Revised X-ray powder diffraction data for polytype 2M₁ of trioctahedral micas

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Literature data for the Miller indices of the reflections in X-ray powder diffraction pattern of the polytype $2M_1$ in trioctahedral micas have been corrected, with new additional data listed.

In their study on synthesis of phlogopite Yoder and Eugster (1954) found the Miller indices of the reflections in the powder diffraction pattern of natural phlogopite $2M_1$ (table 4, p. 172—173, quotation). These data have been cited in several publications (e. g. Bradley and Grim, 1961), and as etalonic data for polytype $2M_1$ of trioctahedral micas they are in usage up now. However, in the mentioned study of Yoder and Eugster, some reflections are indexed wrongly or incompletely.

In Table 1, data for the phlogopite from the study of Yoder and Eugster are compared with powder diffraction data of the biotite polytype $2M_1$ from porphyroblastic gneiss, V. Radetina, Papuk. The measured values of the interplanar spacings, d, of biotite (the averages from the pattern taken in the Guinier — de Wolff film camera and from the counter diffractometer pattern) are listed together with the values $d_{(calc.)}$ calculated from the occurately determined unit cell parameters*. The indexing of the reflections was facilitated using the Weissenberg patterns (zero-, the first- and the second-layer line for the a axis and zero-, the second- and the third-layer line for the b axis) of the biotite polytype $2M_1$.

From Table 1 and the Weissenberg patterns of the biotite polytype $2M_1$ results the following:

In the study of Yoder and Eugster (1954) very strong reflections with $d = 2.624$ Å (indexed as 116) and $d = 1.538$ Å (indexed as 060 and 330) were wrongly indexed. Reflection 116 from the $2M_1$ trioctahedral micas is very weak (see also Zvyagin, 1961) and the correct reflection indices (for $d = 2.624$ Å) are 20$\bar{2}$ and 131. The reflection 330 is extinguished, and the correct indices of the reflection for $d = 1.538$ Å are 060 and 33$\bar{1}$.

* The parameters of the unit cell of the biotite were deduced from the zero-layer rotation patterns around the axes a and b (Popović and Slovenec, 1981).

Table 1. Powder X-ray diffraction data for 2M₁ phlogopite and biotite
Tablica 1. Rendgenografski podaci za prah politipa 2M₁ flogotipa i biotita.

Natural 2M₁ phlogopite Prirodni 2M₁ flogopit (Yoder and Eugster, 1954)			2M₁ biotite (porphyroblastic gneiss, V. Radetina Mt. Papuk) 2M₁ biotit (porfiroblastični gnajs, V. Radetina, Papuk)			
h k l	I	d (Å)	h k l	I (f)	$d_{(obs.)}$ (Å)	$d_{(calc.)}$ (Å)
002	> 100	10.129	002	8	10.062	10.059
004	18	5.056	004			5.030
020 }	19	4.612	020 }	2	4.594	4.634
110 }			110 }			4.620
			11$\bar{1}$ }			4.582
021	7	4.515	021			4.516
112	5	4.079	112			4.079
023	18	3.814	023	< 1	3.82(a)	3.812
			113	< 1	3.669(a)	3.672
11$\bar{4}$	33	3.540	11$\bar{4}$	< 1	3.539	3.544
006	> 100	3.362	006	3	3.354	3.353
114	38	3.283	114			3.277
11$\bar{5}$	9	3.156	11$\bar{5}$			3.159
025	40	3.040	025			3.038
115	8	2.926	115			2.923
11$\bar{6}$	22	2.818	11$\bar{6}$			2.821
			13$\bar{1}$ }	1	2.660(a)	2.665
13$\bar{1}$	22	2.651	200 }			2.665
			20$\bar{2}$ }	10	2.632	2.635
116	> 100	2.624	131 }			2.634
			13$\bar{5}$ }			2.522
008	28	2.522	202 }	< 1	2.516	2.520
			008 }			2.515
			20$\bar{4}$ }	6	2.445	2.448
133	40	2.439	133 }			2.445
117	16	2.361	117			2.359
			040 }			2.317
220 }	9	2.304	22$\bar{1}$ }	1	2.304	2.315
040 }			220 }			2.310
			041 }			2.302
			221 }			2.275
13$\bar{5}$	9	2.270	13$\bar{5}$ }	1	2.275	2.274
			204 }			2.271
			20$\bar{6}$ }	3	2.186	2.184
135	45	2.180	135 }			2.181
224	21	2.039	224			2.039
0.0.10	66	2.017	0.0.10	1	2.012	2.012
			137 }	1	2.003	2.003
137	20	2.000	206 }			2.000
			20$\bar{8}$ }	< 1	1.918	1.917
137	5	1.914	137 }			1.914

h k l	I	d (Å)	h k l	I (f)	$d_{(obs.)}$ (Å)	$d_{(calc.)}$ (Å)
			$1\bar{3}9$ ⎫			1.754
139	3	1.751	208 ⎬		1.749(b)	1.751
			150 ⎭			1.751
			$24\bar{1}$			1.751
227	5	1.737	227			1.735
			153 ⎫			1.682
153	47	1.677	$2.0.\bar{1}0$ ⎬	1	1.679	1.680
			139 ⎭			1.678
060 ⎫	50	1.538	060 ⎫	7	1.546	1.545
330 ⎭			$33\bar{1}$ ⎭			1.545
			331 ⎫			1.532
062	10	1.521	062 ⎭	2	1.529	1.527
			$1.3.\bar{1}3$ ⎫			1.366
			2.0.12 ⎭		1.368(b)	1.364
			$40\bar{2}$ ⎫			1.337
			260 ⎭	2	1.336(a)	1.336
			$33\bar{9}$ ⎫			1.317
			1.3.13 ⎭	1	1.316(a)	1.314
			$26\bar{4}$ ⎫			1.306
			402 ⎭	1	1.306(a)	1.305
			$40\bar{6}$ ⎫			1.278
		.	264 ⎭	< 1	1.277(a)	1.277

I(f) — the relative intensities as observed in the film powder pattern (estimated visually)
(a) — observed only in the film powder pattern
(b) — observed only in the counter diffractometer powder pattern
$d_{(calc.)}$ — calculated from the accurate values of the unit cell parameters:
    $a = 5.351$ (1), $b = 9.268$ (1), $c = 20.201$ (2) Å, $\beta = 95.188$ (2)°
I(f) — relativni intenziteti kako su opaženi na filmskoj difrakcijskoj slici (određeni vizuelno)
(a) — opaženo samo na filmskoj difrakcijskoj slici
(b) — opaženo samo na difrakcijskoj slici dobivenoj s brojačem
$d_{(calc.)}$ — izračunat iz točnih vrijednosti parametara jedinične ćelije:
    $a = 5.351$ (1), $b = 9.268$ (1), $c = 20.201$ (2) Å, $\beta = 95.188$ (2)°

The reflections 220 and 040 ($d = 2.304$ Å) and somewhat stronger reflections $22\bar{1}$ and 041 are at very close Bragg angles Θ. Therefore, the resultant diffraction line consits, besides the reflections 220 and 040, also of the reflections $22\bar{1}$ and 041.

Indices 153 are attributed to the very strong line with $d = 1.677$ Å. However, the intensity of reflection 153 is very low. Tle diffraction line with $d = 1.677$ Å consists, besides the reflection 153, also of stronger reflections $2.0.\bar{1}0$ and 139.

Finally, a number of stronger lines are indexed as $13l$, although these linies also consist of very strong reflections $20l$.

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Difrakcijski podaci za politip 2M₁ trioktaedrijskih tinjaca (korekcija i dopuna literaturnih podataka)

D. Slovenec

U radu o sintezi flogotipa Yoder i Eugster (1954) proveli su i indiciranje refleksa na difrakcijskoj slici praha prirodnog $2M_1$ flogotipa (tablica 4, str. 172—173 cit. rada). Ovi podaci citirani su u nekoliko publikacija (npr. Bradley i Grim, 1961), a kao etalonski podaci za politip $2M_1$ trioktaedrijskih tinjaca koriste se još i danas. Međutim u spomenutom radu Yodera i Eugstera neki refleksi indicirani su pogrešno ili nepotpuno.

U tablici 1 uspoređeni su podaci za flogopit iz rada Yodera i Eugstera s podacima za prah politipa $2M_1$ biotita iz porfiroblastičnog gnjasa, V. Radetina, Papuk. Navedene su izmjerene vrijednosti međumrežnih razmaka d (obs.) biotita (prosjeci sa snimka dobivenog u kameri po Guinieru i de Wolffu i brojačke difrakcijske slike) i vrijednosti d (calc.) izračunate iz točno izmjerenih parametara jedinične ćelije.* Kod indiciranja refleksa upotrijebljeni su i snimci po Weissenbergu (nulta, prva i druga slojna linija oko osi a i nulta, prva, druga i treća slojna linija oko osi b) politipa $2M_1$ biotita. Iz tablice 1 i snimaka po Weissenbergu politipa $2M_1$ biotita izlazi slijedeće.

U radu Yodera i Eugstera (1954) pogrešno su indicirani vrlo jaki refleksi s $d = 2,624$ Å (kao 116) i $d = 1,538$ Å (kao 060 i 330). Refleks 116 kod $2M_1$ trioktaedrijskih tinjaca vrlo je slab (vidi i Zvjagin, 1961). Ispravni indeksi refleksa za $d = 2,624$ Å su 20$\bar{2}$ i 131. Refleks 330 je pogašen, a ispravni indeksi refleksa za $d = 1,538$ Å su 060 i 33$\bar{1}$.

Refleksi 220 i 040 ($d = 2,304$ Å) i nešto jači refleksi 22$\bar{1}$ i 041 nalaze se na vrlo bliskim kutnim položajima Θ.* Stoga rezultantnu difrakcijsku liniju, osim prva dva, tvore i refleksi 22$\bar{1}$ i 041.

Vrlo jakoj liniji s $d = 1,677$ Å pripisani su indeksi 153. Međutim, intenzitet refleksa 153 vrlo je malen. Spomenutu liniju uz refleks 153 tvore i jači refleksi 2.0.$\overline{10}$ i 139.

Konačno, nizu jačih linija pripisani su samo indeksi refleksa 13l, iako te linije tvore i vrlo jaki refleksi 20l.

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* Parametri jedinične ćelije izvedeni su iz nultih slojnih linija difrakcijskih slika rotacije monokristala biotita oko osi a i osi b (Popović i Slovenec, 1981).

* Θ = Braggov kut.