TWO STAGES OF DECREPITATION OF MICAS

W. W. HUTCHISON[1]

ABSTRACT

This investigation has revealed that biotite, muscovite, lepidolite, zinnwaldite and some phlogopites on being heated to 1,000 °C decrepitate in two distinct stages. The lower temperature stage has a sharp peak rate in the range of 305°–340° C for all the micas examined. This stage, which was never exhibited by chlorite, is related to the rapid loss of an extremely small amount of water from the micas. A more vigorous stage of decrepitation starts in the range 550°–750° C and rises to a peak in the range 600°–1,000° C. The temperature of the peak rate and the character of the decrepitation appear to be dependent, in part, on the species of the mica. From heating stage observations, weight loss measurements, chemical analyses and the measurements of the gas pressure over mica during heating, it appears that this stage is mainly caused by the partial loss of lattice water during heating.

In the course of a systematic study of metamorphic rocks and minerals, it was discovered that decrepitation of micas occurred within a broader temperature range than had been previously reported. Consequently an investigation of the decrepitation of the various micas was carried out and the results form the basis of this article.

PREVIOUS WORK

At least seven major causes of decrepitation have been proposed by Smith (1963). These are, fluid inclusions (D1), misfit of solid inclusions (D2), so-called anomalous effects (D3), inversions (D4), decompositions (D5), demetamicization or radiation damage recovery (D6) and melting (D7).

In the course of early reconnaissance work on decrepitation, Smith (1952 and 1953) attributed some decrepitation to the exfoliation of micaceous minerals. These results led Smith (1957) to conclude, "Decrepitation due to the thermal decomposition (D5) often occurs in igneous rocks which contain phyllosilicates such as muscovite, biotite, chlorite and serpentine and other hydrous minerals such as epidote and amphibole. Each has a characteristic temperature at which decomposition gives detectable sounds". No data have yet been published to indicate the precise temperature range in which decrepitation takes place for these various minerals.

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[1] Geological Survey of Canada, Vancouver. This paper is derived from part of the writer's Ph.D. thesis (1962, University of Toronto).

Decrepitation of biotite was recorded by Saha (1959). He detected two stages. The first started at 300° C. and rose rapidly to a peak at 450° C. and the second started at 580° C. and rose to a peak at 630° C. Because the available data on the differential thermal analysis (DTA) of biotites did not indicate any distinct thermal reaction during heating, Saha concluded that the biotite itself was not the cause of decrepitation. He noted 5 to 10 per cent chlorite in the biotite and knowing that chlorite did give rise to thermal reactions in the temperature range in which the decrepitation was recorded, he then came to the conclusion that the decrepitation was related to the decomposition of the included chlorite and not to the decomposition of the biotite.

A careful literature survey revealed no other systematic studies of decrepitation of phyllosilicates.

The aim in the present study was threefold: (*a*) to establish the characteristics of decrepitation of some micas, (*b*) to establish the cause(s) of the decrepitation, (*c*) to appraise the role of decrepitation of micas in geothermometric determinations.

MATERIALS

The phyllosilicates used were phlogopite, biotite, muscovite, lepidolite, zinnwaldite and chlorite. The nature and origin of the samples are listed in the Appendix. The samples were obtained either as cleavage flakes from mineral specimens or extracted from rock specimens.

EXPERIMENTAL TECHNIQUES

To determine the causes of decrepitation in micas, various heating experiments other than decrepitation were also carried out and are described below:

Decrepitation

The apparatus used in the present study was the sensitive electronic rate-recording device designed by Peach and Smith (Peach 1949 and Smith & Peach 1949). A detailed description of this equipment is contained in Smith (1963). Two different methods were used in recording decrepitation. In one, the rate of generation of pulses was fed to a recording milliammeter so that a continuous graph of the rate of decrepitation was obtained (decrepigraph). In the other method, the pulses were totalled by a mechanical counter at the end of every ten degrees rise in temperature. A histogram plot was then compiled of the number of 'pops' per ten degrees centigrade rise versus temperature (decrepigram). Generally the recording milliammeter was used for weak decrepitation and the recording counter for vigorous decrepitation.

The usual procedures in making runs were followed. The samples were first prepared by grinding, clipping with scissors, or grating the phyllosilicate against a file. The mica samples were then sieved. From 0.5 to 2.0 cc. of the $-42+80$ (Tyler) mesh fractions were placed in the sample holder. Heating was maintained at a nearly constant rate close to some rate between 15° to 30° C. per minute.

The correction for the difference in temperature between the sample and the thermocouple located in the wall of the furnace holder was measured directly. To do this the author employed two substances which emitted noise at specific temperatures.* The decrepitation recorded was that generated when quartz and silver nitrate rapidly inverted at 573° C. and 159° C. repectively. The inversion of quartz was found to take place at 572° C. with a standard deviation of ± 2° C. and the inversion of silver nitrate at 155.5° C. with a standard deviation of ±4° C. These results showed that the inherent errors in the difference in temperature between the sample and the thermocouple and in the calibration of the thermocouple were small and within the limits of experimental error.

Because of their importance in any interpretation, some of the factors affecting decrepitation are here mentioned briefly. The amplitude of the peak, for example, increases with the amount of sample but the position or temperature of the peak does not vary. With the decrease in grain size of a phyllosilicate, the amplitude or vigour of the decrepitation also decreases and the position of the peak is displaced to a lower temperature. This decrease in vigour is such that almost no distinct decrepitation can be detected from mica of grain size less than 100 (Tyler) mesh, (i.e. grain sizes commonly used in DTA). The rate of heating does not affect the amount of decrepitation but, if increased, brings about a displacement of the peak to a higher temperature. Because of this and because one predetermined, fixed rate of heating could not be used, all the temperatures of the peaks are reported along with their respective rates of heating. Variation of the settings of the pre-amplifier, amplifier or ratemeter affects the recording of the decrepitation and therefore all settings were kept constant throughout the present work. Generally the reproducibility of decrepitation of phyllosilicates was good. The temperature of the peak

*Only two minerals were found that emitted noise at specific temperatures, so that calibration was based on two determinations. Attempts by previous workers to locate the thermocouple inside the sample holder failed because noise was emitted during heating at points where the thermocouple wires were in contact with the sample holder. Consequently the thermocouple tip had to be seated in a hole drilled into the outside wall of the sample holder close to the location of the sample (see Smith, 1963).

rate of decrepitation could usually be reproduced to within ±10° C and, commonly to within ±5° C. Normally at least two runs were made on each sample if sufficient material was available.

Heating stage microscope

Mica samples to be mounted in the heating stage were carefully cleaved from a mother flake. Usually these flakes were of sufficiently high natural 'polish' that no further polishing was required. Calibration of the thermocouple was carried out by observing the temperatures of melting of tin, zinc, potassium chloride, sodium chloride and silver. The heating rate was maintained close to 15° C. per minute.

Weight loss measurements

The mica was prepared in the same way as for decrepitation and split into six portions, each of which, weighing from 0.75 to 1.75 g, was placed in a small porcelain crucible. Heating was at a rate comparable to that used in decrepitation, the temperature being measured by a thermocouple placed in a dummy crucible. When the required temperature was reached, the samples were held at that temperature for one minute to minimize temperature gradients within the furnace, and then cooled. When cold, each crucible was weighed and re-weighed until a constant weight was obtained.

Chemical analyses

Full details of the preparation of the micas for chemical analyses are contained in Hutchison (1962). Each mica sample was separated into 3 portions, one being heated to 110° C., another to 600° C. and the third to 800° C. in the same way as the samples were heated for weight loss measurements.

Measurements of the variation in gas pressure during heating of mica in a pre-evacuated chamber

The technique employed was similar to that used by Orcel (1927) for the examination of the loss of lattice water from chlorite. The mica was prepared in the same way as for decrepitation, and about 2 grams placed in a vycor test-tube to which a mercury manometer was attached. The internal pressure of the apparatus was brought down to 1 mm of mercury. The test-tube was lowered vertically into a furnace and heated at a rate close to 15° C. per minute. The pressure was read every 20° C. Satisfactory calibration between the temperature indicated by the thermocouple outside the test-tube and the actual temperature of the sample was not obtained. Above 800° C. the indicated temperatures may be in error as much as ±25° C.

EXPERIMENTAL RESULTS

Decrepitation

One of the first discoveries in this study was the detection of two stages of decrepitation in micas. The low temperatute stage was determined to occur in the range 300°–340° C. and the high temperature stage in the range 550°–1,000° C. The decrepitation of each stage was examined separately.

Low temperature stage. The character of the low temperature stage is shown by traces A, B, C, and E of Figure 1. The decrepitation started close to 300° C., rose to a well-defined peak rate in the range 305°–340° C. and was followed by a marked decrease in rate.

In a systematic study of the decrepitation of biotite 113, the temperature of the peak rate of decrepitation was found to bear an apparent linear relationship to the rate of heating (Figure 2). The best fitting line was determined through the use of a linear regression equation. The standard estimate of error was found to be ±2.5° C. Because of this dependence of the temperature of the peak on the rate of heating, the decrepitation peaks of the micas have been reported along with the rate of heating used (Table 1 and Figure 2). All these micas exhibited the low temperature stage of decrepitation. Using the correction for the rate of heating determined for biotite 113 (Figure 2) and assuming that it is the same for all micas, the estimated mean of the decrepitation peaks would be approximately 318° C. at a rate of heating of 25° C. per minute.

TABLE 1. LOW TEMPERATURE STAGE OF DECREPITATION.
Temperature of peak rates of decrepitation of various micas.
B-Biotite, Ms-Muscovite, L-Lepidolite, Z-Zinnwaldite and P-Phlogopite.

Specimen # and type	Temp. of Peak °C	Rate of Heating °C/Min.	Specimen # and type	Temp. of Peak °C	Rate of Heating °C/Min.
14_1B	318	24	119_1P	312	28
14_2B	324	26	119_2P	310	25
43_1B	318	25	120_1P	306	26
43_2B	315	22	135_1Ms	312	26
110_1B	319	25	135_2Ms	319	26
110_2B	324	31	205_1P	324	20
111_1Ms	318	28	207_1P	319	18.5
111_2Ms	309	16.5	207_2P	318	22
112_1Z	330	31	215_1B	322	22
112_2Z	327	24	231_1B	316	20
114_1L	335	31	233_1B	324	20
114_2L	326	26	301_1L	320	21
115_1Ms	319	28	302_1L	316	21
115_2Ms	320	29	302_2L	314	20
117_1Ms	316	26			
117_2Ms	309	24			

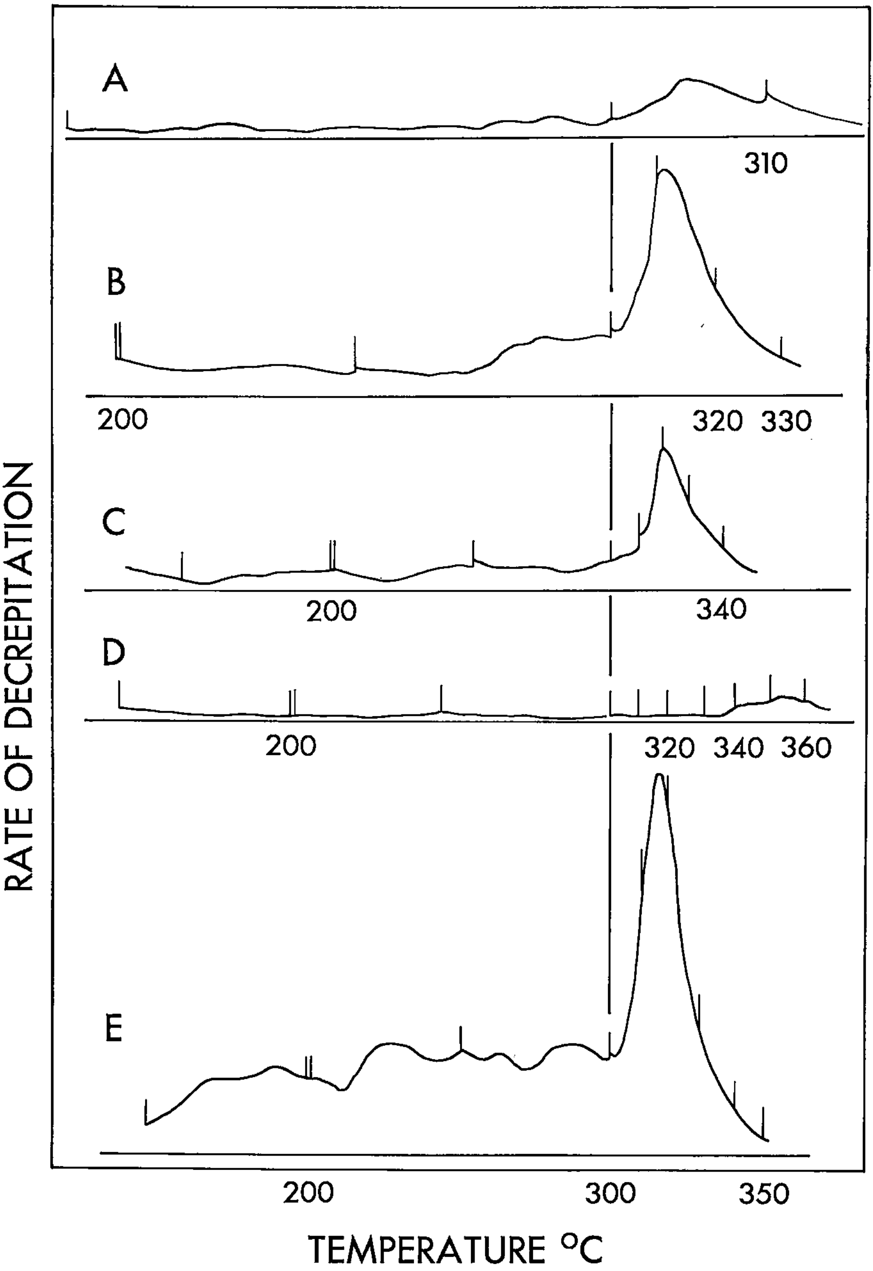

FIG. 1. Decrepigraphs of the low temperature stage of decrepitation in biotite 113. A—Normal. Heating rate 5.5° C/minute. B—Normal. Heating rate 16° C/minute. C—Normal. Heating rate 33° C/minute. D—Re-run of a sample previously heated to 350° C. E—Re-run of sample previously heated above 350° C (sample used in D) and then soaked in water. Heating rate 29.5° C/minute.

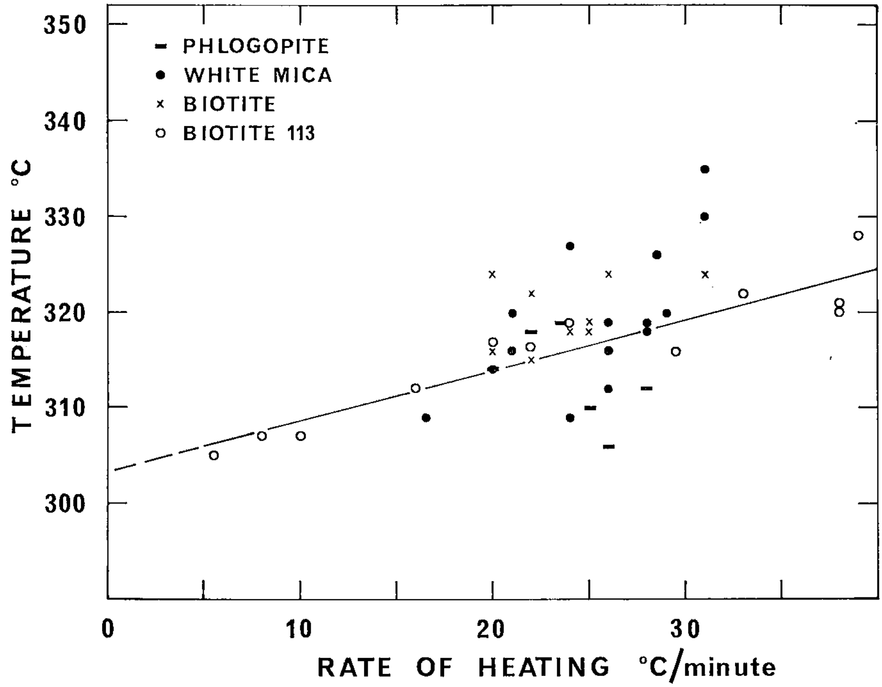

Fig. 2. Low temperature stage at decrepitation. A plot of the temperature of the peak rate against rate of heating for various micas, from data listed in Table 1. White mica comprises muscovite, lepidolite, and the one zinnwaldite sample. The open circles indicate the effect of variation in rate of heating on the temperature of the peak rate of biotite 113. The best line of fit was determined by linear regression.

Chlorite did not exhibit this low temperature stage of decrepitation. Neither did talc, pyrophyllite or vermiculite (Hutchison, 1962). Decrepitation between 300° C. and 340° C. was never detected on reheating a mica charge (Figure 1) even if it had been left in the room atmosphere for a few days. However, decrepitation did recur if the sample, pre-heated to 350° C and later soaked in tap or distilled water, was reheated. If the sample was not air-dried before reheating in the decrepitation apparatus, then a high background was obtained prior to the peak itself (Figure 1). When a sample was preheated in air or vacuum at 250° C for 24 hours, the low temperature stage of decrepitation appeared as usual on heating above 300° C. Decrepitation was never detected during the cooling cycle of a run.

Muscovite, which had been deliberately pitted by hammering, gave rise to a lower amplitude peak than normal but when this mica was hammered under water and then heated, the decrepitation subsequently obtained was much more intense (Figure 3).

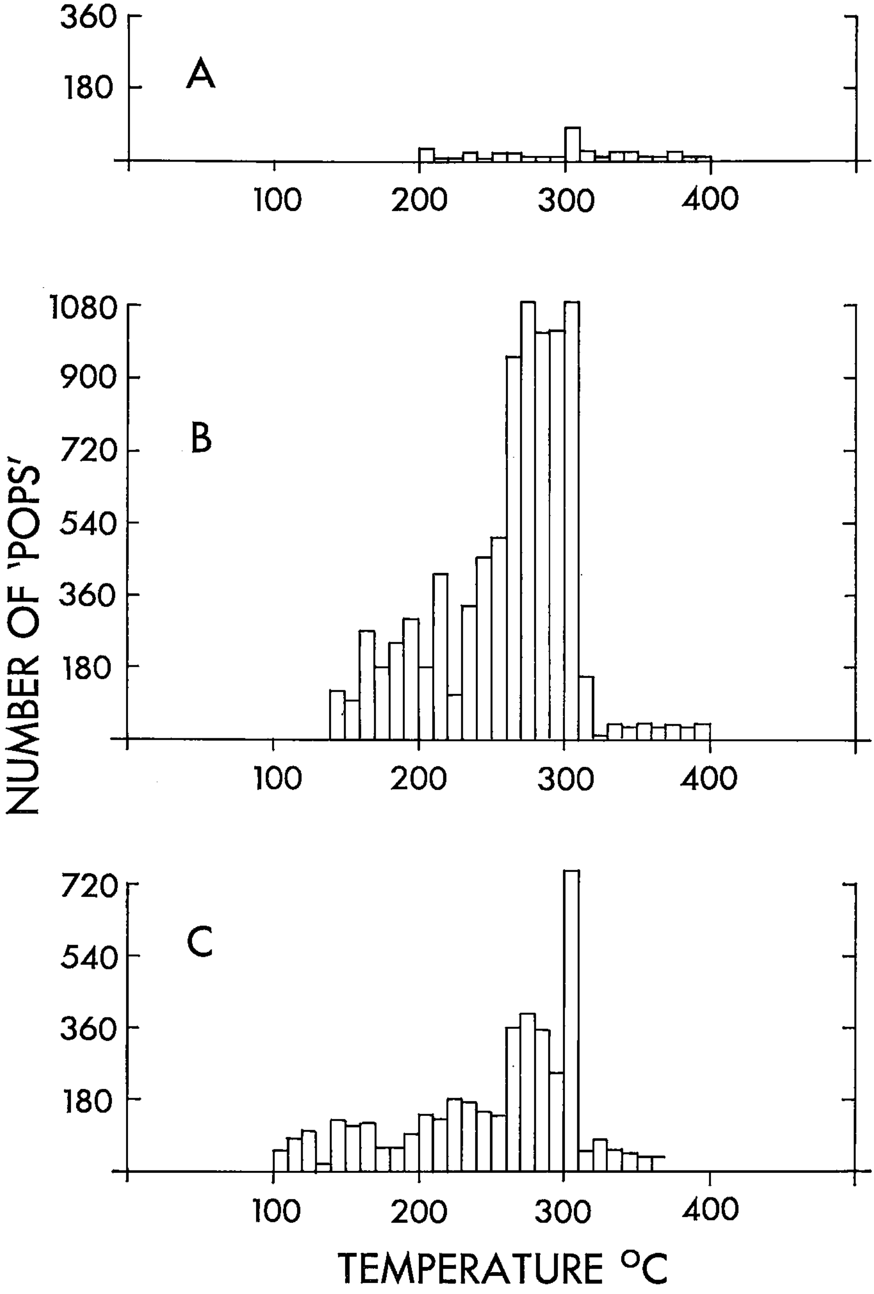

Fig. 3. Decrepigrams of the low temperature stage of decrepitation of muscovite 135. A—Normal. B—Muscovite hammered under water and air dried before heating. C—Same charge as in B. At the end of run B, the charge was soaked in water and then re-run.

High temperature stage. All the micas (except for some phlogopites) and all the chlorites gave rise to a distinct stage of decrepitation in the range of 550°–1,000° C.

The results are tabulated in Table 2, and shown graphically in Figure 4. Some typical decrepigraphs are reproduced in Figure 5. Muscovite, zinnwaldite and lepidolite all displayed vigorous or extremely vigorous decrepitation which started in the range of 550°–790° C. and rose to a peak rate in the range of 775°–860° C. By contrast, phlogopite gave rise either to weak decrepitation or none at all in the range 600°–1,000° C.

TABLE 2. HIGH TEMPERATURE STAGE OF DECREPITATION

Temperature of peak rates of decrepitation of chlorite and various micas. The relative amplitudes of the decrepitation peaks are indicated qualitatively by L—Low, M—Moderate, H—High and VH—Very High. B—Biotite, Ms—Muscovite, L—Lepidolite, Z—Zinnwaldite, P—Phlogopite and C—Chlorite.

Specimen # and type	Temp. of Peak °C	Rate of Heating °C/Min	Amplitude of Peak
1_1Ms	824	23	M
1_2Ms	823	25	M
2_1Ms	815	17	M
2_2Ms	815	20	M
9_1Ms	800	20	H
9_2Ms	812	23	H
111_1Ms	837	17	VH
111_2Ms	843	21	VH
115_1Ms	845	24	VH
115_2Ms	852	24	VH
117_1Ms	815	24	VH
117_2Ms	820	23	VH
135_1Ms	830	24	VH
135_2Ms	824	22	VH
137_1Ms	844	25	VH
112_1Z	805	19	H
112_2Z	800	19	VH
3_1B	714	19	L
3_2B	710	18	L
4_1B	698	21	H
4_2B	706	22	M
6_1B	740	24	M
6_2B	741	22	M
7_1B	735	21	H
7_2B	742	20	H
14_1B	715	21	H
14_2B	712	20	H
43_1B	720	22	M
43_2B	730	23	M
56_1B	740	21	M
56_2B	741	19	M
110_1B	736	21	VH
110_2B	734	23	VH
113_1B	706	22	M
113_2B	702	21	M
114_1L	815	25	H
114_2L	812	24	H
301_1L	781	20	VH
301_2L	786	22	VH
302_1L	812	17	VH
302_2L	825	19	VH
6_1C	764	21	M
6_2C	763	22	M
7_1C	762	22	M
7_2C	756	18	M
8_1C	759	19	L
8_2C	754	21	L
90_1C	748	22	M
91_1C	744	21	M
91_2C	746	23	M
102_1C	747	20	H
102_2C	750	19	H
140_1B	700	22	M
140_2B	706	23	M
146_1B	710	22	M
146_2B	705	21	M
215_1B	718	17	M
231_1B	626	18	H
231_2B	637	18	H
233_1B	635	20	L
106_1P	850	27	L
106_2P	846	23	L
116_1P	827	23	L
116_2P	830	20	L
119_1P	832	25	L
119_2P	838	26	L
120_1P	1015	15	L
207_1P	960	15	M
207_2P	959	17	M

If recorded, decrepitation generally started between 450° and 700° C. and rose to a low, commonly ill-defined, peak rate between 815° and 1,000° C. In one run (120_1) when no high temperature decrepitation was detected up to 1,000° C, heating was continued above this temperature.

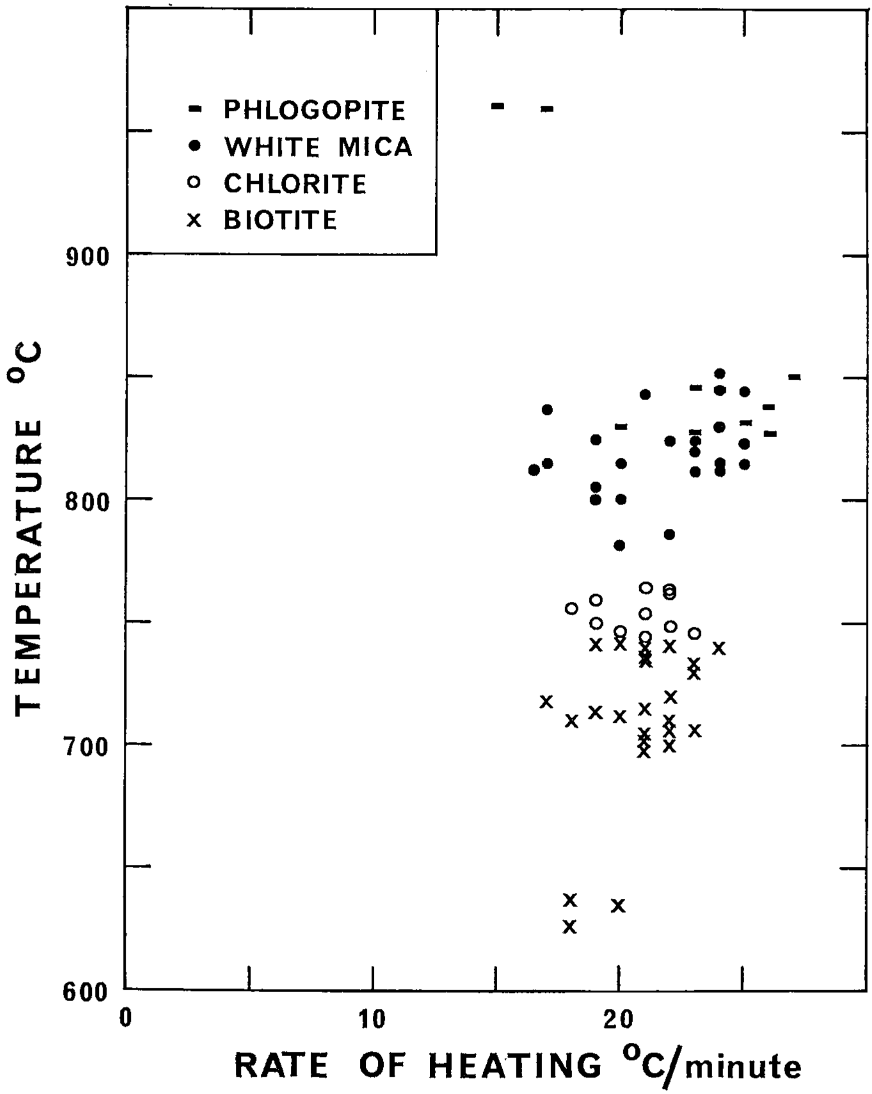

Fig. 4. High temperature stage of decrepitation. A plot of the temperature of the peak rate against rate of heating for chlorite and various micas, from data listed in Table 2.

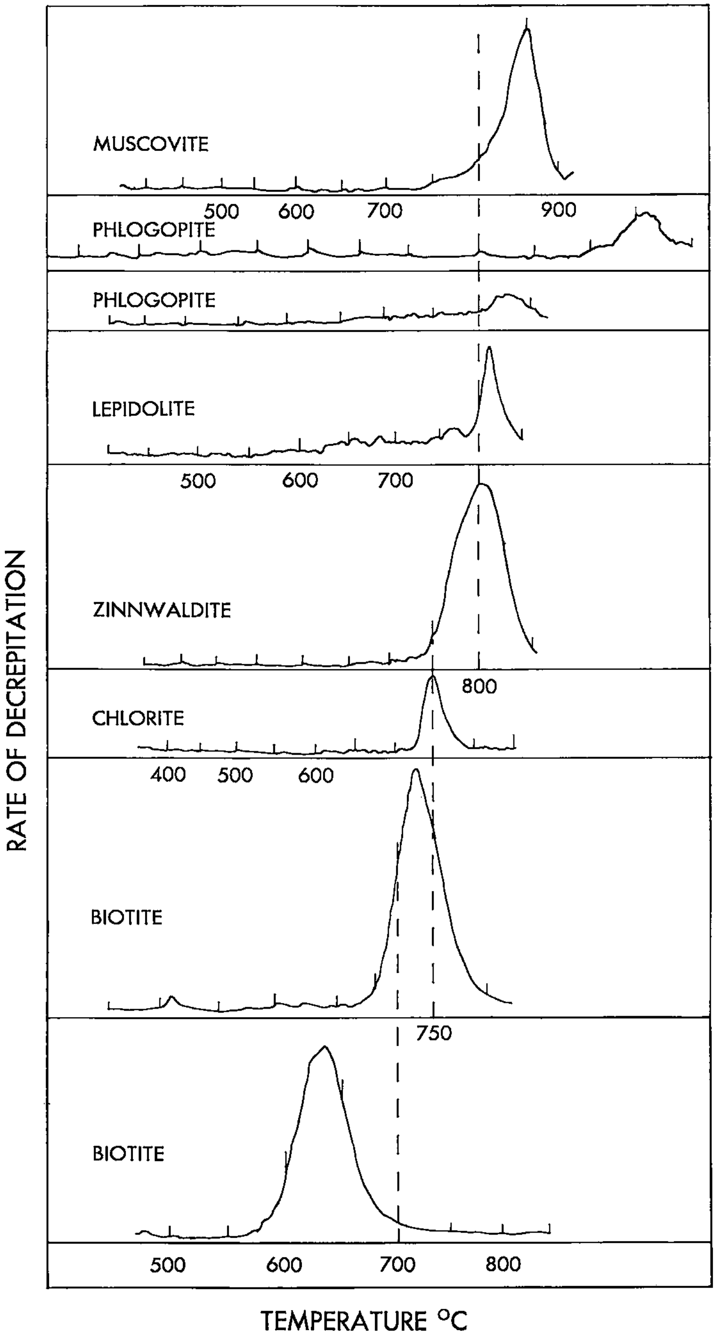

FIG. 5. High temperature stage of decrepitation. Typical decrepigraphs of chlorite and various micas.

Weak decrepitation was then detected with a peak at 1015° C. Both biotite and chlorite gave rise to similar, weak to moderate decrepitation. Whereas the decrepitation of chlorite started abruptly in the range 700°–730° C. and rose to a peak in the range 740°–760° C., the decrepitation of biotite started more progressively in a lower temperature range, 525°–650° C., and rose to a peak in the range 625°–737° C. Some biotites with a high iron oxide content decrepitated at lower temperatures than other biotites (Table 3), but a consistent relationship has not been established. The decrepitation of chlorite and various groups of micas are summarised in Table 4.

TABLE 3. HIGH TEMPERATURE STAGE OF DECREPITATION

The temperature of the peak rate and the ferrous and ferric iron content of some biotites. The temperature of the peak rate has been corrected for the rate of heating using the slope of the line of fit in Fig. 6 on the assumption that this slope is approximately the same for all these biotites. All temperatures of the peaks have been corrected to a rate of heating of 20° C/minute.

Specimen	Fe_2O_3	FeO	Fe_2O_3 and FeO	Temperature of Peak Rate
233	3.50	28.79	32.29	635° ± ?
231	7.20	22.36	29.56	637° ± 6° C
113	5.85	21.02	26.87	700° ± 1° C
140	5.60	19.80	25.40	696° ± 2° C
146	2.25	21.74	23.99	704° ± 2° C
215	1.46	16.62	18.08	727° ± ?

The analyses of samples 215, 231 and 233 were made in the analytical laboratories of the Geological Survey of Canada, under the supervision of Dr. J. A. Maxwell. (Insufficient material was available for re-runs of samples 233 and 215).

TABLE 4. SUMMARY OF THE DECREPITATION CHARACTERISTICS OF CHLORITE AND VARIOUS MICAS

		High Temperature Stage		
Phyllosilicate	Low Temperature Stage	Start °C	Peak °C	Relative amplitude of peaks
Muscovite	Detected	600–650	780–860	Moderate to very high
Phlogopite	Detected	450–700	815–above 1,000	Absent to moderate
Lepidolite	Detected	550–790	775–815	High
Zinnwaldite	Detected	600–700	795–810	Very high
Biotite	Detected	525–650	625–735	Low to high
Chlorite	Not detected	700–730	740–760	Low to high

The effect of the variation of the heating rate on the high temperature decrepitation peaks was examined for biotite 43. The temperature of the peak rate increased with the rate of heating. The relationship was apparently linear (Figure 6) and the best fitting line was determined by linear regression. The standard estimate of error was ±5.5° C.

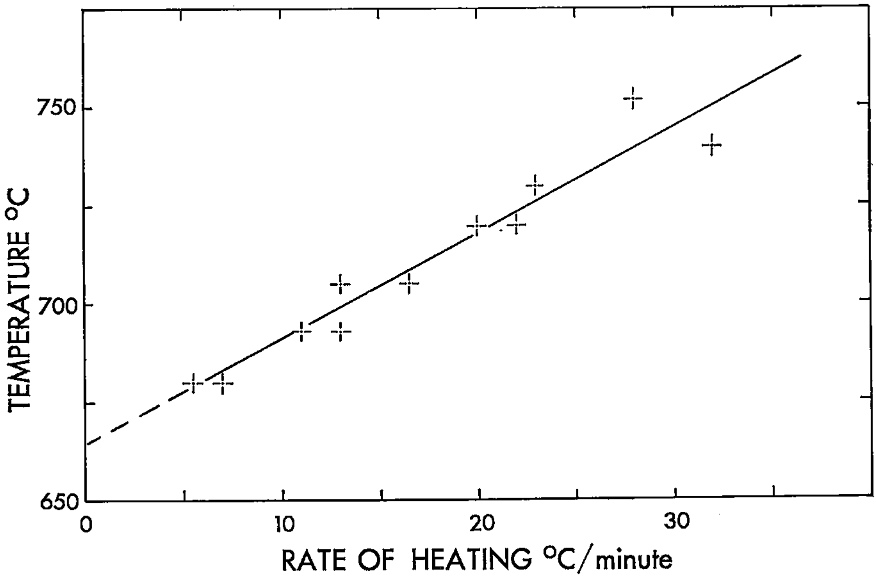

FIG. 6. High temperature stage of decrepitation of biotite 43. The effect of variation in the rate of heating on the temperature of the peak rate. The best line of fit was determined by linear regression.

Heating stage

Low temperature (20°–350°). Twelve heating stage runs were made on different thin flakes cleaved from the same sample of muscovite (135). Change was detected in only four runs and was not restricted to the narrow temperature range of the low temperature stage of decrepitation. Small haloes, commonly quite circular and composed of two or more concentric circles defined by Newton's colour fringes, appeared sporadically in the range of 100°–350° C. (Similar haloes were sometimes seen in the flakes before heating began). These haloes remained stationary and rarely expanded after they had appeared. On cooling the haloes disappeared at much lower temperatures than those at which they had appeared. On reheating, the haloes re-appeared at lower temperatures than on the first cycle of heating.

High temperature (350°–1,000° C.). In most samples of micas, small haloes like those described in the previous section appeared in increasing numbers above 400° C. With increase in temperature, these haloes expanded and at even higher temperatures, began to wander laterally in the plane of the cleavage. On encountering a crack or a boundary of a flake, the haloes disappeared and dark angular areas appeared in their places. Recoils in the mica flakes were occasionally coincident with the disappearance of haloes, suggesting that the latter were caused by small blisters of gas. The dark angular areas were attributed to the scattering of light at two or more interfaces within the mica flake, where splitting had occurred.

Haloes appeared in biotite above 500° C. With continued rise in temperature, they expanded and then began to move laterally. By 600° C. some had encountered boundaries or cracks and disappeared. Above 600° C more and more haloes appeared, expanded and started wandering laterally (Fig. 7). Up to 700° C many more haloes appeared for each halo that disappeared but above 700° C., the disappearance of haloes became much more frequent, and equalled the rate at which new haloes appeared. The disappearance of a halo near the edge of a flake commonly resulted in the appearance of a dark, angular area due to the local splitting (exfoliation) of the mica. Above 700° C. there was an overall darkening due to oxidation but this was less intense than the darkening due to splitting. By 750° C there were so many angular areas, a large number being superimposed on one another, that the mica was rendered effectively opaque. Few haloes appeared in muscovite below 750° C. but above this temperature many appeared, expanded and started to move laterally. Up to 800° C., the rate of appearance of the haloes was much greater than their rate of disappearance but above this temperature the situation was reversed. As in biotite, the disappearance of the haloes left darker angular areas with the result that the muscovite gradually became cloudy and lost its transparency. By 850° C. the muscovite was effectively opaque.

Phlogopite behaved differently from muscovite or biotite. A few haloes appeared in the range of 150°–200° C and also 600°–900° C. These haloes neither expanded nor moved. None of them was seen to disappear. Some flakes were however seen to recoil in the range 800°–850° C.

Weight loss measurements

The results of the weight loss measurements on biotite 43 indicate that the weight loss curves pass through a poorly defined inflection close to 300° C. (Figure 8). Close to 700° C. there is a relatively high rate of weight loss which decreases rapidly above this temperature.

Chemical analyses

Four biotite micas were subjected to complete chemical analyses (Table 5) and heated samples of these were analysed for ferrous and ferric iron, lattice water and fluorine (Table 6). The heated samples (Table 6)

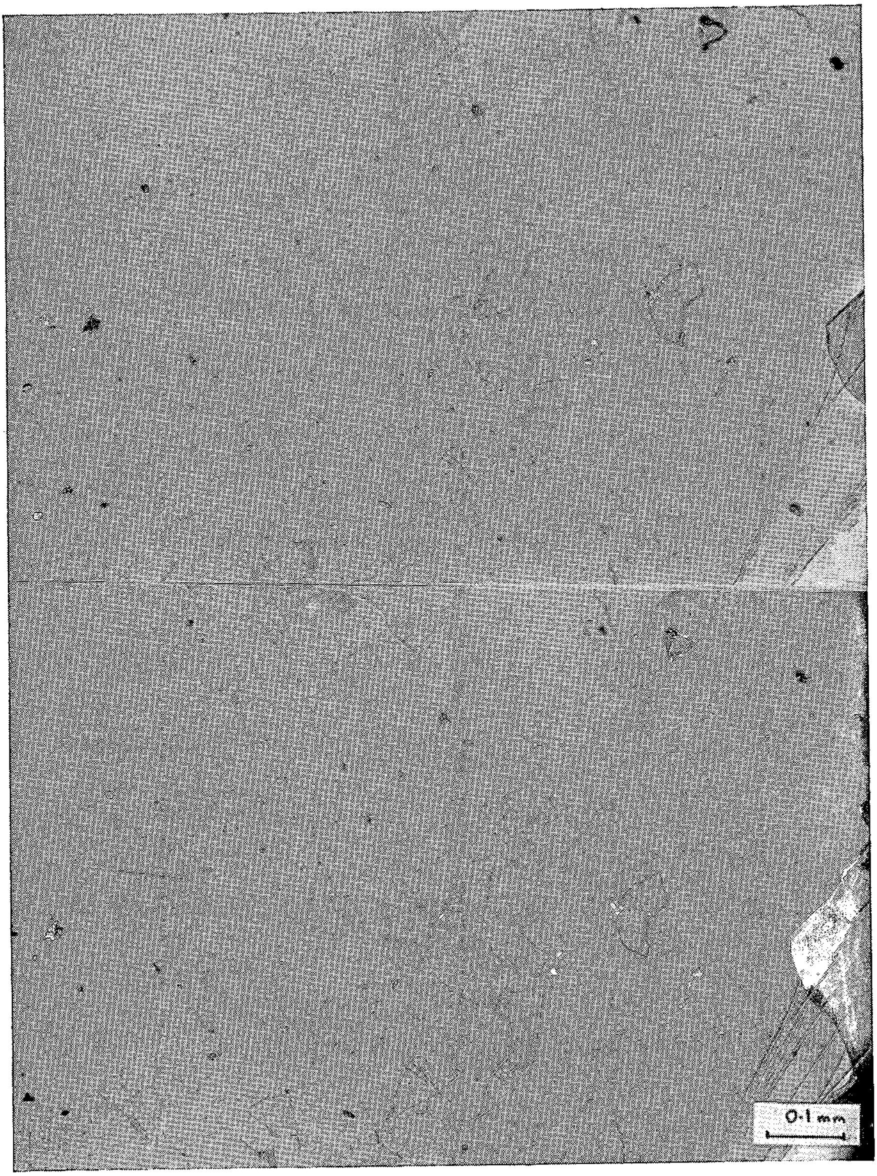

FIG. 7. Photographs of biotite 43 showing the haloes, interpreted to be blisters, that appear in micas during heating. (a) Biotite 43 at 550° C (top). (b) Biotite 43 at 695° C. Note the appearance of a radial fracture (an unusual event) above the halo in the NW quadrant. Note the migration of the halo in the NE quadrant towards the edge of the mica flake and the increase in size of that halo and the one just North of the EW cross hair. The scale is indicated by the bar below the photographs.

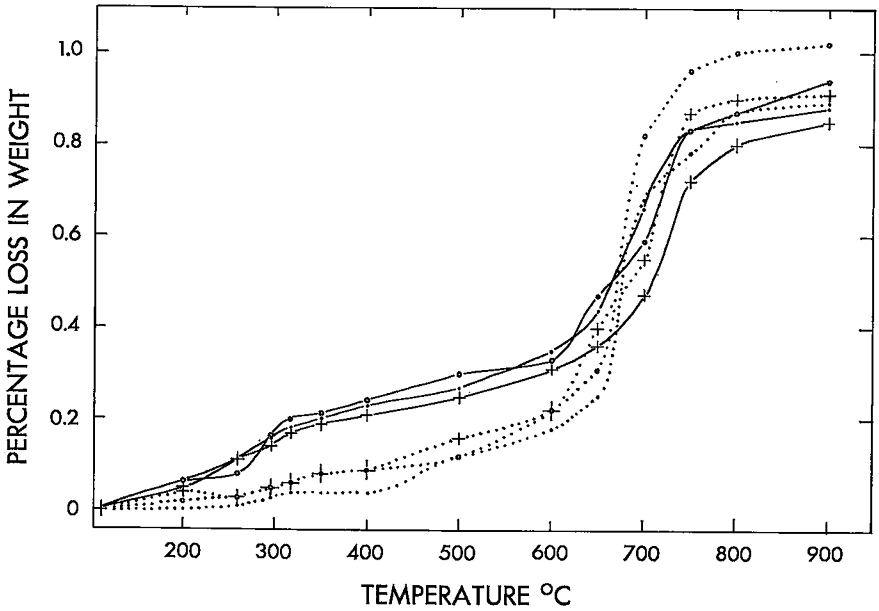

FIG. 8. Weight loss measurements of biotite 43. Solid lines are for three normal sample air dried at 110° C before initial weighing. Dotted lines are for three samples heated at 350° C, soaked in water and then air dried at 110° C before initial weighing.

had a lower percentage of lattice H_2O,F and FeO and a higher percentage of Fe_2O_3. A greater percentage of lattice H_2O was lost between 600°–800° C. than between 110° C. and 600° C. but a greater percentage of F was lost between 110°–600° C. than between 600°–800° C. The loss in weight of the lattice water more than compensated, apparently, the gain in weight due to the oxidation of FeO to Fe_2O_3 (Table 6 and Figure 8).

Variation of gas pressure on heating biotite and muscovite inside a pre-evacuated chamber

Biotite (sample 43) showed a steady low rate of increase in pressure from 200°–600° C. (Figure 9) followed by a sharp increase in rate to a maximum just above 700° C. A high rate was then maintained to the limit of heating 820° C.

A very low increase in pressure over muscovite (sample 111) was recorded from 100° to almost 700° C. but from the latter temperature up to 800° C. there was a continual increase in rate followed by a very marked increase close to 800° C. Above 800° C., slight condensation was noted in the cooler parts of the apparatus so that the high rate recorded

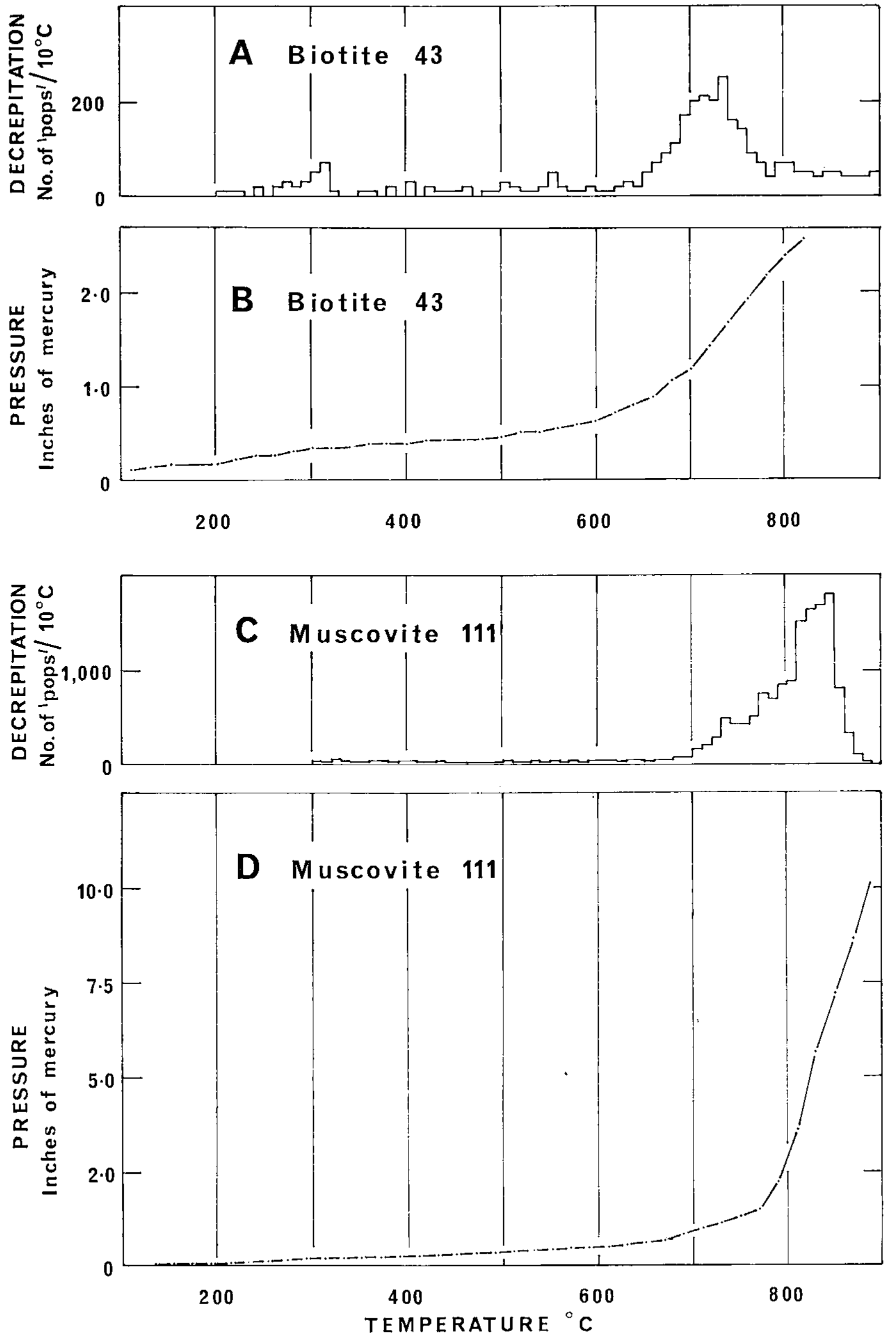

FIG. 9. Variation in gas pressure over biotite and muscovite. B and D show the change of gas pressure with temperature for biotite 43 and muscovite 111 respectively for a rate of heating of 15° C per minute. A and C are decrepigrams of the biotite and muscovite, respectively.

between 800°–850° C. should perhaps be even higher. No increase in rate was detected in the region of the low temperature decrepitation.

TABLE 5. COMPLETE CHEMICAL ANALYSES OF FOUR BIOTITES
Analyst: H. B. Wiik

	43	113	140	146
SiO_2	35.13	33.84	34.61	34.09
TiO_2	2.14	1.25	2.00	1.87
Al_2O_3	12.60	14.70	13.58	19.35
Fe_2O_3	5.67	5.85	5.60	2.25
FeO	20.37	21.02	19.80	21.74
MnO	0.59	0.80	0.62	0.17
MgO	9.48	8.18	9.50	6.51
CaO	0.00	0.00	0.00	0.00
Na_2O	0.62	0.70	0.64	0.55
K_2O	9.01	8.95	8.88	9.12
P_2O_5	0.00	0.00	0.00	0.16
H_2O	3.49	3.69	3.70	4.34
H_2O-	0.27	0.50	0.60	0.13
F	1.56	1.75	1.62	0.20
	100.93	101.23	101.15	100.48
−O for F	0.65	0.73	0.68	0.08
	100.28	100.50	100.47	100.40

TABLE 6. PARTIAL CHEMICAL ANALYSES OF FOUR BIOTITES
(heated to 110° C, 600° C and 800° C)
Analyst: H. B. Wiik

Sample Number	Temperature of Heating	FeO	Fe_2O_3	FeO and Fe_2O_3	H_2O	F
43	Normal—110° C	20.37	5.67	26.04	3.49	1.56
	600° C	11.01	15.23	26.24	3.01	1.48
	800° C	2.95	24.22	27.17	1.66	1.49
113	Normal—110° C	21.02	5.85	26.87	3.69	1.75
	600° C	12.31	14.98	27.29	3.47	1.68
	800° C	4.39	24.24	28.63	2.22	1.55
140	Normal—110° C	19.80	5.60	25.40	3.70	1.62
	600° C	13.32	12.52	25.84	3.69	1.50
	800° C	4.89	21.89	26.78	3.00	1.55
146	Normal—110° C	21.74	2.25	23.99	4.34	0.20
	600° C	10.08	15.06	25.14	3.44	0.15
	800° C	0.72	26.12	26.84	1.61	0.11

DISCUSSION

Low temperature stage. The reappearance of the decrepitation on reheating mica which had been soaked in water is taken as evidence that the decrepitation results from the loss of water. The weight loss measurements and the gas pressure measurements indicate that only a very small amount of water must be involved. From the experimental evidence that (i) the decrepitation was not eliminated by pre-heating the mica flakes for 24 hours in a vacuum at 250° C, (ii) the decrepitation occurs within a very narrow temperature range and (iii) the peak rate of the decrepitation varies only from 305°–340° C, it is concluded that the water is bonded or trapped in the mica flakes. Hammering mica had the effect of causing it to split along the cleavage around the areas of impact. The less intense decrepitation obtained after hammering the mica in air and the more intense decrepitation obtained after hammering the mica under water, points to the water causing the decrepitation being held within the mica flakes. The splitting along the cleavage planes permits water to enter the flakes. This water may either be bonded (adsorbed), perhaps in a regular array, between the mica sheets or trapped like liquid inclusions. Because the decrepitation recurs after simply soaking the mica flake in water for a few minutes, and because haloes (interpreted to be fluid inclusions) do not disappear within the temperature range of this decrepitation, the writer is more inclined to favour the former proposal.

None of the causes of decrepitation mentioned on page 437 satisfactorily accounts for this low temperature stage of decrepitation. Therefore decrepitation due to adsorbed or hygroscopic water should perhaps be distinguished from the other causes and designated separately as D8. The mica used in decrepitation was much coarser than the powdered material normally used in DTA. Knowing that DTA thermal effects vary considerably with the grain size of the material being heated (Grim, 1953 and Tsvetkhov & Valyashikhina, 1956) the author is reluctant to try to compare the decrepitation results with published DTA data on finely ground micas. Coarser flakes of mica however have been used by at least two groups. Tsvetkhov & Valyashikhina (1956) using flakes 1–2 mm^2 recorded very weak, indistinct thermal reactions in the range of 250°–350° C on their DTA traces of muscovite and biotite. Using the $-20+100$ fraction of biotite 43, Mr. B. D. Mitchell and Dr. R. C. MacKenzie, Macaulay Institute (personal communication) recorded a very weak but distinct endothermic reaction in the range 100°–400° C with a peak close to 320° C. These thermal reactions could be due to the loss of adsorbed water thought to be causing the low temperature stage of decrepitation.

High temperature stage. Of the proposed causes of decrepitation listed on page 437, the one most likely to account for this stage of decrepitation is the loss of lattice water during the thermal decomposition (D5) of the mica and chlorite flakes. The weight loss measurements, the chemical analyses, the gas pressure measurements and the heating stage results are consistent with the partial loss of lattice water causing decrepitation. Additional substantiation is the recording by Tsvetkhov & Valyashikhina

(1956) of thermal reactions from muscovite and biotite of similar grain size (1–2 mm^2) as that used in decrepitation. Endothermic reactions in muscovite and endothermic reactions, in part masked by exothermic (oxidation) reactions, in biotite, occur in the same temperature range as the decrepitation. These reactions are shown to be caused by the partial loss of lattice water.

The poorly defined, or complete lack of, decrepitation of phlogopite may be because the maximum rate of loss of lattice water takes place above the limit of heating in decrepitation, i.e. 1,000° C. This suggestion is supported by the report of Kiefer (1949) that the temperatures of the beginning and end of rapid dehydration of phlogopite are 1140° C and 1210° C respectively, whereas for muscovite, they are 785° C and 905° C respectively.

The heating stage results indicate that the decrepitation is not related to the temperature of evolution of gas (or fluid at low temperatures) within the mica flakes, but rather to the temperature of disappearance of gas which escapes explosively and splits the mica locally. That some decrepitation might be caused by the misfit of solid inclusions (D2) cannot be supported. For example phlogopite, which commonly contained a large number of rutile inclusions, decrepitated very weakly. In addition no optical strain effects were observed around any of the inclusions during heating. Decrepitation might also be caused by the loss of radiogenic gases from the mica. Any decrepitation caused by the loss of radiogenic argon, for example, must be slight. Chlorite, which has little interlayer potassium, also has a high temperature stage of decrepitation whereas phlogopite, which contains as much potassium as biotite, has little or no decrepitation.

The results of the chemical analyses of the four biotites along with the weight loss measurements of biotite 113, support the contention that some of the oxygen of the lattice water may have contributed to the oxidation of the ferrous to ferric iron with the resultant release of hydrogen. That this can occur, has been confirmed by Orcel & Renaud (1941) with the identification of hydrogen in the gas evolved during the heating of chlorite. Similarly, hydrogen was reported evolved by Tsvetkhov & Valyashikhina (1956) accompanying the oxidation of ferrous to ferric iron during heating of iron-rich biotite.

SUMMARY AND CONCLUSIONS

The present work reveals that micas decrepitate in two stages. Tentatively the low temperature stage is attributed to the rapid loss of an extremely small amount of adsorbed water from the mica flakes. The high temperature stage is attributed mainly to the partial loss of lattice water.

The dependence of the high temperature stage of decrepitation on the loss of lattice water from the micas, indicates that this type of decrepitation cannot be used in geothermometry. Instead, the decrepitation may be of use for identifying micas and also for examining the decomposition of micas and other phyllosilicates.

The results also suggest that micas, partially or completely included in other minerals, may give rise to decrepitation which might not be easily distinguished from some types of decrepitation used in geothermometry.

The decrepitation technique appears to be more sensitive to certain changes occurring in micas than other dynamic heating techniques. For this reason, decrepitation may be a useful auxiliary tool in the investigation not only of mica, but also of other hydrous minerals thus substantiating a proposal of Smith (1957) that "this might prove to be a useful petrographic method for determining the mineral types present in deuterically altered ferromagnesian minerals".

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APPENDIX

Description and location of specimens. The phyllosilicates used were either separated from crushed rock samples (A) or cleaved from coarse, commonly large, inclusion-free museum mineral speciments (B).

Specimen No.	Phyllosilicate	
1	Muscovite (A)	Muscovite separated from cordierite muscovite gneiss. No inclusions in muscovite. Location unknown. Donor W. W. Moorhouse.
2	Muscovite (A)	Muscovite separated from cordierite muscovite gneiss. No inclusions in muscovite. Location unknown. Donor W. W. Moorhouse.
3	Biotite (A)	Biotite separated from hornblende biotite schist. Biotite contains less than one per cent magnetite and chlorite. Location one quarter mile North of Fishtail Lake on the stream flowing from Luke Lake, Ontario. Donor W. W. Moorhouse.
4	Biotite (A)	Biotite separated from garnet, staurolite, biotite gneiss. Biotite is devoid of inclusions. Location Chisel Lake, Manitoba. Donor H. Williams.
6	Biotite & Chlorite (A)	Biotite and chlorite separated from a garnetiferous amphibolite. Biotite and chlorite occur as discrete flakes but some biotite flakes contain interleaved chlorite. Location Chisel Lake, Manitoba. Donor H. Williams.
7	Biotite & Chlorite (A)	Biotite and chlorite separated from a staurolite biotite schist. Biotite and chlorite occur as discrete flakes but some biotite flakes contain minor interleaved chlorite. Location Chisel Lake, Manitoba. Donor H. Williams.
8	Chlorite (A)	Chlorite separated from meta-graywacke. No inclusions were noted in the chlorite but there was some adhering quartz and feldspar in the fraction prepared for decrepitation. Location Province of Quebec. Donor W. Gilman.

Specimen No.	Phyllosilicate	
9	Muscovite (A)	Muscovite separated from cordierite, anthophyllite muscovite schist. Location North shore of Fishtail Lake, Ontario. Donor W. W. Moorhouse.
14	Biotite (A)	Biotite separated from garnet, kyanite biotite gneiss. Biotite contains very minor chlorite alternation. Location Sudbury, Ontario. Donor D. H. Gorman.
43	Biotite (B)	Cleaved from large biotite book, and is quite free of inclusions. Location Faraday Township, Ontario. Donor D. H. Gorman.
56	Biotite (B)	Cleaved from a large biotite crystal. No inclusions. Location unknown. Donor D. H. Gorman.
90	Chlorite (A)	Chlorite separated from garnet chlorite schist. Chlorite is free of inclusions, but some adhering feldspar was present in the fraction prepared for decrepitation. Location 580 foot level, Snow Lake Mine, Manitoba. Donor F. G. Smith.
91	Chlorite (A)	Chlorite separated from garnet chlorite schist. Chlorite is devoid of inclusions. Location Charlemont, Massachusetts. Donor F. G. Smith.
102	Chlorite (B)	Cleaved from large chlorite specimen. Chlorite is free of inclusions and impurities. Location unknown. Donor D. H. Gorman.
106	Phlogopite (B)	Cleaved from large crystal. Phlogopite contains numerous fine acicular rutile inclusions in trigonal array. Location unknown. Donor D. H. Gorman.
110	Biotite (B)	Cleaved from large crystal. Contains less than 1 per cent magnetite inclusions but locally carbonate forms thin skins on some cleavage surfaces. Location unknown. Donor D. H. Gorman.
111	Muscovite (B)	Cleaved from a large crystal. Muscovite is devoid of inclusions. Location Peru. Donor D. H. Gorman.
112	Zinnwaldite (B)	Cleaved from pegmatitic aggregate of coarse crystals. Zinnwaldite contained very large number of acicular crystals (rutile?) in trigonal array. Location Usikos Lithium Mine, South Africa. Donor D. H. Gorman.
113	Biotite (B)	Cleaved from large book. Biotite is free of inclusions. Location American Nepheline Co. Mine, Nephton, Ontario. Donor D. H. Gorman.
114	Lepidolite (B)	Cleaved from coarse aggregate of mica crystals. Lepidolite is devoid of inclusions. Location Usikos Lithium Mine, South Africa. Donor D. H. Gorman.
115	Muscovite (B)	Cleaved from large flake. Muscovite is devoid of inclusions. Location Bolivia. Donor D. H. Gorman.
116	Phlogopite (B)	Cleaved from large flake. Phlogopite contains abundant acicular crystals of rutile in trigonal array. Location Madagascar. Donor D. H. Gorman.

Specimen No.	Phyllosilicate	
117	Muscovite (B)	Cleaved from large flake. Muscovite is devoid of inclusions. Location Hairi Mica Supply Co., Giridih, India. Donor D. H. Gorman.
119	Phlogopite (B)	Cleaved from large flake. Phlogopite contains acicular inclusions, probably rutile, in trigonal array. Location Ontario. Donor D. H. Gorman.
120	Phlogopite (B)	Cleaved from large flake. Phlogopite contains acicular inclusions of rutile in trigonal array. Location Wakefield, Quebec. Donor D. Gorman.
135	Muscovite (B)	Cleaved from large crystal of muscovite. Locally the mica has iron stain on cleavage surfaces. Location unknown. Donor D. H. Gorman.
137	Muscovite (B)	Cleaved from large crystal. No inclusions. Location unknown. Donor D. H. Gorman.
140	Biotite (B)	Cleaved from large crystal. Biotite contains less than 1 per cent magnetite inclusions and locally has thin wedges of carbonate within cleavage planes. Location Renfrew County, Ontario. Donor D. H. Gorman.
146	Biotite (B)	Cleaved from large crystal. Biotite is devoid of inclusions. Location Murray Bay, Quebec. Donors V. B. Meen and J. Mandarino. Royal Ontario Museum no. R.O.M. 19329.
205	Phlogopite (A or B)	Source of mica unknown. Contains oriented acicular inclusions of rutile. Location unknown. Donor J. Rimsaite.
207	Phlogopite (A or B?)	Source of mica unknown. Phlogopite occurs as coarse flakes free of inclusions but some flakes are blistered. Location unknown. Donor J. Rimsaite.
215	Biotite (A or B?)	Source of mica unknown. Biotite contains about 2 per cent chlorite and less than 1 per cent opaque inclusions. Location unknown. Donor J. Rimsaite.
231	Biotite (A or B?)	Source of mica unknown. Biotite contains trace of chlorite and rare opaque inclusion. Location unknown. Donor J. Rimsaite.
233	Biotite (A or B?)	Source of mica unknown. Biotite contains trace of chlorite. Location unknown. Donor J. Rimsaite.
301	Lepidolite (B)	Mica separated from large book. Lepidolite is free of inclusions. Location Brown Derby Pegmatite, Gunnison Co., Colorado. Donor E. W. Heinrich.
302	Lepidolite (A)	Mica separated from coarse pegmatitic aggregate of quartz, feldspar and mica. Lepidolite occurs as slightly curved crystals which are devoid of inclusions but which are in part intergrown with quartz and very minor biotite. Location Brown Derby Pegmatite, Gunnison Co., Colorado. Donor E. W. Heinrich.

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