

The effect of absorption on X-ray diffraction Weissenberg patterns of epitactically overgrown biotite polytypes

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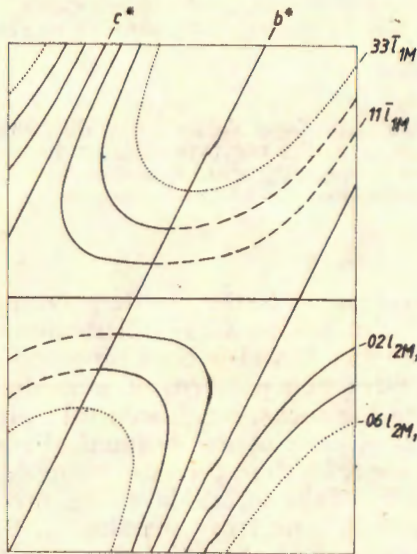
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Characteristic effects in X-ray Weissenberg diffraction patterns caused by epitactic overgrowth of biotite polytypes are studied and explained in terms of the absorption of X-rays, diffracted in one of the individuals, when passing through the other individual.

In the course of investigation of the biotite polytypes in the rocks from the Papuk Mountain by means of X-ray diffraction the epitactic overgrowth of various polytypes has also been observed (Slovenec and Popović, 1981a). Diffraction patterns of individual single crystal-like flakes, peeled out from the rocks, have been taken in a Weissenberg goniometer. The patterns of most of the epitactically overgrown samples show an unusual appearance. A characteristic example is shown in Plate I, Fig. 1: that is the pattern of the second-layer line around one of the 9.2 Å axes of a biotite sample (biotite from metabasite, Brzaja), which represents an epitactic overgrowth of the polytype with seventeen single-mica layers in the unit cell and the polytype 1M. A section of the upper part of this pattern, namely the reciprocal-lattice row parallel to the c^* axis, which lies on the straight-line, enlarged five times, is shown in Plate I, Fig. 2. It is evident that this reciprocal lattice row in the lower part is quite different than in the upper part of the pattern (Plate I, Fig. 1). The lower part contains diffraction maxima $02l$ of the polytype 1M, while the upper part contains corresponding maxima of the polytype with seventeen single-mica layers. Let us consider the cause of this phenomenon in more detail.

Let the polytypes $1M$ and $2M_1$ be overgrown in such a way that the a axis of the polytype $2M_1$ coincides with the direction $[1\bar{1}0]$ of the polytype $1M$, i. e. the polytype $1M$ is rotated around the c^* axis for 60° in relation to the polytype $2M_1$. They form a composite reciprocal lattice in which the following rows are in coincidence: $02l_{2M_1}$ and $11l_{1M}$; $04l_{1M}$ and $22l_{1M}$; $06l_{2M_1}$ and $33l_{1M}$; $20l_{2M_1}$ and $13l_{1M}$ etc. The sample may be transparent for X-rays in such a way that the X-rays diffracted in one of the two polytype individuals are little absorbed when passing through the second individual. Then a composite diffraction pattern is obtained with diffraction maxima of both polytypes spread all over the pattern. In this

case the epitactic overgrowth can be identified according to the criteria suggested by Rieder (1970). On the other hand, the sample may be opaque for the X-rays in such a way, that the X-rays diffracted in one of the two individuals are totally absorbed when passing through the other. Then, in the Weissenberg pattern of the zero-layer line taken around the axis a_{2M_1} , the rows $Ok_{l_{2M_1}}$ and $kk_{l_{1M}}$ are registered in a way schematically shown in Fig. 1. The full lines represent the rows $Ok_{l_{2M_1}}$ with $k \neq 3n$, and the dashed lines rows $kk_{l_{1M}}$ with $k \neq 3n$. The dotted lines represent the rows $0k_{l_{2M_1}}$ and $33l_{1M}$, as the corresponding diffraction maxima of the polytypes $2M_1$ and $1M$ from these rows are at very similar Bragg angles



Text-fig. 1. A schematic representation of X-ray diffraction Weissenberg pattern of the zero-layer line taken around the a axis of the polytype $2M_1$, which is epitactically overgrown with the polytype $1M$, for a case when X-rays, diffracted in one individual, are absorbed totally in the other. The directions $[100]_{2M_1}$ and $[110]_{1M}$ coincide.

Sl. 1. Shematski prikaz rendgenske difrakcijske slike po Weissenbergu nulte slojne linije oko osi a politipa $2M_1$, sraštenog s politipom $1M$ za slučaj kad su zrake, difraktirane u jednom individuumu pri prolazu kroz drugi potpuno apsorbirane. Smjerovi $[100]_{2M_1}$ i $[110]_{1M}$ se podudaraju.

Θ (Table 1). In the pattern of the second-layer line taken around the axis b_{2M_1} , the two coinciding reciprocal lattice rows parallel to c^* , lying on the straight line, are $02l_{2M_1}$ (in one part of the pattern) and $11l_{1M}$ (in the other part of the pattern).

Let another example support the above discussion. Plate II, Fig. 1 shows the pattern of the second-layer line taken around one of the 9.2 \AA axes of a biotite sample (from porphyritic quartz diorite, Pakra). The reciprocal lattice row parallel to the c^* axis, which lies on the straight line, conta-

Table 1. Bragg angles of diffraction maxima 06l of the biotite polytype 2M₁ and the maxima 33l of the biotite polytype 1M for CuK α_1 radiation (biotite from porphyritic quartz diorite, Pakra, Mt. Papuk)*.

Tablica 1. Braggovi kutovi refleksa 06l politipa 2M₁ biotita i refleksa 33l politipa 1M biotita za CuK α_1 zračenje (biotit iz porfiroidnog kvarcdiorita, Pakra, Papuk)*.

2M ₁		1M			
06l	θ (°) CuK α_1	33 \bar{l}	θ (°) CuK α_1	33l	θ (°) CuK α_1
060	29.972	33 $\bar{1}$	29.984		
062	30.358	33 $\bar{2}$	30.361	330	30.380
064	31.499	33 $\bar{3}$	31.492	331	31.529
066	33.353	33 $\bar{4}$	33.333	332	33.390
068	35.865	33 $\bar{5}$	35.839	333	35.908

* The Bragg angles θ of these maxima are calculated from accurate values of unit-cell parameters determined previously (Slovenec and Popović, 1981b).

* Braggovi kutovi θ navedenih refleksa izračunati su iz točnih vrijednosti parametara jedinične ćelije određenih ranije (Slovenec i Popović, 1981b).

ins the diffraction maxima from an eight single-mica layer polytype (the upper part of the pattern; also in Plate II, Fig. 2) and the maxima 02l of the polytype 1M (the lower part of the pattern). The same sample has been carefully cleft in two pieces. Plate II, Fig. 3 shows the diffraction pattern from one of the two pieces, taken around the same 9.2 Å axis, i. e. around the axis *b* of the polytype 1M. The straight line row parallel to the *c** axis contains only the diffraction maxima 02l of the polytype 1M.

The above discussion has been related to two limiting cases of absorption of diffracted X-rays in the sample: first, the rays diffracted in one individual are little absorbed in the other, and second, when they are absorbed totally. However, depending on chemical composition and the sample volume, some samples are intermediate cases. It happens that intense maxima, diffracted in one individual, also pass through the other and are registered in the pattern, while the weaker maxima are absorbed. Moreover, if the number of the intergrown individuals is greater than two, the diffraction pattern may become so complex, that it is not possible to identify the phenomenon of the epitactic overgrowth.

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Utjecaj apsorpcije na izgled rendgenskih difrakcijskih slika po Weissenbergu uzoraka epitaksijskog rasta politipova biotita

D. Slovenec

Prilikom istraživanja politipova biotita iz stijena Papuka opažene su i pojave epitaksijskog rasta različitih politipova (Slovenec i Popović, 1981a). Istraživanje je vršeno uz primjenu rendgenske difrakcije na monokristalu. Difrakcijske slike individualnih listića (kalotina) snimljene su pomoću goniometra po Weissenbergu. Difrakcijske slike većine uzoraka epitaksijskog rasta imaju neobičan izgled. Jedan od karakterističnih snimaka je snimak druge slojne linije oko jedne od osi 9,2 Å uzorka biotita (biotit iz metabazita, Brzaja) koji predstavlja epitaksijski rast politipova sa sedamnaest paketa u jediničnoj ćeliji i politipova 1M (tab. I, sl. 1). Dio tog snimka s gornjim krakom niza paralelnog osi c^* recipročne rešetke (niz registriran na pravcu), povećan pet puta, prikazan je na sl. 2, tab. I. Vidi se iz sl. 1 i 2, tab. I, da su dva kraka niza paralelnog osi c^* bitno različita. Na donjem kraku su refleksi 02l politipova 1M, a na gornjem kraku su refleksi politipova sa sedamnaest paketa u jediničnoj ćeliji. Razmotrimo uzrok ove »neobične« pojave.

Pretpostavimo da su politipovi 1M i 2M₁ srašteni tako da se os a politipova 2M₁ podudara sa smjerom [110] politipova 1M, tj. politip 1M zakrenut je oko osi c^* za 60° u odnosu na politip 2M₁. U složenoj recipročnoj rešetci podudarit će se npr. nizovi: 02l_{2M₁} i 11l_{1M}; 04l_{2M₁} i 22l_{1M}; 06l_{2M₁} i 33l_{1M}; 20l_{2M₁} i 13l_{1M} itd. Ako je uzorak prozračan za rendgenske zrake toliko da su rendgenske zrake, difraktirane u jednom individuumu, pri prolazu kroz drugi malo apsorbirane, dobit ćemo »normalnu« složenu difrakcijsku sliku s efektima oba individuumu po cijeloj difrakcijskoj slici. U tom slučaju epitaksijski rast može se identificirati prema kriterijima koje je predložio Rieder (1970). Međutim, ako je uzorak neprozračan za rendgenske zrake toliko da su rendgenske zrake, difraktirane u jednom individuumu pri prolazu kroz drugi potpuno apsorbirane, na snimku po Weissenbergu nulte slojne linije oko osi a_{2M₁} nizovi Okl_{2M₁} i kkl_{1M} bit će registrirani na način shematski prikazan na sl. 1. Na sl. 1 punom linijom označeni su nizovi Okl_{2M₁} s $k \neq 3n$, a isprekidanom linijom nizovi kkl_{1M} s $k \neq 3n$. Točkastom linijom označeni su nizovi 06l_{2M₁} i 33l_{1M}, jer se odgovarajući difrakcijski maksimumi politipova 2M₁ i 1M iz ovih nizova nalaze na gotovo istim kutnim položajima θ (tablica 1). Na snimku druge slojne linije oko osi b_{2M₁}, na jednom kraku niza paralelnom osi c^* (koji leži na pravcu) nalaziti će se refleksi 02l_{2M₁}, a na drugom refleksi 11l_{1M}.

Gore izloženo potkrijepljuje sljedeći primjer. Na tab. II, sl. 1, prikazan je niz paralelan osi c^* , koji leži na pravcu, registriran na snimku druge slojne linije oko jedne od osi 9,2 Å uzorka biotita iz porfiroidnog kvarcdiorita, Pakra. Na gornjem kraku niza su refleksi osampaketnog politipova (vidi i sl. 2, tab. II). Na donjem kraku niza opažaju se gotovo samo refleksi 02l politipova 1M. Isti listić pažljivo je raskalan i načinjen je snimak druge slojne linije jedne kalotine oko iste osi 9,2 Å, tj. oko osi b politipova 1M; u nizu paralelnom osi c^* koji leži na pravcu (tab. II, sl. 3) vide se samo refleksi 02l politipova 1M.

Razmotrili smo dva krajnja slučaja apsorpcije difraktiranih rendgenskih zraka uzorkom: prvo, da zrake difraktirane u jednom individuumu prolaze i kroz drugi individuum, i drugo, da se potpuno apsorbiraju. Međutim, ovisno o kemijskom sastavu i volumenu individuumu postoje i intermedijarni slučajevi. Naime, zrake većeg intenziteta (refleksi većeg intenziteta) difraktirane u jednom individuumu proći će i kroz drugi a slabije će se apsorbirati. Ako je uz to broj sraštenih individuumu veći od dva, difrakcijska slika uzorka može biti toliko složena da pojavu epitaksijskog rasta nije moguće identificirati.

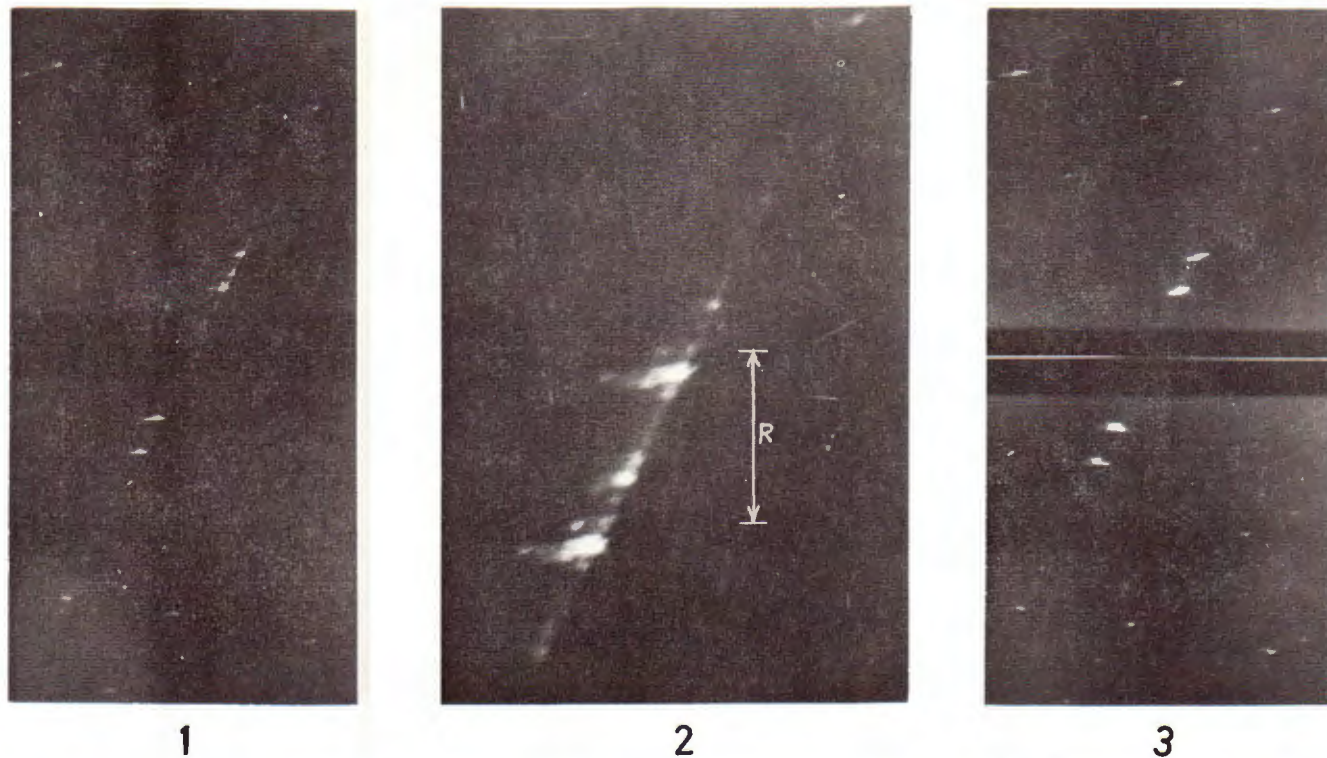


1



2

- Fig. 1. X-ray diffraction Weissenberg pattern of the epitactic overgrowth of the biotite polytype with seventeen single-mica layers in the unit cell and the polytype 1M (biotite from metabasite, Brzaja, Mt. Papuk); the second-layer line, the pattern taken around the b axis of the polytype 1M.
- Sl. 1. Rendgenska difrakcijska slika po Weissenbergu epitaksijskog rasta politipa sa sedamnaest paketa u jediničnoj ćeliji i politipa 1M (biotit iz metabazita, Brzaja, Papuk); druga slojna linija, snimak oko osi b politipa 1M.
- Fig. 2. A section of the upper part of Fig. 1, with the reciprocal-lattice row parallel to c^* , which lies on the straight line, enlarged five times. The distance R corresponds to a single-mica layer of 10 Å of the polytype 1M.
- Sl. 2. Gornji dio snimka na sl. 1 s krakom niza paralelnog osi c^* (niz registriran na pravcu) povećan pet puta. Razmak R odgovara jediničnom paketu od 10 Å politipa 1M.



- Fig. 1. X-ray diffraction Weissenberg pattern of the epitactic overgrowth of the biotite polytype with eight single-mica layers in the unit cell and the polytype 1M (biotite from porphyritic quartz diorite, Pakra, Mt. Papuk); the second-layer line, the pattern taken around the b axis of the polytype 1M.
- Sl. 1. Rendgenska difrakcijska slika po Weissenbergu epitaksijskog rasta politipa s osam paketa u jediničnoj ćeliji i politipa 1M (biotit iz porfiroidnog kvarc-diorita, Pa'kra, Papuk); druga slojna linija, snimak oko osi b politipa 1M).
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- Fig. 3. X-ray diffraction Weissenberg pattern of the second-layer line of the polytype 1M around b axis (one piece of the biotite sample, shown in Fig. 1, obtained by cleaving).
- Sl. 3. Rendgenska difrakcijska slika po Weissenbergu druge slojne linije politipa 1M oko osi b (jedna kalotina listića biotita čiji je snimak prikazan na sl. 1).