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The effect of disc mill grinding on some rock-forming minerals

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ABSTRACT. Swing mill grinding causes appreciable oxidation of chlorite and biotite, and calcite becomes decarbonated. Magnetite is not affected. Relatively large grains can survive up to six minutes continuous grinding.

THE recent developments of rapid instrumental analytical techniques necessitates faster sample preparation, particularly in the comminution of the rock or mineral specimen to a powdered sample. The swing mill has proved to be a very efficient rock crushing device, but there has been very little investigation on the effect of this crushing technique on the chemical composition of rocks and minerals. Fitton and Gill (1970) have investigated the effect of swing mill grinding on the ferrous oxide content of rocks, and have shown that there is, in some cases, a drastic lowering of the ferrous oxide content on grinding. This paper presents the results of further investigation of the effect of this grinding technique on rocks and minerals.

Materials investigated included a greenschist containing calcite and several hydrous minerals; pure samples of the minerals chlorite, biotite, magnetite, and actinolite; and a welded tuff containing no hydrous phases and a negligible water content. The grinding techniques followed are essentially similar to those described by Fitton and Gill (op. cit.) using a Tema laboratory disc mill model T100 with a tungsten carbide grinding barrel. Various crushing techniques were used on 50 g of fresh 1 cm cubes of sample. Results are given in Tables I and II.

Analytical techniques used included Wilson (1955) for ferrous iron, Peck (1964) for the determination of carbon dioxide, and the Penfield technique for the determination of total water.

Results

Grain size. Following crushing of the greenschist for varying lengths of time, a size analysis was carried out on a 50 g sample. Results are essentially as expected, and similar to those presented by Leake *et al.* (1969). The grinding technique is very efficient, in all cases more than 65% of the total sample passed the 230 mesh 4.00 φ sieve. A disturbing feature of the grain size analyses was the presence of single grains retained on the 120 mesh 3.0 φ sieve, although the quantities in all cases were less than 1%. Presumably these larger grain sizes have been cushioned to some extent by the packing of very small particles around them. One of these large grains in a small sample taken for analyses (e.g. ICP emission spectrography or neutron activation analyses) could produce anomalous results, particularly if the large grain is monomineralic. Sieving during crushing should remove this problem.

Carbon dioxide content. There is a dramatic drop in the carbon dioxide content of the greenschist on crushing, the decrease being greatest in the rocks crushed for the longest time (Table I). This decrease is probably due to the breakdown of the calcite caused by the transient high temperatures developed in the mill during crushing, although

TABLE I. *Water and carbon dioxide contents of samples following grinding*

	CO_2		H_2O^+	H_2O^-
Secs.*	A	B	B	B
0	21.98	3.10	—	-
15	—	2.50	—	—
45	—	1.64	—	—
60	21.80	1.53	3.68	0.03
120	—	—	3.82	0.10
180	21.61	0.85	3.70	0.14
240	—	—	3.75	0.39
300	—	—	3.89	0.42
360	20.70	0.70	3.80	0.54

A = 1/1 calcite-quartz mix. B = greenschist.
* Total grinding time in seconds.

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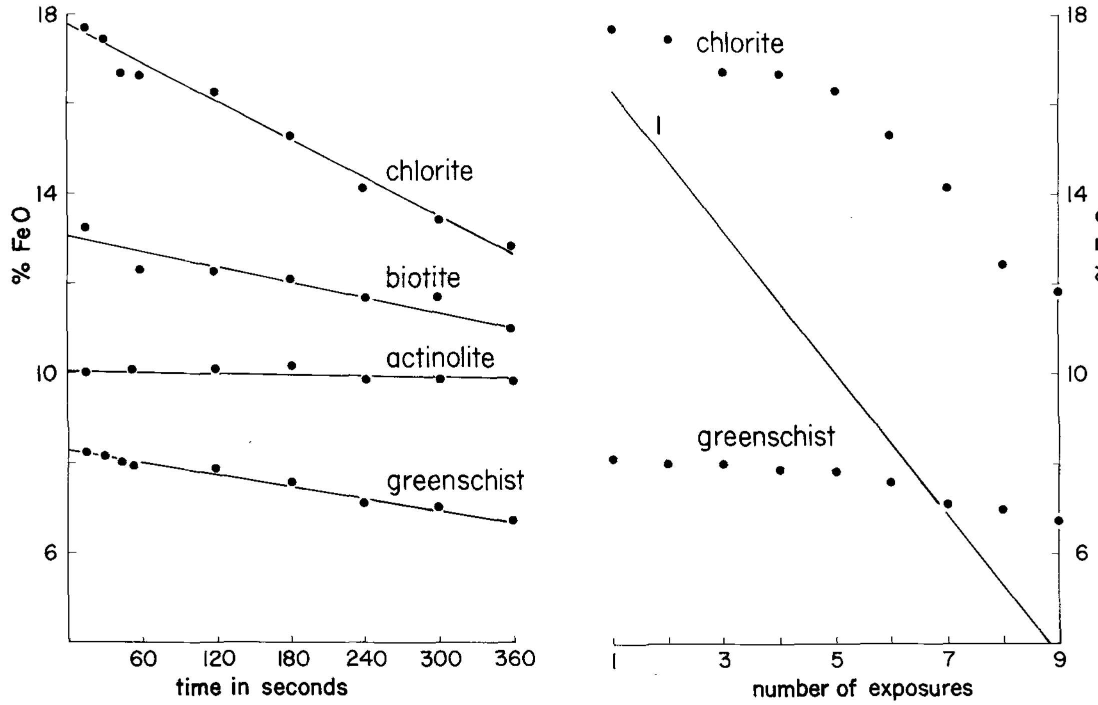

FIGS. 1 and 2. FIG. 1 (*left*). Variation in FeO content with total grinding time. FIG. 2 (*right*). Variation in FeO content with the number of times mill opened for sampling and hence access of atmospheric oxygen to sample. (1) oxidation curve for chlorite assuming all available atmospheric oxygen in mill is used for oxidation.

overall the temperature of the mill never rises above approximately 50 °C even after six minutes grinding. A loss of carbon dioxide was noted for a 50/50 mix of quartz and calcite (Table I). The percentage loss was not as high as for the greenschists, possibly reflecting the less severe crushing of 50 % calcite in quartz compared with 7 % calcite in the greenschist.

Water content. The absorbed water content increases with increasing crushing time, reflecting the decreasing grain size and increasing surface area as pointed out by Mauzelius (1907). The structural water shows very little variation, the variation is not systematic and may be a reflection of the precision of the sampling and the analytical technique.

Ferrous oxide content. Results are given in Table II and shown in fig. 1. The ferrous oxide content of the greenschist shows a marked decrease with crushing, comparable to that quoted for similar rocks by Fitton and Gill (1970). Up to a total of three minutes grinding time the continuously ground samples show similar oxidation to those samples where grinding has been interrupted by sampling. For six minutes continuous grinding oxidation is less pronounced than for six minutes grinding interrupted by sampling (and hence access to atmospheric oxygen). These results led Fitton and Gill (op. cit.) to suggest that atmospheric oxygen was responsible for the oxidation. The results also support their conclusion that hydrous

TABLE II. *FeO contents of rocks and minerals sampled at intervals during a 360 sec. grinding period*

Secs.	1	2	3	4	5	6
15	—	8.12	17.70	13.27	10.02	—
30	—	8.08	17.47	—	—	—
45	—	8.02	16.77	—	—	—
60	7.77	7.93	16.73	12.36	10.12	30.52
120	7.82	7.85	16.34	12.33	10.18	30.61
180	7.87	7.60*	15.32	12.13	10.19	30.64
240	7.77	7.14	14.18	11.67	9.85	30.50
300	7.89	7.03	13.43	11.70	9.89	30.52
360	7.89	6.76†	12.83	11.00	9.86	30.59

1. welded tuff	4. biotite
2. greenschist	5. actinolite
3. chlorite	6. magnetite

* 180 seconds continuous grinding = 7.57 %

† 360 seconds continuous grinding = 7.07 %

silicates are very susceptible to oxidation. Oxidation could be brought about by structurally combined water in the hydrous minerals, similar to the oxidation of biotite as observed by Tsvetkov and Valyashikhina (1956). An andesitic welded tuff with 0.3 % total water and no hydrous minerals showed no oxidation on prolonged grinding (Table II).

There are four ferrous-iron-bearing minerals in the greenschist, any or all of which could be oxidized during grinding. Separate, monomineralic samples of each of the four minerals, chlorite, biotite, actinolite, and magnetite were individually crushed and their ferrous iron contents determined. Magnetite shows no detectable oxidation on crushing, in agreement with the results on the magnetite-bearing welded tuff. Actinolite does not appear to become significantly oxidized on crushing. Chlorite shows more oxidation than biotite. Some form of auto-oxidation of these hydrous minerals may have taken place, i.e. FeO being oxidized by hydroxyl groups in the mineral structure. However, the analytical techniques are not sufficiently precise to check this hypothesis, particularly if some of the oxidation is due to atmospheric oxygen. Atmospheric oxidation appears to be the most important process in view of the fact that there is a good correlation between oxidation and the number of times the mill is opened to the air (fig. 2). Maximum possible oxidation of the chlorite due to the availability of atmospheric oxygen every time the mill is opened for sampling, is shown on fig. 2.

Mineralogy. With increasing grinding times the X-ray diffraction peaks of minerals in the greenschist become progressively smaller, and the peak to background ratio decreases. Individually crushed minerals showed similar changes, the greatest changes being shown by chlorite. Typical results are shown in fig. 3. The degradation is presumably due to a combination of decreasing crystallite size and increasing lattice distortion with increasing grinding time. No peak shift was observed on any of the minerals investigated, although Bain (1972) has reported small changes in lattice parameters following extreme oxidation. Mechanical grinding can convert calcite to aragonite, e.g. Jamieson and Goldsmith (1960). No aragonite reflections were observed in any of the calcite-bearing samples, even after six minutes grinding.

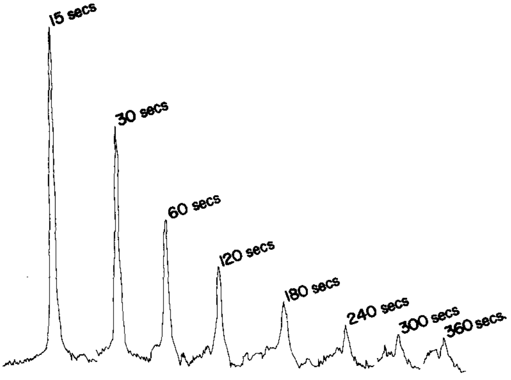

FIG. 3. Diffractometer trace of 003 peak of chlorite crushing for varying times. Trace taken using Cu-$K\alpha$ radiation, Ni filter.

Causes of chemical changes

The swing mill crushing technique involves the impact and sliding of ring and disc anvils with the sample. High, local transient temperatures, up to 1000 °C, sufficient to promote rapid oxidation of small mineral grains, can be developed between sliding surfaces as a result of friction (Bowden and Thomas, 1954), and presumably similar temperatures could be produced at the contact surface between the mill and a mineral grain. There is a reasonable correlation between mineral hardness and oxidation as well as mineral water content and oxidation. Transient high temperatures are presumably also responsible for the dissociation of calcite, particularly since the admixture of other minerals lowers the dissociation temperature, (Warne and McKenzie, 1971). Pressure has a significant effect on the breakdown of carbonates, although the pressures developed in the present crushing system are unknown.

Conclusions

The swing mill is a very effective method of reducing rock to a powdered form suitable for chemical analyses. However, there are some disadvantages.

In nonhomogeneous materials, some of the more resistant components may not be crushed to the same size as less resistant components. This may be important where very small quantities are required for analysis.

Calcite tends to become decarbonated during crushing.

Some hydrous minerals are oxidizing during crushing. The degree of oxidation varies with the mineral.

X-ray diffraction patterns of crushed material become increasingly degraded with increasing crushing times.

The suggestion of Fitton and Gill (1970) to remove samples for FeO analysis from the mill after 30 secs. grinding is recommended. Alternatively a different, less severe crushing technique may be used on a separate aliquot of the sample.

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