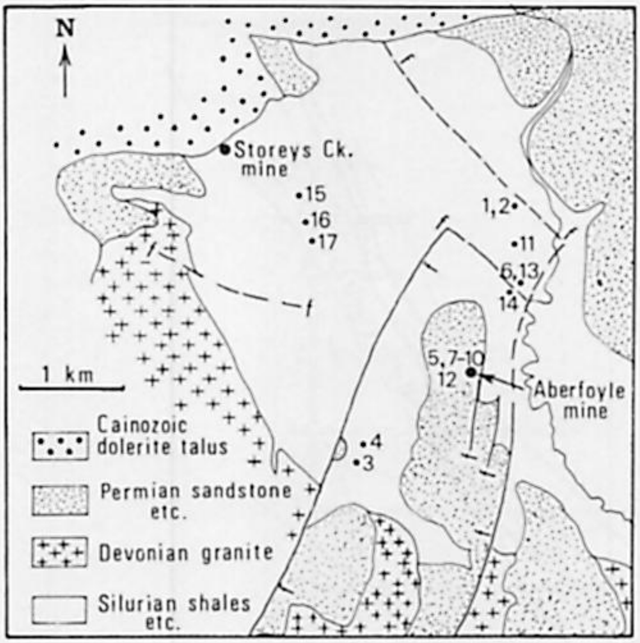

Fig. 2. Geological sketch map of the Aberfoyle-Storeys Creek district, showing locations of quartz veins used for the decrepitometric study. After Blissett, 1959.

Each sample was also examined for evidence of deformation and recrystallization in polished thick and thin section. Estimates of inclusion size distribution and inclusion abundance were made on representative samples following the procedure outlined in Chivas and Wilkins (1977).

Heating and freezing stage examination of selected samples was carried out using a Chaixmeca apparatus. Compositions of gases in individual inclusions were determined by Raman microprobe (Dhamelincourt et al., 1979) and compared with mass spectrometric examination of gases released from crushed samples. The latter results, being only semiquantitative, are not given in this paper.

RESULTS

Decrepitometry

To compare decrepigrams we use as simple parameters total counts/gram, numbers of peaks and their temperatures, and peak heights and ratios. The peak height is the number of counts in the 10°C interval with maximum count rate. The data are listed in Table I and a selection of the decrepigrams is shown in Fig. 3.

The ore veins typically show distinctly bimodal decrepigrams with peaks at 380–440°C and 520–535°C, a high proportion of the decrepitations occurring in the lower temperature peak. The barren veins also give bimodal histograms, but the peaks overlap to produce a shoulder at 440—460°C which leads onto an intense peak at 520—540°C. Whereas the ratio of heights of higher and lower temperature peaks is <2.7 for the ore veins, this ratio is >2.7 for the pre-mineralization barren veins (Fig. 4). However certain mildly deformed but apparently unmineralized veins from the Eastern Hill prospect also plot in the ore field.

Inclusion types and composition

Heating and freezing stage investigations reveal rather subtle differences between the fluid inclusions of the hydrothermal ore vein quartz and the barren pre-mineralization quartz (Wilkins and Ewald, in prep.). Inclusions in the ore vein quartz are of two main types:

(a) "CO_2-rich" — inclusions with highly variable H_2O and CO_2 contents in which liquid or solid CO_2 can be induced to form by freezing. Calculations based on volume and gas phase density measurements, together with the assumption that inclusions contain only H_2O and CO_2, show that the apparent compositional range is 3—72 mole % CO_2. The gas phase has a highly variable density at ambient temperature. Most inclusions of this type homogenize above 300°C and both liquid and gas phase homogenizations occur.

(b) Aqueous inclusions with a vapour phase of low density. Homogenization temperatures are usually relatively low, 100—200°C, but they can be as high as 375°C and homogenization usually occurs into the liquid phase.

A similar assemblage of inclusions occurs in the pre-mineralization vein quartz. Inclusions almost filled with a CO_2-rich gas are common, however in contrast to the ore vein quartz, liquid CO_2 at ambient temperature (21°C) is rarely seen. In all samples of both hydrothermal vein quartz and pre-mineralization vein quartz, significant necking-down of the inclusions has widened the apparent range of fluid composition and increased the range of homogenization temperatures.

The gases of 18 "CO_2-rich" inclusions in quartz of both hydrothermal and pre-mineralization origin were analyzed by laser Raman microprobe (Dhamelincourt et al., 1979). The results are plotted in Fig. 5. In all inclusions the gas phase is dominantly composed of CO_2, with all of the inclusions from hydrothermal ore vein quartz having >93 mole % of this component. Minor amounts of N_2, CH_4 and H_2S are present in the inclusions in hydrothermal quartz with greater amounts of these components, predominantly nitrogen and methane, in the pre-mineralization veins. This explains the observation above that liquid CO_2 visible at ambient temperature is rare in the pre-mineralization vein inclusions, because nitrogen and methane lower the liquid-vapour homogenization temperature of CO_2 (Hollister and Burrass, 1976; Ghilaumou et al., 1981). It should also be noted that the two severely deformed pre-mineralization veins located within a metre of a large ore vein contain gases which are particularly CO_2-rich, possibly due to infiltration of

TABLE I

Microscopic observations and experimental data on vein quartz from the Aberfoyle district

Location	Decrepigram shown in Fig. 3	Decrepitation		
		Total activity (counts/g)	Peak (°C)	Peak heights (counts)
Pre-mineralization veins				
1. # 13 level, Lutwyche development, 1 m from Battery ore vein		11,356	440sh*/520	460/1642
2. Duplicate sample same location as 1.	g	15,151	450sh/530	540/2151
3. Brock's prospect, Rossarden, outcrop	i	25,510 26,220	450sh/530 460sh/530	1050/3732 1282/3265
4. McDonalds prospect, Rossarden, outcrop	h	27,921	460sh/540	1309/3708
Ore veins				
5. # 13 level, Aberfoyle mine, contact vein	e	1,774	380/535	167/56
6. Surface outcrop of ?Battery ore vein		4,289 5,130	400/535 400/525	181/315 221/351
7. # 6 level, Aberfoyle	d	9,452	390/530	453/803
8. # 4 level, Aberfoyle	c	10,506	430/530	463/966
9. # 7 level, Aberfoyle		10,734	420/530	587/947
10. # 3 level, Aberfoyle		10,869	430/530	446/1177
11. Surface ore vein, Lutwyche area		14,019	430/535	629/1031
12. # 1 level, Aberfoyle	b	18,836	440/520	1006/1562
13. Surface ore vein, Battery area		24,164	420/525	1457/1607
14. Surface, Kookaburra prospect	a	35,583	410/520	1954/1449
Eastern Hill veins				
15. Unmineralized veins		9,633	420/540	487/733
16. Unmineralized veins		10,835	420/525	453/869
17. Unmineralized veins	f	16,894	420/530	732/1648

*sh shoulder, not a well-defined peak. ** — not determined.

Abundance (visible inclusions/g)	Gas analyses (mole %)				Notes on deformation
	CO_2	N_2	CH_4	H_2S	
—**	90.3	3.9	5.8	<0.1	Severely deformed, extensively recrystallized
4.2×10^8	95.1	2.8	2.1	<0.1	Severely deformed, extensively recrystallized
3.6×10^8	83.6	9.7	6.3	0.4	Severely deformed, partly recrystallized
4.3×10^8	73.4	25.1	1.4	<0.1	Severely deformed, incipient recrystallization
4.8×10^8	—	—	—	—	Undulose extinction, deformation bands, incipient recrystallization
8.0×10^8	95.2 97.7 97.0	3.0 1.2 1.5	1.4 0.8 1.5	0.4 0.3 <0.1	Undulose extinction, deformation bands, subgrains, incipient recrystallization
—	—	—	—	—	Undeformed
—	—	—	—	—	Undulose extinction, deformation bands
—	—	—	—	—	Deformation bands, incipient recrystallization
—	—	—	—	—	Deformation bands
—	96.6	2.5	0.9	<0.1	Undulose extinction, subgrains, some recrystallization
2.0×10^8	93.9 94.2 94.1	1.7 1.9 2.2	4.4 3.2 3.0	<0.1 0.6 0.6	Undulose extinction, subgrains
—	—	—	—	—	Undulose extinction, deformation bands
—	97.7	1.1	1.1	0.1	Minor deformation bands
—	87.6	3.0	9.3	<0.1	Deformation bands
—	92.6	1.7	5.6	0.1	Deformation bands, subgrains, minor recrystallization
—	89.6	1.6	8.5	0.3	Deformation bands, subgrains

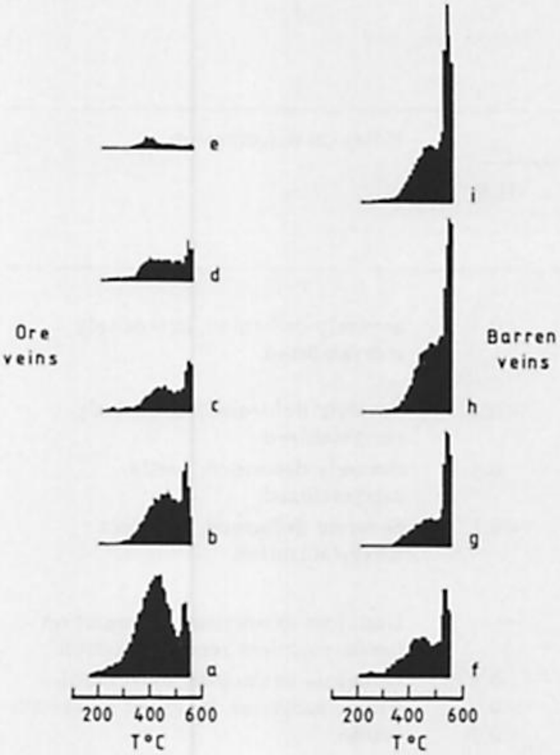

Fig. 3. Decrepigrams of quartz from Aberfoyle mine ore veins (a—e), an Eastern Hill vein (f), and pre-mineralization veins (g—i). See Table I for details.

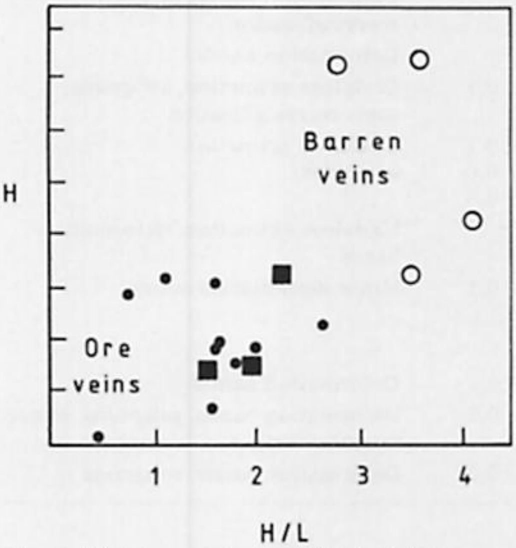

Fig. 4. High-temperature peak height (*H*) versus the ratio of high to low-temperature peak heights (*H/L*), in decrepigrams of vein quartz samples from the Aberfoyle district. Ore veins •, pre-mineralization barren veins ○ and Eastern Hill veins ▪.

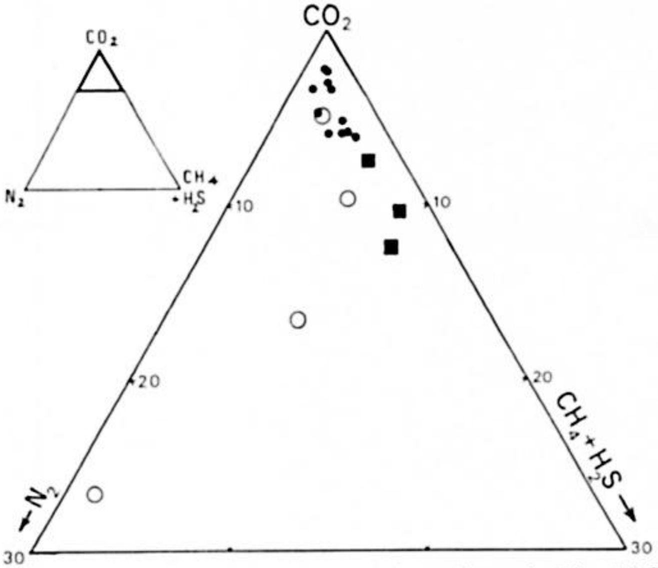

Fig. 5. Raman microprobe gas analyses (in mole %) of "CO_2-rich" fluid inclusions in Aberfoyle district quartz veins. Legend as in Fig. 4.

fluids from this source. It is also significant that, regardless of the degree of deformation of the ore veins, the gas analyses still fall within the restricted field of this group. Gases of the mildly deformed Eastern Hill hydrothermal veins are distinctly richer in CH_4 and they are distinguished by having a CH_4/N_2 ratio >3.

Heating stage observations

As an aid in interpreting the decrepigrams, the homogenization temperatures of inclusions in a representative hydrothermal ore vein were compared with those of a pre-mineralization vein. The histograms (Fig. 6) are both bimodal with peaks at 100–200°C and 300–400°C. It is important to note that the bimodality of the decrepigrams cannot be the result of successive decrepitation of assemblages of secondary and primary inclusions as most inclusions in the hydrothermal ore vein quartz are associated with healed fractures and are clearly secondary in origin. For the deformed and recrystallized quartz of the pre-mineralization veins, the distinction between primary and secondary inclusions is meaningless.

At first sight the two peaks of the Th histograms (Fig. 6) would appear to correlate with the two peaks on the decrepigrams, allowing for the expected displacement of decrepitation peaks towards higher temperatures. For quartz this displacement occurs because the internal pressure usually only becomes sufficient to cause failure of the cavity walls some tens of degrees above the homogenization temperature, depending on the size of the inclusions (Leroy, 1979).

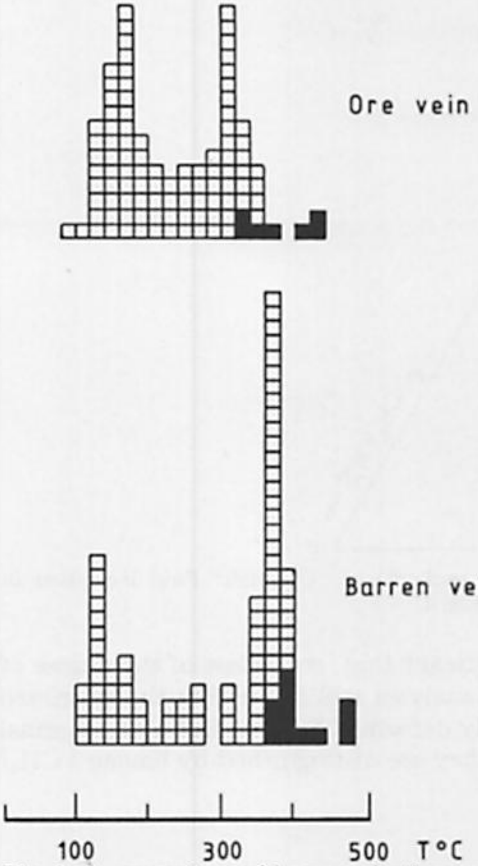

Fig. 6. A comparison of homogenization temperature distributions for fluid inclusions in a representative ore vein (sample 12) and a representative pre-mineralization vein (sample 4). Filled squares indicate homogenization into gas phase.

However, in a separate set of tests in which 0.5 mm polished plates were examined at 50°C intervals to 550°C it was observed that all inclusions homogenizing in the 100—200°C range (mainly aqueous inclusions) decrepitate by 250°C, before the decrepitometer records any significant response from these materials. The decrepitation peak at 380—440°C for the hydrothermal vein quartz correlates with the presence of CO_2-rich inclusions which homogenize into the liquid phase. These begin to decrepitate at approximately 300°C and are entirely destroyed by 500°C, leaving only those CO_2-rich inclusions which homogenize into the gas phase and any aqueous inclusions which homogenize at high temperature, to contribute to the higher temperature decrepitation peak. By 500°C all inclusions >3 μm diameter in the hydrothermal vein quartz are destroyed and only inclusions below this size can contribute to the high temperature decrepitation peak in this material.

Examination of polished plates of pre-mineralization vein quartz, treated similarly, shows that "CO_2-rich" inclusions which homogenize into the liquid phase do so at temperatures 50°C higher than those in the hydrothermal vein quartz. These inclusions give rise to the intense decrepitation peak at 520–540°C. At 550°C about 20% of the inclusions >5 μm diameter remained intact. The origin of the 440–460°C shoulder in the pre-mineralization vein quartz decrepigrams is not clear.

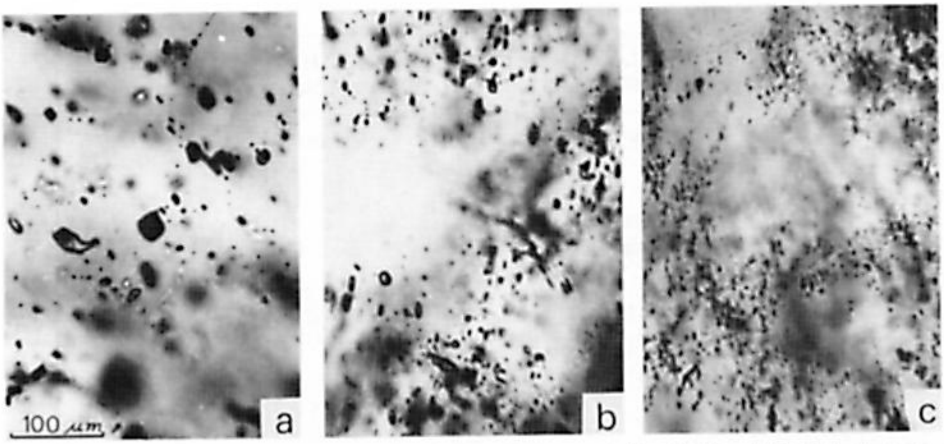

Fig. 7. A comparison of micrographs showing representative fields of fluid inclusions at the same magnification in quartz veins having high (a, sample 12), medium (b, sample 8) and low (c, sample 5) decrepimetric activities. See Table I.

Inclusion abundance and size distribution

Counts of total visible inclusions per unit volume on six samples (Table I) show that there is poor correlation ($r = -0.56$) between inclusion abundance and decrepimetric activity. Due to the difficulty of measuring the abundance, the figures given in Table I may be overestimated by as much as an order of magnitude. Nevertheless, calculations indicate that only about one in 10^4–10^5 visible inclusions gives rise to an event which is recorded by the decrepitometer at the instrument settings used. This is to be expected and in part reflects the fact that a substantial proportion of the very small inclusions does not decrepitate in the experimental temperature range (cf. Leroy, 1979).

The important relationship between the size distribution of inclusions in a sample and the intensity of its decrepimetric activity is illustrated in the set of photomicrographs (Fig. 7), taken under moderate magnification, which show inclusions in quartz of high, medium and low decrepimetric activity. A random selection of 500 inclusions in the high and low activity samples were measured to give the size distributions in Table II. Although these data suggest that the observed level of decrepimetric activity can be substantially accounted for by decrepitation of the larger inclusions, our previous observations on heated polished sections also indicate that in some cases small inclusions can contribute to the decrepigram by virtue of the very high pressures they develop (Leroy, 1979).

TABLE II

Inclusion size distribution of samples with low and high decrepitation activities

Inclusion diameter (μm)	Low decrepitation activity. Sample 5 (%)	High decrepitation activity. Sample 12 (%)
<3	93.9	84.8
3—6	5.4	9.9
6—9	0.6	4.1
9—12	rare	1.0
>12	rare	0.1
Visually estimated total inclusions/g	4.8×10^{8}	2.0×10^{8}

DISCUSSION

Most quartz veins contain inclusions which homogenize over a wide range of temperature, either because fluid has been trapped as primary or secondary inclusions over a large temperature range, or because the original inclusions have been modified by subsequent deformation or by necking-down during cooling. The Aberfoyle inclusion assemblages are complex for all of these reasons. For such material the concept of a particular decrepitation temperature (Td), defined as the temperature of onset of massive decrepitation of the inclusions (Scott, 1948), is without much meaning, therefore the graphical determination of this quantity has not been attempted.

With such material it is not easy to determine the reasons for the observed differences between decrepigrams. The controls on the intensity of decrepitation (counts/gram) seem to be, primarily, inclusion size and abundance. Total decrepitation counts is not a useful classificatory parameter in the Aberfoyle area as both the lowest (sample 5) and the highest (sample 14) measured decrepitation intensities were obtained from hydrothermal ore veins.

A priori, one would expect ductile deformation and especially recrystallization of a vein to result in a general decrease in intensity of decrepitation, because there is a tendency for inclusions to be eliminated during these processes (Boyle, 1954; Wilkins and Barkas, 1978). Although this holds within veins of one generation and particularly for the hydrothermal ore veins, it does not hold for comparisons between veins of different generations at Aberfoyle. It is of particular interest that although the inclusion population is modified during ductile deformation and the absolute intensity of decrepitation greatly decreased, the positions and relative intensities of the decrepitation peaks maintain their original character. The decrepigrams do not simply differentiate between relatively deformed and undeformed suites of veins. Similarly, the integrity of the gas phase composition of the inclusions seems to be maintained during deformation, although clearly the possibility that later fluids are superimposed during fracture healing in a zone of later hydrothermal activity should be borne in mind.

The number of peaks and their positions are controlled by subtle differences in inclusion size distributions as well as fluid composition and density of the different generations of secondary inclusions. The relative intensities of the peaks depend upon the relative abundances and sizes of inclusions belonging to the different generations. These factors are all extremely difficult to evaluate. The remarkable fact is that inclusion assemblages having rather subtle differences in homogenization temperature distribution and fluid composition lead to consistently different decrepigrams. This also shows that decrepigrams may provide a useful guide for selection of specimens for microthermometric studies.

Exploration significance

In the Aberfoyle area, distinction between the vein types, particularly in drill core, can be difficult using geological criteria. The approach to the problem through decrepitometry is based upon the supposition that the fluids trapped in the hydrothermal veins are similar, whether they are visibly mineralized or not, but that they differ from those trapped in the pre-mineralization veins of whatever origin and that they will, therefore, give rise to a different decrepimetric response.

First we consider the near miss situation where a drill core has intercepted an unmineralized portion of an irregularly mineralized ore vein. On the basis of our results the nature of the vein should be readily identified by the decrepigram. Similarly, a pre-mineralization vein should be readily identified, with the qualification that if it is in close proximity to a hydrothermal vein it may have collected some of the ore fluids and taken on some of the character of the ore veins.

Some barren veins may either belong to a different hydrothermal system or be spatially or temporally separated veins related to the mineralized hydrothermal system. In the latter case, if the temporal and spatial changes in fluid composition and temperature are small, the barren veins will give rise to decrepigrams which are similar to those from mineralized veins. However, a complete gradational sequence of decrepigrams can occur in response to gradual changes in the fluid composition and temperature.

The Eastern Hill veins seem to be of this type as their decrepigrams are intermediate between the hydrothermal ore veins and the pre-mineralization veins, although they plot within the ore vein field in Fig. 4. The Eastern Hill veins are also comparable to the ore veins in their mild degree of deformation. The chemistry of the inclusions is not known in detail, but they differ from the ore veins and the pre-mineralization veins in the higher CH_4/N_2 ratio of their gas phase. Ore veins have been recorded from the Eastern Hill area (Blissett, 1959), but the relationships of our samples to them are uncertain.

It should also be noted that, although the few gas analyses presented were done in support of the decrepimetric study, gas phase analysis of fluid inclusions shows promise of being a most useful tool to classify veins in its own right. However, a few supplementary gas analyses on inclusions in cassiterite and scheelite from the ore veins (R.W.T. Wilkins and A. Ewald, in prep.) show that the ore fluids had a wider range of gas composition than that indicated by the analyses of inclusions in quartz.

CONCLUSIONS

A number of general conclusions can be drawn from the Aberfoyle study. Decrepitometry is a simple technique well adapted to the routine examination of large numbers of samples in a survey operation. In the number, position, and absolute and relative intensity of peaks, the decrepigrams are responsive to a complex set of factors originating in characteristics of the fluid inclusions and the nature of the host mineral. Without concomitant microscopic examination of selected samples, the factors controlling the form of the decrepigrams will not be known. Nevertheless simple manipulation of parameters derived from the decrepigrams may lead to a classification of veins, on a local scale, which could be valuable in mineral exploration.

The method assumes that there will be differences in the nature of trapped fluids in different generations of veins. Where these differences are great, readily distinguishable decrepigrams will result. However, even if the differences are subtle, as is the case at Aberfoyle, the method appears to be sufficiently responsive to be usefully employed in the classification of veins.

For this purpose, graphical determination of temperatures of decrepitation (Td) is unnecessary, indeed in the common situation of quartz veins containing inclusions which have a range of fluid densities and compositions and which homogenize over a wide range of temperature, this quantity has little meaning.

Although it is not possible to infer that a vein is mineralized from its decrepigram, it seems to be possible to tell if a vein belongs to a generation of mineralized veins. Thus it may be particularly useful in the near-miss situation where drill core intersects irregularly mineralized quartz veins, particularly if other criteria are difficult to apply.

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