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EXAMINATION OF ROCK SURFACES WITH THE SCANNING ELECTRON MICROSCOPE

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EXAMEN DE LA SURFACE DES ROCHES A L'AIDE DU MICROSCOPE ELECTRONIQUE A BALAYAGE

SOMMAIRE

Une surface de roche (grauwacke), a été examinée au microscope électronique à balayage, avant et après, gravure de la surface pour des périodes de temps successivement plus longues dans du 2M NaOH à 38° C (100 °F). La même région de l'échantillon a été relocalisée au microscope afin de pouvoir étudier les effets de l'alcali sur le minerai et la microstructure a pu être examinée par comparaison directe des micrographes. La structure feuilletée des silicates manifestait une exfoliation.

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Short Technical Note

Examination of rock surfaces with the scanning electron microscope

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SUMMARY

A fracture surface of rock (greywacke) was examined with the scanning electron microscope prior to, and after, surface etching for successively increasing time intervals in 2 M NaOH at 38°C (100°F). The same area of rock was successfully relocated in the microscope so that the effect of the alkali upon minerals and microstructure could be observed by direct comparison of micrographs. Layer-structure silicates showed exfoliation.

A fracture surface of rock (greywacke) was examined with the scanning electron microscope, prior to, and following successively increasing time intervals in 2 M NaOH at 38°C (100°F). As rock is non-conducting, a thin metal coating is required to prevent the build-up, on the surface, of an electric charge that would otherwise cause a serious loss in resolution. Aluminium coating was used because it is rapidly removed by the alkali exposing the bare rock to attack. In other experiments the rock was not coated and the microscope was operated at a low voltage to minimize charge build-up. Results were generally less satisfactory with non-coated samples.

A magnification of $\times 230$ was used and a mosaic constructed that covered an area of 3 or 4 mm by $1\frac{1}{2}$ or 2 mm of sample surface. One or more photographs at $\times 2300$ magnification were taken in each frame of the mosaic as well as a small number of photographs at higher magnification in order to study the effect on the fabric of the rock of increasing periods of time in alkali (Gillott, 1969). This could be accomplished most directly by observing the same area of sample before and after it had been immersed in 2 M NaOH.

In the first experiments two orthogonal lines were scratched on the sample surface. The etching action of the alkali tended to obscure these markings, however, and in later experiments a piece of gold leaf was attached to the sample. Modified sample holders were made. The side of the stub was threaded and a screw-on cap constructed with a rim that overlapped the outermost portion of the sample. When the cap was screwed down over the sample the rim held both the gold leaf and sample in place. The cap and sample holder stub were made of stainless steel so that sample, holder and gold leaf marker could all be immersed in alkali as a unit. After a specified time this unit was removed from alkali and the sample was re-examined on the microscope; the same area of sample could be relocated without too much difficulty.

Fig. 1 presents views of a fracture surface of untreated rock and views of the same area after 1 month in 2 M NaOH at 38°C (100°F). Fig. 1(a and b) represent one frame each of the respective mosaics; Fig. 1(c and d) are enlargements of the central portion of each. In the lower-power magnification there are feldspars, quartz and mica-type minerals. Comparison shows that many of the minerals are relatively unaffected by the alkali after 1 month though the layer-structure silicates show exfoliation. This is most clearly seen in the views taken at higher magnification (c and d). The significance of this work to the understanding of the mechanism of alkali-aggregate reaction involving greywackes and phyllites (Duncan & Swenson, 1969) will be described in detail elsewhere.

Fig. 1. Scanning electron micrographs of selected area of greywacke before and after treatment with alkali, taken at 20 kV accelerating potential on a Cambridge Stereoscan Mk II scanning electron microscope. (a, c) Untreated; (b, d) 1 month 2 M NaOH at 38°C.

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