Effects of Preferred Orientation on X-Ray Diffraction Patterns of Gypsum

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Abstract

Intensities of the stronger reflections *020, 021,* and *041* obtained from X-ray powder photographs of gypsum from pyritic shale and other sources differ from those listed in the Joint Committee on Powder Diffraction Standards files. The differences are shown to be due to the effect of preferred orientation on (010) planes.

Two X-ray diffraction patterns for gypsum are listed in the Joint Committee on Powder Diffraction Standards (JCPDS) files, both shown as highly reliable. Intensities of the stronger reflections, notably *020, 021,* and *041,* measured with a Debye-Scherrer camera from a sample of gypsum taken from black pyritic shale (Penner, Eden, and Grattan-Bellew, 1973) differed significantly from those on JCPDS cards 6-46 and 21-816 (Table 1).

Samples of gypsum from other sources produced diffractograms similar to those of the gypsum taken from the shale (Table 2). Diffractograms obtained by means of a powder diffractometer showed intensities that matched those on JCPDS card 6-46. All samples for the diffractometer were prepared by sedimenting gypsum on a glass slide. Gypsum, when ground, forms either flat, plate-like crystals or acicular crystals, both having well developed (010) faces (Fig. 1). The crystals tend to lie on the (010) faces, giving rise to marked, preferred orientation that results in greatly enhanced intensity of the *020* reflection observed in diffractometer traces.

The intensities shown on JCPDS card 6-46 were also obtained from a diffractometer trace. The usual methods of sample preparation (Klug and Alexander, 1974) were used without success to try to eliminate preferred orientation from the diffractometer mounts. A dilute suspension of gypsum sedimented on a flat piece of polyurethane foam made into a sample holder produced a pattern of intensities essentially free from the effect of preferred orientation, but the intensities were so weak that only the strongest could be observed. When the intensities were increased by the addition of more gypsum, preferred orientation was reintroduced.

Debye-Scherrer photographs were obtained from

TABLE 1. Interplanar Spacings, Intensities, and Indices of Gypsum

-A-			-B-		-C-		
D	I/I_o	HKL	D	I/I_o	D	I/I_o	HKL
7.61	45	020	7.6454	80	7.56	100	020
4.74	4	11$\bar{1}$	4.7606	3	-	-	-
4.28	90	021	4.2820	100	4.27	50	12$\bar{1}$
3.80	8	{130, 040}	3.8018	18	3.79	20	{031, 040}
3.17	4	111	3.1690	15	3.163	4	11$\bar{2}$
3.07	30	041	3.0623	92	3.059	55	14$\bar{1}$
2.871	100	{22$\bar{1}$, 200}	2.8699	59	2.867	25	002
2.788	20	11$\bar{2}$	2.7955	12	2.786	6	21$\bar{1}$
2.684	50	{150, 220}	2.6812	37	2.679	28	{051, 022}
2.595	2	{15$\bar{1}$, 002}	2.58	6	2.591	4	{150, 20$\bar{2}$}
-	-	-	-	-	2.530	<1	060
2.486	20	20$\bar{2}$	2.4998	11	2.495	6	200
2.475	2	13$\bar{2}$	-	-	-	-	-
2.454	6	022	2.4538	9	2.450	4	22$\bar{2}$
2.406	2	24$\bar{1}$	2.4028	4	2.400	4	141
2.220	6	151	2.2125	19	2.216	6	15$\bar{2}$
2.142	2	042	-	-	2.139	2	24$\bar{2}$
2.087	14	24$\bar{2}$	2.0785	30	2.080	10	12$\bar{3}$
2.073	20	{31$\bar{1}$, 15$\bar{2}$}	-	-	2.073	8	{11$\bar{2}$, 25$\bar{1}$}
2.048	4	112	2.0473	-	-	-	-
1.993	2	17$\bar{1}$	1.9918	3	1.990	4	170
1.954	6	31$\bar{2}$	1.9491	2	1.953	2	211
1.900	4	{3$\bar{1}$0, $\bar{2}$60}	1.9009	19	1.898	16	{080, 062}
1.8..	6	241	1.8787	18	1.879	10	14$\bar{3}$
1.865	4	$\bar{1}\bar{1}$3	-	-	1.864	4	31$\bar{2}$
1.812	4	062	1.8104	19	1.812	10	26$\bar{2}$
1.798	6	$\bar{2}\bar{2}$3	-	-	1.796	4	32$\bar{1}$
1.778	4	26$\bar{2}$	1.7758	14	1.778	10	260
-	-	-	-	-	1.711	2	25$\bar{3}$
1.685	2	023	-	-	1.684	2	32$\bar{3}$
1.664	4	24$\bar{3}$	1.6622	8	1.664	4	34$\bar{1}$
1.646	2	261	-	-	1.645	2	16$\bar{3}$
1.622	4	{202, 28$\bar{1}$}	1.6172	12	1.621	6	20$\bar{4}$,ETC.
-	-	-	-	-	1.599	<1	35$\bar{2}$,190
1.587	2	{311, 222}	1.5797	5	1.584	2	22$\bar{4}$,ETC.
1.552	2	40$\bar{2}$	-	-	1.532	2	28$\bar{2}$
1.521	2	42$\bar{2}$,ETC.	-	-	1.522	2	222,13$\bar{4}$
1.440	4	441	-	-	-	-	-
1.434	4	{133, 400}					
1.418	2	20$\bar{4}$					
1.402	2	42$\bar{3}$					

-A- JCPDS card 21-816 (with indices listed on card)
-B- Authors No. 1073
-C- JCPDS card 6-0046 (with indices listed on card)

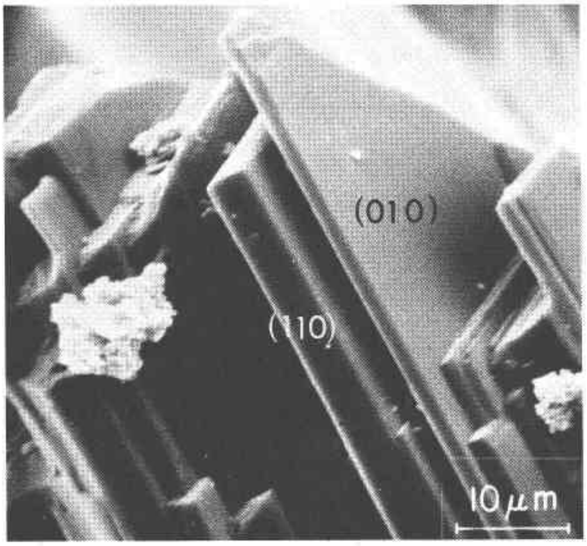
Brackets join indices of reflections not resolved on diffractograms.

TABLE 2. Interplanar Spacings and Intensities of Gypsum

	-A-		-B-		-C-		-D-		-E-	
HKL	D	I/I_o	D	I/I_o	D	I/I_o	D	I/I_o	D	I/I_o
020	7.5879	84	7.6454	80	7.5806	81	7.5891	100	7.576	100
$11\bar{1}$	4.7450	1	4.7606	3	4.7354	2	-	-	-	-
021	4.2741	100	4.2820	100	4.2822	100	4.2716	54	4.276	6
130 040 }	3.7943	14	3.8018	18	3.8181	18	3.7968	23	3.798	22
111	3.1611	3	3.1690	15	3.1640	18	-	-	-	-
041	3.0597	73	3.0623	92	3.0676	73	3.0601	52	3.061	7
$22\bar{1}$	2.8690	44	2.8699	59	2.8744	53	2.8669	22	2.866	1
200					NOT OBSERVED					
$11\bar{2}$	2.7841	7	2.7955	12	2.7955	7	2.7876	3	-	-
150 220 }	2.6799	36	2.6812	37	2.6852	31	2.6880	16	2.68	2
151 002 }	2.5890	3	2.58	6	2.5945	5	2.5971	3	-	-
$20\bar{2}$	2.49	11	2.4998	11	2.4931	11	2.4977	3	-	-
$13\bar{2}$					NOT OBSERVED					
022	2.4507	9	2.4538	9	2.4535	8	2.4546	2	-	-
$24\bar{1}$	2.4082	3	2.4028	4	2.4059	5	2.4062	1	-	-
151	2.2130	12	2.2125	19	2.2151	13	2.2220	8	2.21	1
042	-	-	-	-	2.1348	6	-	-	-	-
$24\bar{2}$	2.0792	30	2.0785	30	2.0809	27	2.0837	10	-	-
221					NOT OBSERVED					
311 $15\bar{2}$ }					NOT OBSERVED					
112	2.0434	3	2.0473	-	2.0407	10	-	-	-	-
$17\bar{1}$	1.9878	1	1.9918	3	1.9938	7	1.9956	2	-	-
312	1.9512	1	1.9491	2	1.9193	7	1.9572	1	-	-
$3\bar{1}0$ 260 }	1.8953	19	1.9009	19	1.9010	15	1.9044	9	1.899	2
080					NOT OBSERVED					
241	1.8733	16	1.8787	18	1.8788	14	1.8851	5	-	-
$\bar{1}\bar{1}3$					NOT OBSERVED					
082	1.8069	17	1.8104	19	1.8104	12	1.8159	6	-	-
$\bar{2}\bar{2}3$					NOT OBSERVED					
262	1.7775	14	1.7758	14	1.7759	11	1.7841	6	-	-
023					NOT OBSERVED					
$24\bar{3}$	1.6614	7	1.6622	8	1.6636	17	1.6676	2	-	-
261	-	-	-	-	-	-	-	-	1.6481	1
202 $28\bar{1}$+ }	1.6200	10	1.6117	12	1.6204	10	1.6234	5	-	-
311 222+ }	-	-	1.5797	5	1.6060	11	1.5859	1	-	-
133	-	-	1.4361	6	1.4381	11	-	-	-	-

-A- PWD photo 1079 (hydrated pottery plaster)
-B- PWD photo 1073 (gypsum from pyritic shale)
-C- PWD photo selenite
-D- Diffractometer trace of selenite on urethane foam sample holder
-E- Diffractometer trace of selenite, smear mount on glass slide

Brackets join reflections which were not resolved.



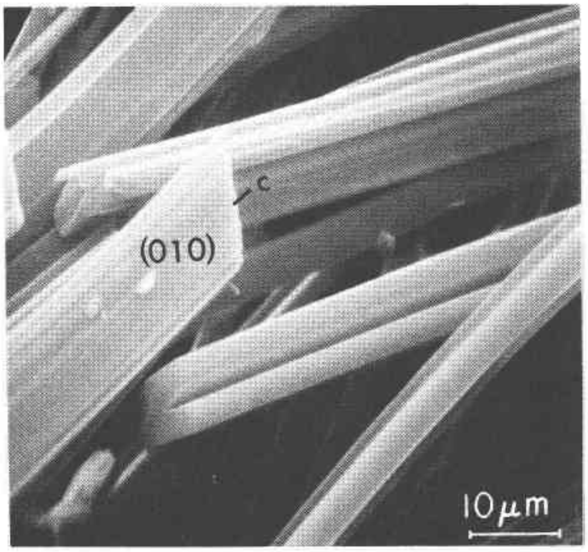


FIG. 1. Gypsum crystals on the surface of fragments of shale, magnified about 2,000 times in the scanning electron microscope. Either tabular morphology (1a) or acicular morphology (1b) is obtained when larger crystals of gypsum are crushed for X-ray powder diffraction examination. It is evident that it would be difficult to avoid preferred orientation in diffractometer mounts owing to the tendency of the crystals to lie on the well developed (010) faces.

samples of gypsum packed into lithium glass capillary tubes; intensities were measured from microdensitometer traces of the photographs. Intensities from the powder photographs matched those obtained from a crystal of gypsum mounted on a single crystal diffractometer, indicating that the X-ray powder photographs were essentially free from the effect of preferred orientation. A similar pattern of intensities was observed on a powder pattern obtained from a single crystal mounted on a Gandolfi camera (Gandolfi, 1967) and by Becherer and Fiedler (1955) using a Debye-Scherrer camera.

The intensities listed on JCPDS card 21-816 are different from those of card 6-46 and from those found by the author (Table 1). The reason for this is not apparent because the geometry of the transmission powder camera and the experimental details used to obtain the intensity data for card 21-816 are not given.

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