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REDUCING PREFERRED ORIENTATION IN DIFFRACTOMETER SAMPLES

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INTRODUCTION

The problem of eliminating preferred orientation in powdered samples for diffractometer study was solved by Flörke and Saalfeld (1955) who suspended one gram of powdered material (<60 microns diameter) in 10 ml of a solution consisting of cellulose acetate dissolved and properly thinned in acetone. For clay minerals, which exhibited swelling in the

foregoing solution, a solution of polystyrol in benzol was substituted. Optimum viscosity of solution was obtained for an approximately 2% solution of cellulose acetate in acetone or a 3–4 percent solution of polystyrol in benzol. The powdered material, now suspended in the plastic solution, was then forcibly sprayed from a spray gun such as those used in painting, the spray being directed into a glass box approximately 40×60 cm in size. The tiny droplets of the suspension then congeal in midair and collect within the glass box as tiny, spherical aggregates of grains. The aggregates are then usually fixed in the sample holder with a solution of vaseline in benzol.

Other published methods of reducing preferred orientation usually require a modification of the sample holder plus careful packing of the sample into this holder. The sample is then retained in the holder by a glass plate (Niskanen, 1964) or by a beryllium platelet (Lerz and Kramer, 1966). These plates absorb the diffracted beams and, consequently, Bragg-angle-dependent corrections for the diffracted line intensities are required.

The method for reducing preferred orientation to be described next has the advantage of requiring no additional equipment or modification of existing equipment. The only prior preparation required is purchase of a can of pressurized, aerosol plastic spray—for example, Blair Spray-Clear or Krylon—and of a roll of food wrap—that is, Saran Wrap, Handi-Wrap, *etc.* Description of this easily used technique together with a comparison of results for this technique with those for the Flörke-Saalfeld method follow next.

Suggested New Method

In this alternative method the powdered mineral (<44 microns) is sieved onto the bottom of a glass beaker, preferably leaving no portion of the glass bottom exposed. The powder's surface is next lightly sprayed with plastic from the aerosol spray-can, the can's nozzle being held about eight inches away. The individual droplets of plastic clump the mineral grains into aggregates which, after a few minutes of drying time, can be brushed out of the beaker and sieved to separate the >115 mesh grain-aggregates. A small piece of the plastic food-wrap is stretched taut over the opening in a standard aluminum sample holder and taped in place. This plastic film is then coated with a thin veneer of Duco cement onto which the grain aggregates are immediately sprinkled. After a short drying period, the sample holder is gently tilted to remove any excess aggregates. The sample holder is then inserted into the Norelco goniometer. To compensate for the thickness of the layer of sample atop the holder, cover glass spacers may be inserted at the contact between the holder and the

half-cylinder of metal against which, in Norelco goniometers, this holder is snugly held (by a spring clip). This insures that the sample will be correctly centered on the rotational axis of the goniometer. After X-ray analysis, the aggregate-covered plastic film may be removed from the sample holder and filed for future reference. The glass beaker may be easily cleaned with acetone after each sample preparation.

The food wrap alone gives distinct peaks in the 0–25° (2θ) region for

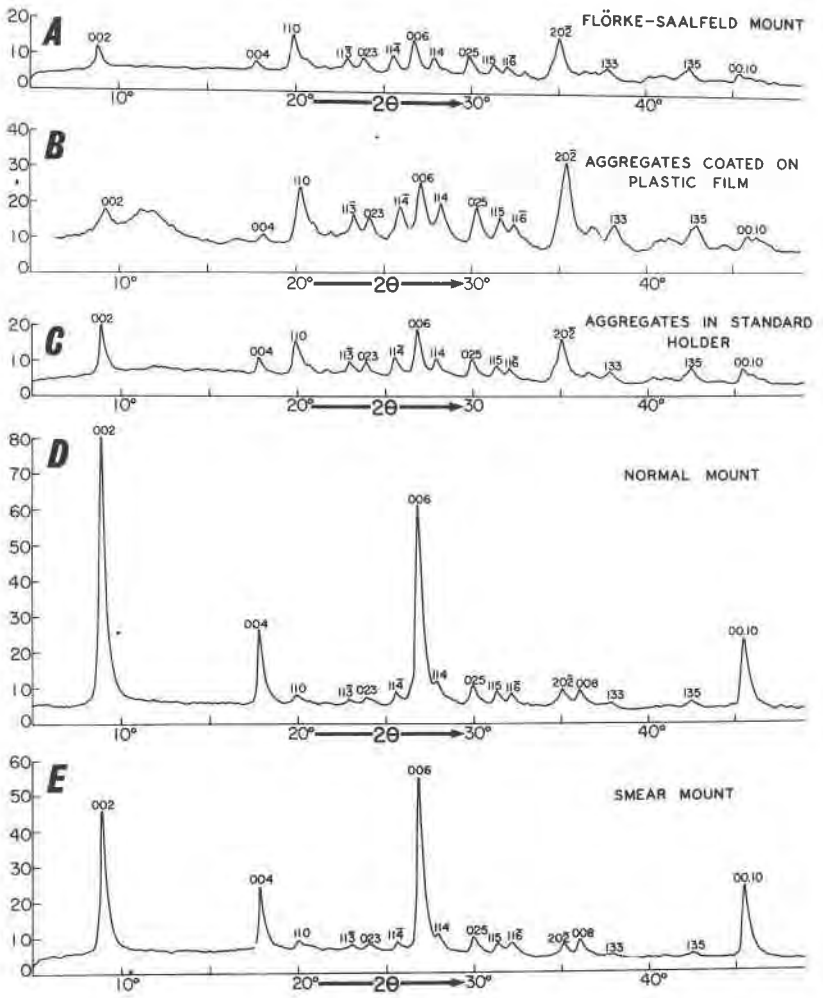

FIG. 1. Diffractograms for muscovite from Middletown, Conn. showing the effect of different methods of sample preparation. In (B) the poorly defined peaks at 11–12° 2θ result from the underlying plastic film.

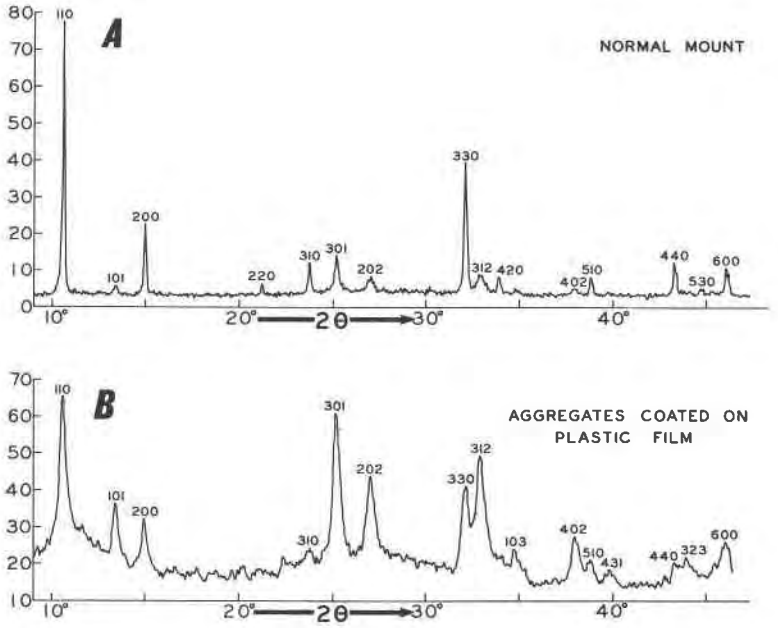

Fig. 2. Diffractograms of synthetic $K_2Pb_4Si_8O_{21}$, a tetragonal material which yields needle-shaped cleavage fragments because of its pronounced {110} cleavage.

CuKα radiation. However, after the wrap is coated with the grain-aggregates, these low angle peaks are greatly supressed by absorption. Background beyond 25° (2θ) is extremely low. If the background in the 0–25° region is found to interfere with a peak which is being investigated, the grain aggregates may be sprinkled directly onto a Duco coated glass slide or packed very loosely into a standard aluminum sample-holder.

Comparison of Results

In view of the well known difficulties of inhibiting preferred orientation in mica samples, a muscovite from Middletown, Connecticut, served to test the method. Diffractograms were obtained for samples of this muscovite prepared by (1) the Flörke-Saalfeld method (Fig. 1A), (2) our grain aggregates sprinkled on plastic food wrap (Fig. 1B), (3) our grain aggregates packed into an aluminum sample-holder (Fig. 1C), (4) the normal technique of placing untreated (<44 micron) powder into the sample holder (Fig. 1D), and (5) a smear-mount of <44 micron powder (Fig. 1E). Degree of preferred orientation can be assessed by comparing the height of the 110 peak to the various basal peaks, particularly 002 and 006. The Flörke-Saalfeld sample and our grain-aggregate sample sprinkled on

plastic wrap are quite similar. For each, the 110 peak falls somewhat above the line joining the tops of the 002 and 006 peaks. For our alternative method using the aluminum sample-holder (Fig. 1C), the 110 peak falls somewhat below this reference line indicating that some degree of preferred orientation has returned, perhaps because of fragmentation of some of the aggregates as they were packed into the holder. For the normal and smear mount preparations (Figs. 1D and E) the relative height of the 110 peak decreases markedly in respect to the basal peaks, thereby betraying considerable increase in the preferred orientation.

The effect of our technique on needle-shaped cleavage fragments was investigated using a synthetic $K_2Pb_4Si_8O_{21}$ with tetragonal symmetry which exhibits perfect {110} cleavage (Gibbs et al., 1962). Note that the hk0 reflections are enhanced, relative to other peaks, by the preferred orientation present in the normal aluminum sample holder mount (Fig. 2A) but considerably suppressed by our grain aggregate mounting technique (Fig. 2B).

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THE EFFECT OF SURFACE PROPERTIES ON REFRACTIVE INDEX DETERMINATION BY THE BREWSTER ANGLE METHOD

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The relationship between the refractive index of a substance and the angle at which light reflected from a polished surface of the substance

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