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# Chemical Analysis of Slickensides From Coal Mine Roof Shale

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# Chemical Analysis of Slickensides From Coal Mine Roof Shale

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# CHEMICAL ANALYSIS OF SLICKENSIDES FROM COAL MINE ROOF SHALE

by

George A. Savanick[1] and Rayfield W. Cabaniss[2]

## ABSTRACT

Coal mine roof shales fracture preferentially at slickensided surfaces because of weak or nonexistent adhesion between two slickensided rock surfaces. The purpose of this Bureau of Mines research was to characterize chemically the thin film of smooth, glossy material that makes up the slickenside. This material was analyzed by electron spectroscopy and ion scattering spectrometry while in place on the rock surface and by standard methods of quantitative chemical analysis on concentrate removed from the rock. These analyses showed that the chemistry and mineralogy of the slickensides are similar to that of the adjacent shale.

## INTRODUCTION

Shales in the roof of coal mines often break preferentially along slickensided surfaces forming blocks of rock which are loosely held in the roof. These rock blocks can fall without warning and cause serious injury to miners (fig. 1)(4).[3] These rock masses are especially treacherous because their presence cannot always be detected and because their dimensions are often smaller than the roofbolt spacing in a normally adequately supported area of the roof so that they can fall between the bolts.

The surface of these rock blocks is often coated with a thin film of glossy material. This material is often known by the term "slickensides" because it is slick to the feel. Since the roof rock breaks preferentially along the slickensided surfaces, the slickensides evidently define a surface of weakness in the roof rock. This paper describes the chemistry and mineralogy of the slickenside material.

This problem was approached by performing a detailed chemical and mineralogical analysis of slickenside material taken from roof rock collected from working mines as part of the Bureau of Mines Chemical Stabilization of Coal

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[1] Research physicist.

[2] Chemist.

[3] Underlined numbers in parentheses refer to list of references at the end of this report.

FIGURE 1. - Illustration of the hazards posed by fall of a kettle bottom.

Mine Strata Program. The results of these analyses were compared with those of similar analyses from areas adjacent to the slickensides to determine how the slickensided areas differ from the bulk of the shale.

Some of the analyses could be performed while the slickenside was in place on the fracture surface. These included light and electron microscopy of the surface morphology, film thickness measurements, electron diffraction to determine the degree of crystallinity of the surface of the film, and ion scattering and electron spectrographic measurements of the chemical composition of the surface film.

It was impossible, however, to do a quantitative chemical and mineralogical analysis of the in place slickenside. To do this the slickenside had to be separated from the rock substrate. After the separation, the material was subjected to various techniques of quantitative chemical analysis. The minerals present were identified by X-ray diffraction, and a chemical norm was calculated from the chemical analysis to make a quantitative estimate of the mineralogical composition of the specimen.

This report includes a thorough chemical study of slickensides found in roof falls from Pennsylvania, West Virginia, and Kentucky. A detailed chemical and mineralogical analysis of these samples is tabulated and compared with similar analyses of adjacent shale from the same roof fall.

## ACKNOWLEDGMENTS

The authors gratefully acknowledge the assistance rendered by T. C. Shelton, G. Robert Vandenbos, and R. F. Goff. Some of the samples were collected by Dr. Shelton, Virginia Polytechnic and State University. The quantitative chemical analyses of the samples were performed in the Analytical Chemistry Laboratory of the Twin Cities Metallurgy Research Center. Mr. Vandenbos performed the scanning electron microscopy and measured the film thickness. Mr. Robert F. Goff of the 3M Company provided the Ion Scattering Spectrometer measurements. The work was supported under the Bureau of Mines Chemical Stabilization of Coal Mine Strata Program.

## SAMPLING

Data on slickensides came from five mines: the Wierton Steel Company mine at Isabella, Pa.; the Gateway Coal Company mine at Lippincott, Pa.; the Armco Steel Company mine at Mountcoal, W. Va.; the Matthews No. 1 mine at Middlesboro, Ky.; and the U. S. Steel Company mine No. 4 at Gary, W. Va. The samples from the Isabella and Gateway mines were collected by the authors and were in the form of potholes in the shale above the Pittsburgh coal seam which fell between the roofbolts in a working section of the mine. The Matthews, Armco, and U. S. Steel Company samples were collected by T. C. Shelton as part of sample procurement for the Bureau of Mines project on Chemical Stabilization of Coal Mine Strata.

The sample from Mountcoal, W. Va., was taken from the shale under the No. 2 gas seam of coal. The sample from the Matthews No. 1 mine was fallen roof rock from the shale above the Jelleco coal seam. The sample from Gary, W. Va., was fallen roof rock from the shale above the Pocahontas No. 3 coal seam.

## EXPERIMENTAL PROCEDURE

### Characterization of Intact Slickensides

A cursory examination revealed that the fracture surfaces of many of the fallen rocks were coated with a black, glossy, thin film. Since information on the surface of this film is necessary to understand its adhesive properties, it was necessary to characterize this film in place on the fracture surface. This fracture severely restricted the analysis to techniques capable of sensing the surface without interacting with the substrate.

Each slickensided surface was examined microscopically. A reflected light microscope and a JEOLCO JSM-2[4] scanning electron microscope was used

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[4] Reference to specific equipment or manufacturers does not imply endorsement by the Bureau of Mines.

to reveal surface features larger than 250 A. A Bendix Proficorder Model 6, a profile measuring instrument, was used to give a rough estimate of the thickness of the slickenside film. This estimate was inferred by measuring the height of a step between the slickenside film and the substrate formed by scraping off a portion of film and leaving an adjacent area intact. An uncertainty exists in this measurement because of the difficulty involved in defining just when the substrate is reached during scraping. The only criterion available for judging when the substrate was reached was the appearance of the dull grey color characteristic of the shale.

The degree of crystallinity of the slickensided surfaces was determined by performing high-energy reflection electron diffraction with an RCA-EMU 3 transmission electron microscope. The slickensided surfaces yielded a diffraction pattern characteristic of randomly oriented crystallites in a polycrystalline solid; thus it can be safely inferred that the slickensides contain fine grained crystalline material.

A qualitative chemical analysis of these surfaces was performed with an energy dispersive X-ray analyzer attached to the scanning electron microscope (SEM). This analysis indicated the presence of silicon, aluminum, and potassium in these films. These chemical data were corroborated by two methods of surface elemental analysis: ion scattering spectrometry (2) and electron spectroscopy for chemical analysis (ESCA) (3). Ion scattering spectrometry was performed on slickensides from the Isabella mine by bombarding the surface with a beam of noble gas ions and recording the energy spectrum of the ions scattered at a given angle. The energy at which peaks occur correspond to the mass of each constituent on the surface. The ion scattering spectrogram contained prominent oxygen, silicon, and aluminum peaks and indicated the probable presence of aluminum. Although carbon is obviously present in these shales, it was not detected in this spectrum because ion scattering spectroscopy is not particularly sensitive to it.

Slickensides from the U. S. Steel Company mine at Gary, W. Va., were analyzed by ESCA. In this method of surface elemental analysis, the surface of the specimen was bombarded by a beam of soft X-rays, and the energy spectrum of the electrons ejected from the surface atoms was recorded. Since the energy of the photoelectrons is directly related to the energy levels of the electron shells surrounding the nucleus of the atom, this relation is the basis for identifying surface atoms. The spectrum obtained from this analysis revealed the presence of carbon, nitrogen, silicon, aluminum, potassium, and sodium on the slickenside surface.

A quantitative chemical analysis of the slickensides from the Isabella mine was performed using a Materials Analysis Company (MAC) electron probe microanalyzer operated at 15 kV.

## CHARACTERIZATION OF THE SLICKENSIDES AFTER REMOVAL FROM THE ROCK

The in situ analysis of the slickensides did not yield enough data to adequately characterize their chemistry and to compare the composition of the slickensides with that of the surrounding roof rock. Hence the slickensides had to be removed from the rock so that the composition of the slickensides could be determined by conventional methods of analyzing bulk samples.

After being carefully removed with a scalpel, the slickenside was subjected to X-ray diffraction analysis to identify the minerals present. Chemical compositions were assigned to these minerals according to the standard analyses listed in Deere, Howie, and Zussman (1). These compositions were compared with the chemical composition of the slickensides as determined by standard methods of quantitative chemical analysis (table 1). This comparison was performed with a least squares computer method developed by Wright and Doherty (5). By indicating the relative abundance of the minerals in the slickensides, the results of this computation served as the basis for comparing slickensides from different mines.

TABLE 1. - Analytical methods used to perform quantitative chemical analyses

| Oxide | Method |
|---|---|
| $TiO_2$ .......... | Calorimetric. |
| $SiO_2$ .......... | Gravimetric. |
| $Al_2O_3$ ......... | Atomic absorption. |
| CaO........... | Atomic absorption. |
| $K_2O$ ........... | Atomic absorption. |
| $Na_2O$ .......... | Atomic absorption. |
| S............. | Combustion. |
| C............. | Combustion. |
| $H_2O^+$ .......... | Weight loss after heating for 4 hr at 500° C. |
| $H_2O^-$ .......... | Weight loss after heating for 4 hr at 105° C. |

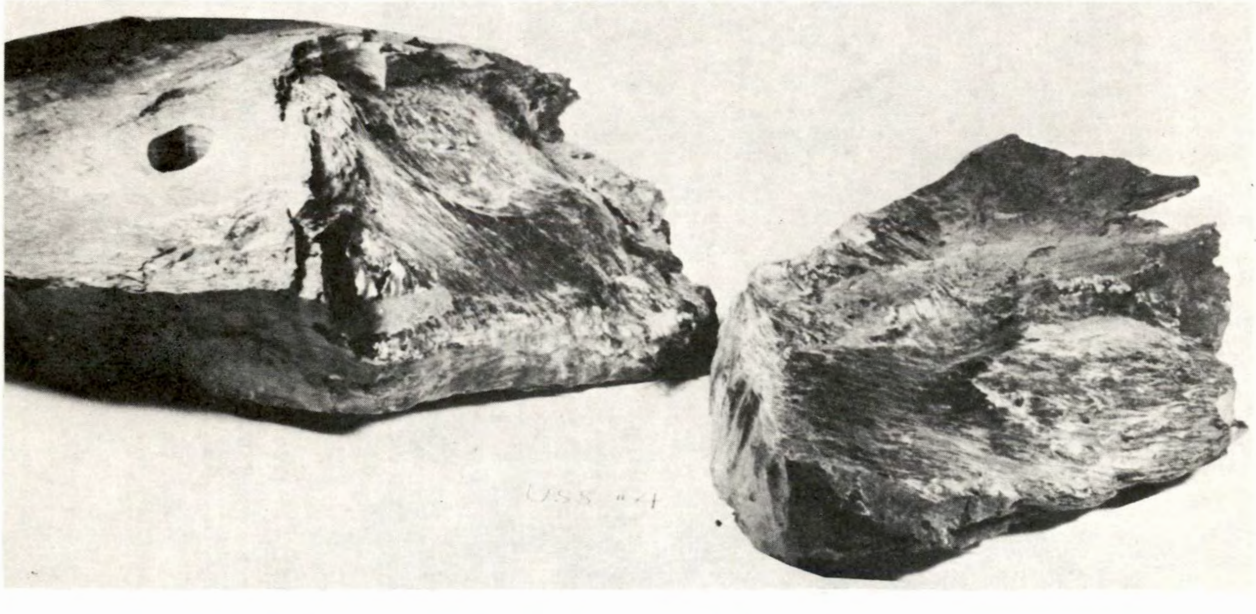

FIGURE 2. - Shale broken at a slickensided interface.

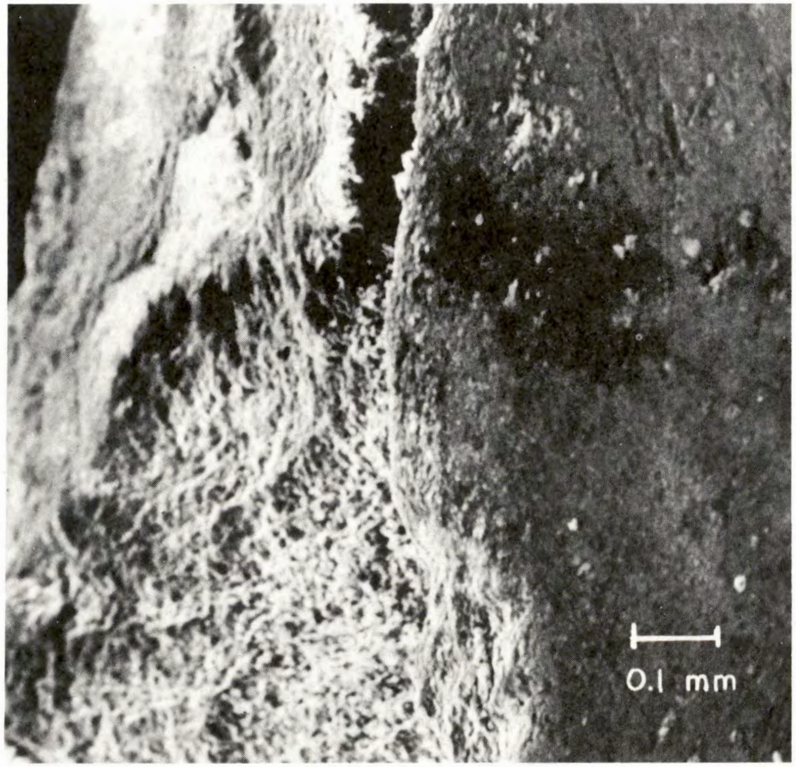

FIGURE 3. - Scanning electron micrograph showing surface morphology of the slickensides (right) and the neighboring shale (left).

FIGURE 4. - Grooves in a slickensided surface.

All samples except the slickensides from the Isabella mine were analyzed using the methods tabulated in table 1. Because of the small amount of slickenside material available from the Isabella mine, a quantitative chemical analysis of this material was performed using MAC electron probe microanalyzer operated at 15 kV.

An infrared spectogram was obtained from each sample with a Perkin-Elmer model 621 infrared spectorphotometer using the potassium bromide pellet technique. These spectra showed absorption frequencies characteristic of the hydroxide ions in the clay minerals as well as those of the $CH_2$ and $CH_3$ from hydrocarbons present in the material.

## EXPERIMENTAL RESULTS

### In Situ Measurements

Matched pieces of mine shale broken at a slickensided surface are shown in figure 2. Note that both surfaces of the matched pieces are glossy, an indication that the weakness of the rock in this region results either from the absence of bonding or from the presence of very weak bonding between two separate slickensided surfaces.

Scanning electron microscopy of such slickensided surfaces revealed that the texture of the slickensides is much different from that of the bulk of the shale (fig. 3), and showed grooves in some of the slickensides (fig. 4).

The differences in texture might be a reflection of crushing caused by the motion of the shale on one side of the slickensided surface with respect to the adjacent shale. The presence of grooves in the slickensides is also evidence of differential motion across the slickensided surface.

Thichness measurements of the in situ film with a profilometer indicate that the films are about 3μm thick. A more precise measurement was not feasible because difficulties were encountered in identifying the lower boundary of the film.

Reflection diffraction patterns of the slickensides contained rings characteristic of polycrystalline material. Further examination of the slickensides with polarized light in a petrographic microscope revealed the presence of birefringent material intermixed with a phase which remains extinguished upon rotation of the stage. Thus there may be an amorphous phase intermixed with fine grained crystalline material.

### Measurement of Slickenside Concentrate

In order to obtain a more quantitative characterization of the film, experiments were performed on the slickensided material removed from the fracture surface by scraping with a scalpel. X-ray powder diffraction patterns were taken of the slickensides and compared with similar diffraction patterns of the shale adjacent to the slickensided surfaces. The interplanar spacings derived from these patterns (designated as d(A) in table 2) demonstrate that the slickensides contain crystalline material and that the slickensides contain the same minerals as the adjacent shale (table 3).

TABLE 2. - Interplanar spacings and relative intensities read from X-ray diffraction patterns of slickensides and the substrate covered by slickensides from shales from five coal mines

| Isabella slickensides | | Isabella substrate | | Gateway slickensides | | Gateway shale | | Armco No. 7 slickensides | | Armco No. 7 shale | | U. S. Steel No. 4 mine slickensides | | U. S. Steel No. 4 mine shale | | Matthews slickensides | | Matthews substrate | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] | d(A)[1] | I[2] |
| 10.12 | 1 | 9.99 | 10 | 10.00 | 1 | 7.20 | 2 | 10.0 | 1 | 14.4 | 10 | 14.2 | 1 | 14.10 | | 10.0 | 10 | 14.2 | 5 |
| 7.22 | 3 | 7.17 | 8 | 7.15 | 1 | 4.55 | 1 | 7.1 | 1 | 10.05 | 49 | 10.0 | 5 | 10.10 | | 7.16 | 7 | 10.0 | 30 |
| 5.11 | 1 | 5.00 | 4 | 4.45 | 10 | 4.25 | 10 | 4.49 | 10 | 7.08 | 30 | 7.1 | 3 | 7.08 | | 5.0 | 1 | 7.1 | 20 |
| 4.53 | 15 | 4.47 | 20 | 4.25 | 20 | 3.55 | 1 | 4.25 | 10 | 4.94 | 17 | 5.0 | 1 | 4.98 | | 4.48 | 25 | 4.99 | 7 |
| 4.31 | 18 | 4.26 | 40 | 3.55 | 1 | 3.32 | 100 | 3.50 | 1 | 4.72 | 7 | 4.50 | 10 | 4.71 | | 4.27 | 30 | 4.70 | 1 |
| 3.93 | 1 | 4.14 | 2 | 3.32 | 100 | 2.70 | 1 | 3.32 | 100 | 4.48 | 30 | 4.28 | 5 | 4.47 | | 3.60 | 20 | 4.48 | 27 |
| 3.60 | 5 | 4.10 | 1 | 2.97 | 1 | 2.55 | 2 | 3.20 | 1 | 4.28 | 50 | 3.90 | 1 | 4.26 | | 3.52 | 3 | 4.25 | 47 |
| 3.53 | 5 | 3.89 | 1 | 2.70 | 1 | 2.45 | 1 | 2.88 | 10 | 4.11 | 3 | 3.73 | 1 | 3.88 | | 3.35 | 100 | 3.87 | 1 |
| 3.36 | 100 | 3.85 | 1 | 2.55 | 20 | 2.33 | 1 | 2.58 | 1 | 3.88 | 8 | 3.50 | 2 | 3.74 | | 3.20 | 2 | 3.72 | 2 |
| 3.02 | 1 | 3.72 | 1 | 2.45 | 5 | 2.27 | 1 | 2.56 | 10 | 3.74 | 8 | 3.32 | 100 | 3.53 | | 2.95 | 5 | 3.53 | 15 |
| 2.88 | 1 | 3.58 | 3 | 2.28 | 5 | 2.24 | 1 | 2.45 | 5 | 3.54 | 23 | 3.21 | 2 | 3.34 | | 2.86 | 1 | 3.34 | 100 |
| 2.58 | 15 | 3.52 | 4 | 2.24 | 2 | 2.13 | 1 | 2.37 | 1 | 3.51 | 18 | 2.87 | 1 | 3.20 | | 2.80 | 40 | 2.98 | 10 |
| 2.47 | 7 | 3.35 | 100 | 2.13 | 5 | 1.98 | 1 | 2.27 | 2 | 3.34 | 100 | 2.80 | 1 | 2.99 | | 2.57 | 25 | 2.85 | 5 |
| 2.40 | 1 | 3.21 | 2 | 1.99 | 2 | | | 2.23 | 1 | 3.20 | 20 | 2.60 | 1 | 2.86 | | 2.46 | 15 | 2.79 | 15 |
| 2.36 | 1 | 2.99 | 2 | | | | | | | 3.00 | 16 | 2.57 | 20 | 2.82 | | 2.35 | 90 | 2.56 | 30 |
| 2.30 | 4 | 2.87 | 2 | | | | | | | 2.87 | 8 | 2.46 | 2 | 2.79 | | 2.285 | 15 | 2.45 | 15 |
| 2.25 | 3 | 2.56 | 15 | | | | | | | 2.80 | 5 | 2.39 | 1 | 2.58 | | 2.13 | 15 | 2.38 | 10 |
| 2.14 | 5 | 2.50 | 1 | | | | | | | 2.59 | 20 | 2.28 | 2 | 2.56 | | 2.03 | 100 | 2.35 | 30 |
| 2.01 | 1 | 2.46 | 10 | | | | | | | 2.57 | 38 | 2.24 | 1 | 2.50 | | 1.975 | 10 | 2.28 | 15 |
| 1.99 | 2 | 2.39 | 1 | | | | | | | 2.49 | 2 | 2.13 | 10 | 2.45 | | 1.815 | 20 | 2.125 | 15 |
| 1.83 | 7 | 2.34 | 1 | | | | | | | 2.46 | 22 | 2.00 | 1 | 2.39 | | 1.735 | 10 | 2.035 | 40 |
| 1.68 | 2 | 2.28 | 10 | | | | | | | 2.39 | 10 | 1.98 | 1 | 2.28 | | 1.671 | 3 | 1.99 | 10 |
| | | 2.24 | 5 | | | | | | | 2.38 | 10 | | | 2.24 | | 1.542 | 15 | 1.96 | 10 |
| | | 2.13 | 10 | | | | | | | 2.34 | 40 | | | 2.15 | | 1.505 | 10 | 1.815 | 20 |
| | | 2.00 | 1 | | | | | | | 2.29 | 12 | | | 2.14 | | 1.435 | 70 | 1.541 | 20 |
| | | 1.98 | 4 | | | | | | | 2.24 | 7 | | | | | 1.383 | 5 | 1.502 | 15 |
| | | 1.95 | 1 | | | | | | | | | | | | | | | 1.454 | 1 |
| | | | | | | | | | | | | | | | | | | 1.434 | 60 |
| 1.66 | 1 | 1.85 | 20 | 1.82 | 10 | 1.82 | 10 | 2.13 | 25 | 2.18 | 1 | 1.72 | 10 | | | 1.376 | 7 | 1.383 | 10 |
| 1.55 | 7 | 1.80 | 2 | 1.67 | 1 | 1.67 | 1 | 2.03 | 45 | 2.13 | 1 | 1.67 | 1 | | | 1.371 | 15 | 1.375 | 10 |
| 1.51 | 2 | 1.67 | 1 | 1.54 | 5 | 1.54 | 5 | 1.998 | 10 | 2.02 | 1 | 1.54 | 10 | | | 1.286 | 1 | 1.372 | 10 |
| 1.49 | 2 | 1.66 | 1 | 1.49 | 1 | 1.49 | 1 | 1.983 | 8 | 1.98 | 1 | 1.50 | 2 | | | 1.256 | 1 | 1.288 | 2 |
| 1.46 | 1 | 1.61 | 1 | 1.45 | 1 | 1.45 | 1 | 1.821 | 1 | 1.82 | 5 | 1.45 | 1 | | | 1.225 | 3 | 1.256 | 2 |
| 1.39 | 5 | 1.54 | 15 | 1.38 | 1 | 1.38 | 1 | 1.673 | 5 | 1.67 | 1 | 1.38 | 10 | | | 1.198 | 1 | 1.222 | 80 |
| 1.38 | 5 | 1.50 | 1 | 1.37 | 1 | 1.37 | 1 | 1.661 | 4 | 1.54 | 20 | 1.37 | 10 | | | 1.180 | 1 | 1.200 | 2 |
| 1.37 | 3 | 1.45 | 2 | 1.29 | 1 | 1.29 | 1 | 1.647 | 4 | 1.50 | 1 | 1.29 | 1 | | | 1.170 | 1 | 1.180 | 2 |
| 1.29 | 1 | 1.42 | 1 | 1.26 | 1 | 1.26 | 1 | 1.544 | 17 | 1.45 | 1 | 1.23 | 1 | | | | | 1.153 | 1 |
| 1.27 | 1 | 1.38 | 4 | 1.23 | 1 | 1.23 | 1 | 1.503 | 10 | 1.38 | 10 | 1.20 | 2 | | | | | | |
| 1.23 | 1 | 1.37 | 8 | 1.20 | 1 | 1.20 | 1 | 1.450 | 2 | 1.370 | 15 | 1.19 | 1 | | | | | | |
| 1.20 | 1 | 1.29 | 3 | 1.17 | 1 | 1.17 | 1 | 1.435 | 62 | 1.372 | | 1.183 | 3 | | | | | | |
| 1.18 | 1 | | | | | | | 1.384 | 10 | 1.286 | 1 | 1.18 | 3 | | | | | | |
| | | | | | | | | 1.377 | 12 | 1.255 | 1 | | | | | | | | |
| | | | | | | | | | | 1.254 | 1 | | | | | | | | |
| | | | | | | | | | | 1.199 | 1 | | | | | | | | |
| | | | | | | | | | | 1.197 | 1 | | | | | | | | |
| | | | | | | | | | | 1.174 | 1 | | | | | | | | |
| | | | | | | | | | | 1.170 | 1 | | | | | | | | |
| | | | | | | | | | | 1.168 | 1 | | | | | | | | |

[1] The symbol d(A) designates the spacing in angstroms between lattice planes.

[2] The symbol I designates the relative intensities of peaks in the diffraction pattern, the most intense peak being given an intensity of 100.

Tables 2 through 5 compare analyses of slickensides with analyses of the associated shale. In some cases it was possible to obtain a sample of the shale comprising the surface covered by the slickensides. These samples are designated as "substrate" in tables 2 through 5. The term "shale" in these tables refers to samples of the rock containing the analyzed slickensides but not necessarily immediately adjacent to the slickensided surface.

A quantitative chemical analysis was performed on various selected areas of the shale. These analyses (table 4) showed that in general the chemistry of the slickensides was similar to that of the surrounding shale.

In an attempt to quantify the relative abundance of the minerals in the sample, a computer program was used to fit the chemical analyses (table 4) to standard analyses given in a book by Deere, Howie, and Zussman (1) for the minerals identified in the X-ray diffraction analysis.

The results of this analysis (table 5) are meant to give an approximate rather than an absolute determination of the relative abundance of these minerals. Since no independent method of quantitative compound analysis was available, there was no way to check these results and some error is possible.

One possibility of error is that an uncertainty exists in the selection of the composition for the sheet silicate (illite, kaolinite, and chlorite) standards. These minerals can vary widely in chemical composition. In addition, the X-ray diffraction technique is not capable of detecting small amounts of a mineral in a complex mixture of minerals such as shale. It is possible, therefore, that small concentrations of minerals may escape detection by this technique. In fact the presence of small concentrations of pyrite and calcite had to be assumed in order to account for the CaO and $FeS_2$ reported in the chemical analysis (table 3).

Because of the uncertainties inherent in this calculation it is not certain when the drastic differences in the mineralogical compositions between the slickenside and the adjacent shale from the same mine (for example, the difference of 22.69 percent in the illite content between the Isabella slickenside and the Isabella whole rock) are real or artifacts induced by the computation. The latter is assumed to be the case because table 3 shows no parallel large differences in the chemistry of these samples. All that can be said for certain is that the slickensides contain the same minerals as the surrounding shale and that in general the proportions of the minerals are somewhat similar in these samples.

TABLE 3. - Minerals identified as present in the slickensides and the surrounding shale from the X-ray powder diffraction data listed in table 2

| Mineral | Isabella shale | Isabella slickensides | Armco No. 7 shale | Armco No. 7 substrate | Armco No. 7 slickensides | Matthews shale | Matthews substrate | Matthews slickensides | U. S. Steel No. 4 shale | U. S. Steel No. 4 substrate | U. S. Steel No. 4 slicken sides | Gateway shale | Gateway substrate | Gateway slickensides |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Quartz..... | X | X | X | X | X | X | X | X | X | X | X | X | X | X |
| Illite..... | X | X | X | X | X | X | X | X | X | X | X | X | X | X |
| Kaolinite.. | X | X | X | X | X | X | X | X | X | X | X | X | X | X |
| Chlorite... | | | X | X | X | X | X | X | X | X | X | | | |

X = Present.

TABLE 4. - Chemical composition of coal mine roof shale

| Oxides | Isabella shale, wt-pct | Isabella slickensides wt-pct | Armco No. 7 shale wt-pct | Armco No. 7 substrate, wt-pct | Armco No. 7 slickensides, wt-pct | Matthews shale, wt-pct | Matthews substrate, wt-pct | Matthews slickensides, wt-pct | U. S. Steel No. 4 shale, wt-pct | U. S. Steel No. 4 substrate, wt-pct | U. S. Steel No. 4 slickensides, wt-pct | Gateway shale, wt-pct | Gateway substrate, wt-pct | Gateway slickensides, wt-pct |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $SiO_2$..... | 54.50 | 45.44 | 55.36 | 55.72 | 53.38 | 50.38 | 51.20 | 49.26 | 55.82 | 53.20 | 50.24 | 52.46 | 54.16 | 54.20 |
| $Al_2O_3$.... | 22.47 | 26.00 | 22.29 | 22.29 | 22.49 | 19.62 | 22.59 | 19.28 | 25.87 | 25.01 | 20.16 | 19.95 | 22.26 | 22.47 |
| $Fe_2O_3$.... | 1.60 | 4.34 | 1.23 | 1.01 | 1.74 | .57 | 1.29 | 1.46 | 2.31 | 1.19 | 1.76 | 1.76 | 2.20 | 1.60 |
| FeO...... | 1.44 | 1.44 | 3.35 | 3.22 | 3.31 | 7.22 | 3.47 | 7.19 | 2.95 | 2.62 | 3.46 | .53 | .57 | 1.44 |
| MgO...... | .82 | .96 | 2.13 | 2.11 | 2.20 | 1.55 | 1.51 | 1.50 | 1.85 | 1.79 | 1.92 | .85 | .86 | .82 |
| CaO...... | .84 | .81 | .76 | .75 | .64 | .51 | .53 | .49 | .82 | .93 | .93 | .78 | .70 | .76 |
| $Na_2O$..... | .53 | 1.87 | .31 | .33 | .45 | .32 | .32 | .34 | .29 | .28 | .29 | .49 | .50 | .53 |
| $K_2O$...... | 2.99 | 3.26 | 4.38 | 4.18 | 5.70 | 3.70 | 3.73 | 3.25 | 3.72 | 5.36 | 4.52 | 2.46 | 2.86 | 2.99 |
| $FeS_2$..... | 1.08 | .03 | .05 | .05 | .22 | .10 | .28 | .15 | .08 | .01 | .37 | 2.20 | 1.46 | 1.08 |
| C........ | 4.28 | 9.00 | .52 | .60 | 1.96 | 3.19 | 5.69 | 2.90 | 2.22 | 2.20 | 5.72 | 7.46 | 5.23 | 4.30 |
| $H_2O^+$..... | 9.47 | 9.47 | 3.39 | 3.74 | 4.67 | 8.54 | 10.66 | 9.84 | 5.57 | 5.90 | 7.85 | 12.91 | 9.83 | 9.47 |
| $H_2O^-$..... | 1.26 | 1.26 | .68 | .64 | 1.11 | 1.03 | 1.00 | 1.84 | .79 | .87 | .72 | 1.27 | 1.22 | 1.26 |
| Total.... | 101.28 | 103.88 | 94.48 | 94.88 | 97.87 | 96.73 | 102.27 | 97.50 | 102.29 | 99.36 | 97.94 | 103.12 | 101.85 | 100.92 |

TABLE 5. - Mineralogical composition of coal mine shales

| Mineral | Isabella shale, wt-pct | Isabella slickensides wt-pct | Armco No. 7 shale wt-pct | Armco No. 7 substrate, wt-pct | Armco No. 7 slickensides, wt-pct | Matthews shale, wt-pct | Matthews substrate, wt-pct | Matthews slickensides, wt-pct | U. S. Steel No. 4 shale, wt-pct | U. S. Steel No. 4 substrate, wt-pct | U. S. Steel No. 4 slickensides, wt-pct | Gateway shale, wt-pct | Gateway substrate, wt-pct | Gateway slickensides, wt-pct |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Quartz.... | 13.80 | 15.93 | 11.59 | 13.39 | 2.83 | 13.62 | 9.98 | 13.57 | 6.51 | 3.23 | 7.16 | 15.89 | 12.36 | 13.59 |
| Illite.... | 35.68 | 12.99 | 58.18 | 53.37 | 72.11 | 35.25 | 35.71 | 34.18 | 54.06 | 60.12 | 54.22 | 26.59 | 38.12 | 35.83 |
| Calcite... | .76 | 1.25 | .35 | .40 | - | .22 | .21 | .21 | .39 | .53 | .67 | .79 | .45 | .62 |
| Kaolinite. | 42.85 | 61.54 | 24.79 | 27.45 | 17.74 | 26.35 | 38.13 | 27.25 | 33.34 | 30.90 | 22.87 | 43.50 | 40.76 | 42.99 |
| Chlorite.. | - | - | 8.40 | 8.12 | 7.99 | 18.99 | 9.54 | 20.12 | 6.97 | 6.09 | 9.46 | - | - | - |
| Pyrite.... | 1.06 | - | .07 | .07 | .22 | .10 | .27 | .15 | .08 | .01 | .38 | 2.15 | 1.43 | 1.06 |
| Carbon.... | 4.20 | 8.61 | .56 | .86 | 1.99 | 3.19 | 5.54 | 2.96 | 2.16 | 2.20 | 5.80 | 7.19 | 5.11 | 4.24 |
| Total..... | 98.35 | 100.32 | 103.94 | 103.66 | 102.88 | 97.72 | 99.38 | 98.44 | 103.51 | 103.08 | 100.58 | 96.11 | 98.23 | 98.33 |

## CONCLUSIONS

The data presented above justify the following conclusions:

1. The slickensides are composed of a thin (about 3-μm) film of a glossy material coating the surface of the shale.

2. This film is composed predominately of silicates intermixed with a lesser but significant amount of hydrocarbons.

3. This film contains fine grained crystalline material.

4. The chemical composition of the slickensides is similar to that of the surrounding shale.

The problem of accounting for the weakness of slickensided surfaces has not received the attention from researchers commensurate with the safety problem posed by slickensides in coal mine roof shale. The results reported in this report are a preliminary step in the direction of accounting for weakness at slickensided surfaces. More research in this field could aid in the development of methods for detecting or strengthening slickensided surfaces in coal mine roofs.

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