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# THE PECULIARITIES OF DIFFRACTION BY NON-CHIRAL NANOTUBES

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**Abstract.** The quantitative theory of diffraction by azimuthally ordered circular nanotubes of any chemical composition is offered. The pseudoorthogonality effect, earlier found out experimentally, is considered. The obtained results are compared with X-ray diffraction patterns of oriented preparations of chrysotile nanotubes.

**Keywords:** circular nanotube, diffraction, electron microdiffraction, layer line, armchair, zigzag, chrysotile.

## 1. Introduction

Structure of circular nanotubes (Fig. 1), being a special case of the chiral ones with a zero chiral angle [1], and diffraction by them were considered by several authors in the 50’s of the last century on the example of chrysotile nanotubes [2-7]. Similar results were obtained, however the most promising appears the approach offered by E. Whittaker. The apparatus proposed in this article is the further development of his approach. After the discovery of carbon nanotubes their two circular structures have been called “armchair” and “zigzag”.

The cylindrical coordinates of the *j*-th atom of *v*-th Bravais cell of *n*-th site circle of *m*-th cylindrical layer of multilayer circular nanotubes are [1]:

$$\begin{cases} r_{mj} = r_m + x_j = r_0 + md + x_j \\ j_{mnj} = \dfrac{b}{r_m} n + e_m + \dfrac{y_j}{r_m} \\ z_{mnj} = an + \Delta z_m + z_j \end{cases}$$

$$m = 0 \div M - 1$$

$$v = 0 \div p_m - 1 \qquad p_m = \frac{2pr_m}{b} \qquad (1)$$

$$n = 0 \div N - 1$$

It differs from those proposed by Whittaker [5] only by the choice of coordinate system. Here *a* and *b* – the lattice parameters of layer, corresponding to longitudinal and circular directions, accordingly; *d* – thickness of layer; $x_j$, $y_j$ and $z_j$ – radial, circular and longitudinal linear coordinates of atom in a cell, accordingly; $\rho_0$ – radius of internal layer; *M* – number of layers; *N* – length of nanotube in terms of *a*. Value $p_m$ – quantity of cells on a circle of *m*-th cylinder – is integer by definition.

The atoms of a unit cell, having identical radial coordinate, belong to one atomic sheet. The origin in the radial direction of the cell is selected on the sheet on which the cell size along the arc of a circle does not change with changes in the radius of the layer. Chrysotile has five sheets, and the origin is selected on the level of octahedral cations of magnesium; in carbon nanotubes there is only one sheet and hence the choice is obvious.

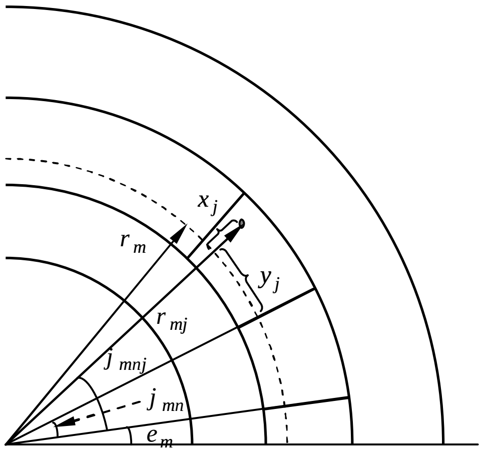


**Fig. 1.** Designations in circular nanotube

The azimuthal angle $\varepsilon_m$ and longitudinal shift $\Delta z_m$ determine an origin of lattice on the *m*-th cylinder; the character of their change determines the azimuthal and longitudinal shift polytype modifications [1] of multiwall circular nanotube, accordingly.

The essential point of Whittaker's model is the assumption that the initial angular phases $\varepsilon_m$ are absolutely random [5], that is the cylindrical lattices of circular (and chiral) nanotubes have azimuthally disordered character. The alternative proposed by the authors of this article is the model of azimuthally ordered circular (and chiral) nanotubes. The authors do not refute a possibility of existence of azimuthally disordered nanotubes, however personal experience of the researches (nanotubes from more than 30 chrysotile deposits in the USSR) allows them to speak about at least the prevalence of the azimuthally ordered model among the chiral nanotubes.

This article is the first of a series of articles devoted to diffraction by circular, chiral and spiral multiwall nanotubes of arbitrary chemical composition. The modeling profiles of diffraction are calculated for cases of electron microdiffraction by single nanotube and of X-ray diffraction by oriented preparations of chrysotile nanotubes and are compared with the results of the research by the method of X-ray scanning of reciprocal space of textured polycrystals [8]. Correction factors (polarization, geometrical, absorption, *etc.*) at this stage of development were not considered.

## 2. Diffuse Reflexes

Whittaker has divided all nanotube reflexes *hkl* into two groups: "strong" reflexes, whose index *k* is equal to zero, and the form, as a whole, corresponds to usual crystal reflexes, and "diffuse" with $k \neq 0$ and asymmetric form. If you enter in reciprocal space the cylindrical coordinates $\{R, \varphi^*, z^*\}$, all reflexes of circular nanotube will be located extremely on so-called "layer planes" with coordinates $z^* = h/a$.

Let us consider section of reciprocal space by a plane $\{R, z^*\}$ passing through a nanotube axis, which corresponds to electron microdiffraction experiment on a single nanotube or to method of scanning of reciprocal space using oriented preparation of nanotubes. In this plane the reflexes will appear on so-called "layer lines", which are the crossings of the plane $\{R, z^*\}$ with layer planes. So, for example, the picture looks to the electron microdiffraction pattern of single circular nanotube, oriented perpendicularly to an electron beam.

The expression for amplitude of single circular nanotube diffuse reflexes *hk*0, obtained by Whittaker [5], up to the choice of coordinate system is as follows:

$$A_D^{hk0}\left(R,\varphi^*\right)=\sum_{m=0}^{M-1} p_m E_m \sum_j Y_{mj} U_j \times$$
$$\times\cos\left[2\pi k\frac{y_j}{b}+kp_m\left(\varepsilon_m-\varphi^*\right)\right] \qquad (2)$$

where $E_m=\exp\left(2\pi ih\frac{\Delta z_m}{a}\right)$, $Y_{mj}=2i^{kp_m}J_{kp_m}\left(2\pi R r_{mj}\right)$, $U_j=f_j\left(R^*\right)\exp\left(2\pi ih\frac{z_j}{a}\right)$, $f_j(R^*)$ – atomic scattering function, $R^*=\sqrt{R^2+\left(h/a\right)^2}$. Corresponding intensity:

$$I_D^{hk0}\left(R,\varphi^*\right)=4\left|\sum_{m=0}^{M-1} i^{kp_m} p_m \exp\left(2\pi ih\frac{\Delta z_m}{a}\right)\sum_j f_j\left(R^*\right)\times\right.$$
$$\times\exp\left(2\pi ih\frac{z_j}{a}\right)J_{kp_m}\left(2\pi R r_{mj}\right)\times \qquad (3)$$
$$\left.\times\cos\left[2\pi k\frac{y_j}{b}+kp_m\left(\varepsilon_m-\varphi^*\right)\right]\right|^2$$

Let us assume that initial angular phases $\varepsilon_m$ can have both random (Whittaker's model), and ordered nature. As Whittaker calculated diffraction only by the polycrystalline oriented preparation, let us consider the intensity of single nanotube diffuse reflexes with $\varepsilon_m$-random; for this let us present (3) as:

$$I_D^{hk0}\left(R,\varphi^*\right)=\sum_{m,m'}\sum_{j,j'} p_m p_{m'} E_m E_{m'}^* Y_{mj} Y_{m'j'}^* U_j U_{j'}^* \times$$
$$\times\cos\left[2\pi k\frac{y_j}{b}+kp_m\left(\varepsilon_m-\varphi^*\right)\right]\times$$
$$\times\cos\left[2\pi k\frac{y_{j'}}{b}+kp_{m'}\left(\varepsilon_{m'}-\varphi^*\right)\right]= \qquad (4)$$
$$=\sum_{m,m'}\sum_{j,j'} p_m p_{m'} E_m E_{m'}^* Y_{mj} Y_{m'j'}^* U_j U_{j'}^* \times$$
$$\times\frac{1}{2}\left\{\cos\left[\frac{2\pi k}{b}\left(y_j+y_{j'}\right)\right]\cos\left[k\left(p_m\varepsilon_m+\right.\right.\right.$$
$$\left.\left.+p_{m'}\varepsilon_{m'}\right)-k\varphi^*\left(p_m+p_{m'}\right)\right]-$$
$$-\sin\left[\frac{2\pi k}{b}\left(y_j+y_{j'}\right)\right]\sin\left[k\left(p_m\varepsilon_m+\right.\right.$$
$$\left.\left.+p_{m'}\varepsilon_{m'}\right)-k\varphi^*\left(p_m+p_{m'}\right)\right]+$$
$$+\cos\left[\frac{2\pi k}{b}\left(y_j-y_{j'}\right)\right]\cos\left[k\left(p_m\varepsilon_m-\right.\right.$$
$$\left.\left.-p_{m'}\varepsilon_{m'}\right)-k\varphi^*\left(p_m-p_{m'}\right)\right]-$$
$$-\sin\left[\frac{2\pi k}{b}\left(y_j-y_{j'}\right)\right]\sin\left[k\left(p_m\varepsilon_m-\right.\right.$$
$$\left.\left.\left.-p_{m'}\varepsilon_{m'}\right)-k\varphi^*\left(p_m-p_{m'}\right)\right]\right\}$$

Summing over *m* and *m*' retains the terms corresponding to the third addendum when *m*' = *m*, only;

all the rest are equal to zero because of random character of the initial angular phases. This is fulfilled the better the greater the number of layers *M* in nanotube. The angular dependence of intensity disappears and the final expression looks like:

$$I_D^{hk0}(R)=\frac{1}{2}\sum_m p_m^2\left|E_m\right|^2\sum_{j,j'}Y_{mj}Y_{mj'}^*U_jU_{j'}^*\times$$
$$\times\cos\left[\frac{2pk}{b}\left(y_j-y_{j'}\right)\right]=$$
$$=2\sum_{m=0}^{M-1}p_m^2\left[\left|\sum_j f_j\left(R^*\right)\exp\left(2pih\frac{z_j}{a}\right)\times\right.\right.$$
$$\left.\times J_{kp_m}\left(2pRr_{mj}\right)\cos\left(2pk\frac{y_j}{b}\right)\right|^2+$$
$$\left.+\left|\sum_j f_j\left(R^*\right)\exp\left(2pih\frac{z_j}{a}\right)J_{kp_m}\left(2pRr_{mj}\right)\sin\left(2pk\frac{y_j}{b}\right)\right|^2\right]\quad(5)$$

as $\left|E_m\right|^2=\left|i^{kp_m}\right|^2=1$.

Thus, in the Whittaker's model the intensity of diffuse reflexes of the single multiwall circular nanotube does not depend on the angle $\varphi^*$ and interlayer shifts. It is obvious that in case of oriented polycrystalline preparation of azimuthally disordered circular nanotubes the intensity of diffuse reflexes is described by the expression (5) too, averaged over the spreading of the parameters $\rho_0$ and *M*. An example of calculation of diffuse reflexes of the chrysotile 1st layer line (the number of layer line is equal to value of index *h*) for last case in comparison with experiment [17] is given in Fig. 2.

Expression (3) with $\varepsilon_m$-ordered is a basis for calculation of single azimuthally ordered circular nanotube diffuse reflexes. Value $\varepsilon_m$ can be equal, for example, to 0 (orthogonal polytype) or $\Delta bm/\rho_m$, $0 < \Delta b < b$ (azimuthal monoclinic polytype modification). Examples of calculations of azimuthal shift polytypes of circular lattice with $g = 5$ [1] reflex 020 intensity distributions on a plane $\{R, \varphi^*\}$ are shown in Fig. 3 (the lines are drawn at the levels from 0 up to 10 % through 1 %).

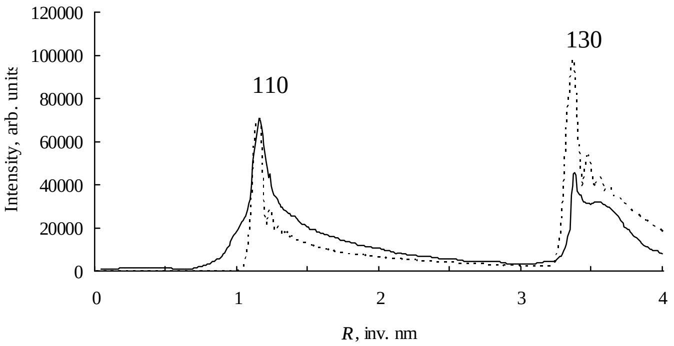
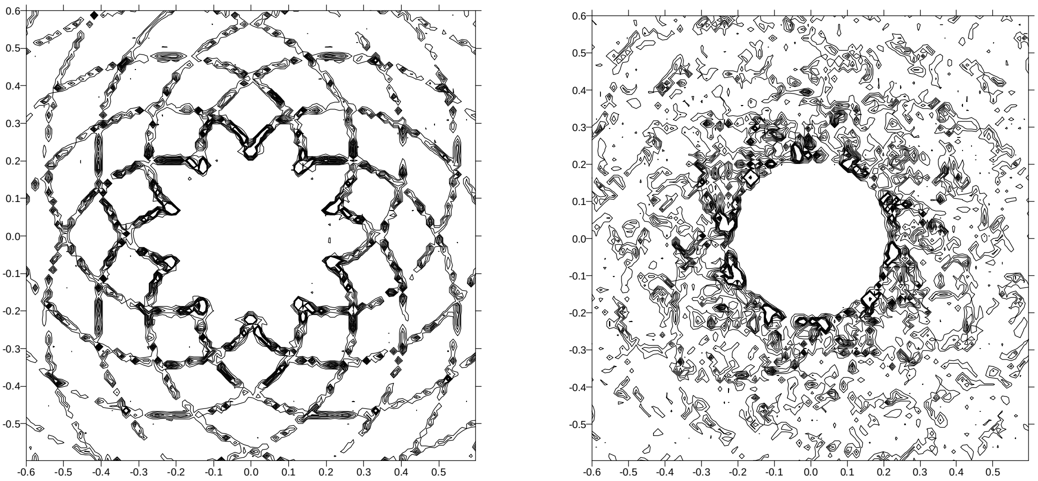


**Fig. 2.** The diffuse reflexes of chrysolite 1st layer line in Whittaker's model (shaped line) in comparison with the experimental ones (continuous line)



a b

**Fig. 3.** The distribution of intensity of circular lattice polytypes reflex 020 on a plane $\{R, \varphi^*\}$: orthogonal (a) and monoclinic with $\Delta b = b/3$ (b)

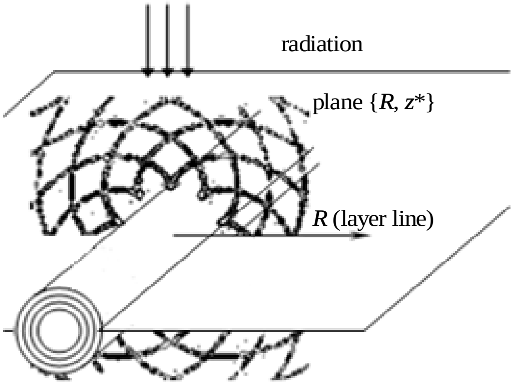


**Fig. 4.** Orientation of diffuse reflex on a plane {*R*, *φ**} around radiation and layer line

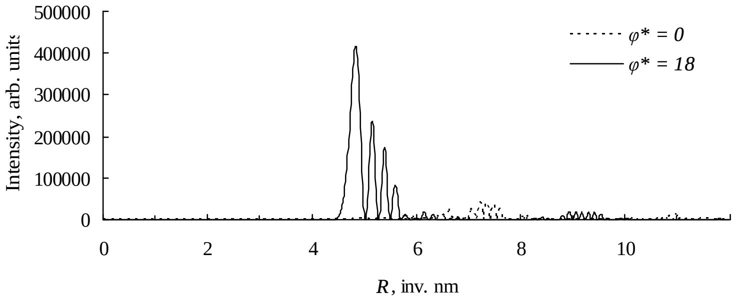


**Fig. 5.** A reflex 020 of carbon "armchair" nanotube's azimuthal monoclinic polytype (Δ*b* = *b*/3) with various values *φ** (degrees)

It is necessary to take into account the spiral-like character of intensity distribution of azimuthally ordered nanotube diffuse reflexes in research of single nanotubes by the method of electron microdiffraction. The point is that the sites of a reciprocal lattice rotate together with nanotube while the layer line is always perpendicular to direction of incident radiation (Fig. 4). Hence, the distribution of intensity on a layer line depends on the orientation of nanotube around its axis relatively an electron beam. In Fig. 5 the examples of calculated profiles of a reflex 020 of carbon "armchair" nanotube azimuthal monoclinic polytypes on the layer lines corresponding to directions given in Fig. 3b by shaped and continuous lines (the profiles in Fig. 5 are drawn by the corresponding lines) are shown.

The experimental registration of distributions shown in Fig. 3 is possible only by electron microdiffraction method with nanotube orientation parallel to an electron beam and only for a layer plane with $h = 0$ (that is taking place through the origin of reciprocal space). Nevertheless, the calculation of intensity distributions of diffuse reflexes on the other layer planes is necessary for description of position and form of diffuse reflexes on corresponding layer lines for the purpose of obtaining of structural information.

Transiting to modeling of diffuse reflexes of oriented polycrystalline preparations of the azimuthally ordered circular nanotubes, it is necessary to average intensity (3) by the angle $\varphi^*$ (and also by the distribution of parameters $\rho_0$ and $M$). Let us use (4), supposing $\varepsilon_m$-ordered:

$$I_D^{hk0}(R) = \frac{1}{2p}\int_0^{2p} I_D^{hk0}(R, j^*)\,dj^* =$$

$$= \sum_{m,m'}\sum_{j,j'} p_m p_{m'} E_m E^*_{m'} Y_{mj} Y^*_{m'j'} U_j U^*_{j'} \times$$

$$\times \frac{1}{2p}\int_0^{2p} \cos\left[2p\,k\frac{y_j}{b} + kp_m\left(e_m - j^*\right)\right] \times$$

$$\times \cos\left[2p\,k\frac{y_{j'}}{b} + kp_{m'}\left(e_{m'} - j^*\right)\right] dj^* =$$

$$= \frac{1}{4p}\sum_{m,m'}\sum_{j,j'} p_m p_{m'} E_m E^*_{m'} Y_{mj} Y^*_{m'j'} U_j U^*_{j'} \times$$

$$\times \left\{ \cos\left[\frac{2p\,k}{b}\left(y_j + y_{j'}\right) + k\left(p_m e_m + \right.\right.\right.$$

$$\left.\left. + p_{m'} e_{m'}\right)\right] \int_0^{2p} \cos\left[k\left(p_m + p_{m'}\right) j^*\right] dj^* +$$

$$+\sin\left[\frac{2pk}{b}\left(y_j+y_{j'}\right)+k\left(p_m e_m+\right.\right.$$

$$\left.\left.+p_{m'}e_{m'}\right)\right]\int_0^{2p}\sin\left[k\left(p_m+p_{m'}\right)j^*\right]dj^*+$$

$$+\cos\left[\frac{2pk}{b}\left(y_j-y_{j'}\right)+k\left(p_m e_m-\right.\right.$$

$$\left.\left.-p_{m'}e_{m'}\right)\right]\int_0^{2p}\cos\left[k\left(p_m-p_{m'}\right)j^*\right]dj^*+$$

$$+\sin\left[\frac{2pk}{b}\left(y_j-y_{j'}\right)+k\left(p_m e_m-\right.\right.$$

$$\left.\left.-p_{m'}e_{m'}\right)\right]\int_0^{2p}\sin\left[k\left(p_m-p_{m'}\right)j^*\right]dj^*\Bigg\}.$$

As numbers $k$ and $p_m$ are integer by definition, only the third integral is not equal to zero and only when $m' = m$. Then we again, as well as in Whittaker's model, obtain (5) and all its consequences.

In all polycrystalline cases positions of the main maxima of diffuse reflexes are obtained by equating of Bessel function argument (with $x_j = 0$) to its index and take places near to points $R_{hk0}=\frac{k}{b}$ slightly displacing towards the large $R$, depending on the average radius of nanotube. The displacement is different for various diffuse reflexes and can be used for measurement of average radius.

## 3. Strong Reflexes and Pseudoorthogonality Effect

Following Whittaker [5] the intensity of strong reflexes $h0l$ of single circular nanotube is described by the expression:

$$I_S^{h0l}(R)=\left|\sum_{m=0}^{M-1}p_m\exp\left(2pih\frac{\Delta z_m}{a}\right)\sum_j f_j\left(R^*\right)\times\right.$$

$$\left.\times\exp\left(2pih\frac{z_j}{a}\right)J_0\left(2pRr_{mj}\right)\right|^2 \qquad (6)$$

It has not the angular dependence and maxima of reflexes of, for example, longitudinal monoclinic polytype modification with $\Delta z_m = m\Delta z$, where $\Delta z$ – the constant longitudinal interlayer shift, are located in points

$$R_{h0l}=\frac{1}{d}\left|h\frac{\Delta z}{a}+l\right| \qquad (7)$$

the positions of an orthogonal polytype reflexes are obtained from (7) with $\Delta z = 0$. It is obvious that the intensity (6) does not depend on the character of azimuthal ordering and (after averaging by distribution of parameters $\rho_0$ and $M$) describes also the case of an oriented polycrystal.

In view of the absence of strong reflexes sets profiles, including the origins of layer lines, in Whittaker's articles, the authors have performed these calculations by the expression (6). In Fig. 6 the example of profile calculation for strong reflexes of a 2nd layer line of oriented polycrystalline preparation of chrysotile nanotubes of longitudinal monoclinic polytype modification with constant interlayer shift is shown (shaped line).

The profile contains a set of strong reflexes $20l$ in positions (7), corresponding to so-called clino-chrysotile with $d$ = 0.73 nm, $a$ = 0.534 nm and $\Delta z = -a/13$. The last parameter determines splitting of pairs $20l-20\bar{l}$. However at the origin of a layer line the narrow reflex is clearly observed, whose location contradicts the expression (7). Its position corresponds to a reflex 200 of orthogonal polytype, while the calculation was made for a monoclinic model.

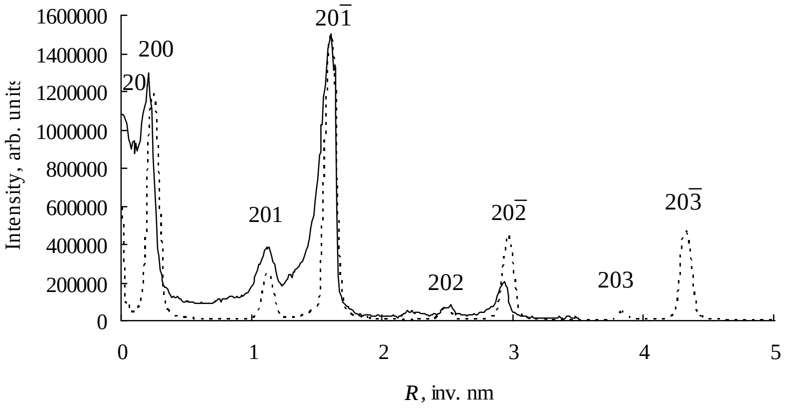


**Fig. 6**. Strong reflexes of a 2-nd layer line of chrysotile longitudinal monoclinic polytype (shaped line) in comparison with the experimental ones (continuous line)

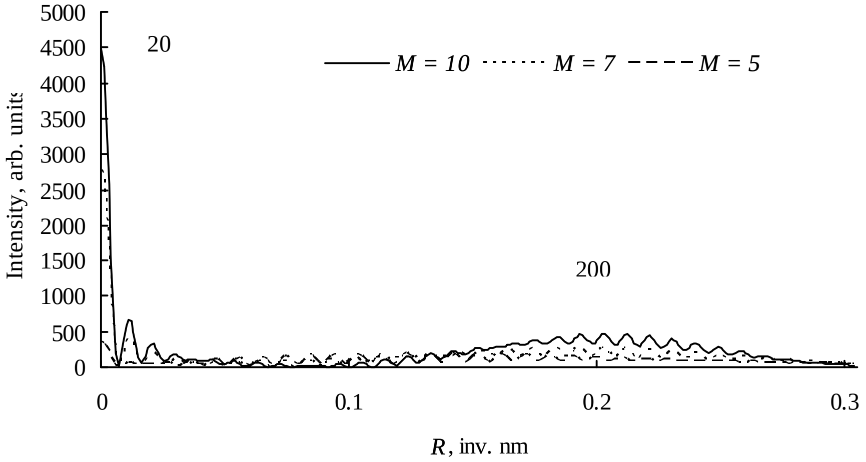


**Fig. 7.** Strong reflexes of the beginning of 2nd layer line of monoclinic circular lattice with various number of layers

Meanwhile, such reflections are often observed in a research of single nanotubes of chrysotile by electron microdiffraction: the location of reflections on the whole is consistent with monoclinic polytype, with plus *h*00 reflections of orthogonal polytype in complete absence of any other of his reflections [18]. The origin of these reflexes *h*00 remained unknown and they were named "pseudo-orthogonal". Subsequently, during the researches by the method of X-ray scanning of reciprocal space, on the diffraction patterns of chrysotiles from some deposits this effect was faced too (Fig. 6, continuous line). Hence, as it was shown in macroscopic X-ray experiment, this is a rather mass phenomenon.

Also there is a problem of indexing of this reflex. As the crystal system, whose diffraction pattern is simulated, is uniphase all its reflexes should be described by a uniform system of indexes. The Bravais cell of chrysotile allows existence of reflexes *hkl* with even values of $h + k$. For strong ($k = 0$) reflexes of the second layer line ($h = 2$) this means the presence of a set 20*l* with $l = 0, \pm1, \pm2, \ldots$. However pseudoorthogonal reflex index is not enough: for all indexes, including 200, there are own reflexes. Hence, it is possible to put a question only about the appropriateness of using the indexes *h*, *k* and *l* of a basic set of reflexes for indexing of pseudoorthogonal reflexes.

The applicability of index *h* is evident: the pseudoorthogonal reflex belongs to a layer plane with the certain value of *h*. The expression (7), describing strong reflexes, that is reflexes with $k = 0$, generates, the pseudoorthogonal reflexes also. Hence, the value of index *k* can also be assigned to them.

To determine the applicability of index *l* it is enough to consider a case of diffraction by monoclinic lattice:

$$I_S^{h0l}(R) = \left| \sum_{m=0}^{M-1} p_m \exp\left( 2p\, ih \frac{\Delta z}{a} m \right) J_0 \left( 2p R r_m \right) \right|^2 \qquad (8)$$

The index *l* of the basic set of reflexes arises from the summation in (8) over *m* as a condition of extremum of this sum. Actually, it numbers points of extremum (7). The points of extremum begin when $R = h\Delta z/ad$ and spread further towards the large values of *R*. However the pseudoorthogonal reflex takes place when $R = 0$. Therefore, it does not enter into the set of reflexes, numbered by index *l*, and this index cannot be assigned to it.

On the other hand, when *R* is so small the sum of oscillations of Bessel function "tails", multiplied by the oscillating exponent, have no (when $h \neq 0$) character of $\delta$-function, that is can not give a reflex connected with any integer parameter. Therefore in Fig. 6 and 7 the pseudoorthogonal reflexes have indexes 20.

The pseudoorthogonal reflex is the result of summation of the main maxima of Bessel functions of the zero order, multiplied by $p_m$ and a complex exponent. Its abnormally small width is determined by the large factor $2\pi\rho_m$ in argument of Bessel function. The dependence of a pseudoorthogonal reflex on the number of layers is shown in Fig. 7. It is necessary to take into account during the experimental registration of pseudoorthogonal reflexes that after effect of apparatus function of experimental device the intensity of so narrow maxima is sharply reduced and can become comparable with other reflexes of the diffraction pattern.

## 4. Conclusions

Modeling of single circular nanotube diffuse reflexes shows essential difference between the

Whittaker's model and the azimuthally ordered model and allows determining the azimuthal shift polytype modification in the last case. The greatest effect is achieved by modeling distribution of intensity in a plane {$R$, $\varphi^*$}, corresponding, in the case of zero layer plane, to electron microdiffraction by nanotube oriented parallel to electron beam. However such experimental technique is just starting to develop.

Diffuse reflexes of polycrystalline preparations of circular nanotubes do not allow to distinguish the Whittaker's and azimuthally ordered models and to determine the polytype modifications, but their modeling allows determining parameter ***b*** and average radius of nanotubes. The calculations of intensity distribution lengthways layer lines of oriented polycrystalline samples of circular nanotubes have shown the presence of oscillations in the tails of diffuse reflexes, which have never been observed experimentally. These oscillations could not be smoothed out, neither the author nor Whittaker, by his own admission [7], for any values of the parameters set by averaging the nanotubes. This allows making conclusion about the insignificant concentration of circular nanotubes in the investigated samples.

The modeling of strong reflexes both of single circular nanotubes and their polycrystalline preparations allows to determine longitudinal shift polytype modification and parameters ***d*** and $\Delta z/a$.

The research of pseudoorthogonality effect has shown that the cylindrical crystals have an additional set of pseudoorthogonal reflexes of type *h*0. The authors assume that these reflexes arise owing to the fact that even a separate atomic circle, being a two-dimensional object, is capable of scattering falling radiation directly. Thus, the pseudoorthogonality effect is the additional coherent scattering when radiation is diffracted by nanotubes. The pseudoorthogonality effect is specific for curvilinear crystals and cannot have analogues among the rectilinear ones. In further articles of this series the character of pseudoorthogonality effect in other types of nanotubes will be considered.

In the next article of this series the quantitative theory of diffraction by chiral nanotubes of arbitrary chemical composition will be presented, from which the results of this article will follow as a special case of zero-carbon chirality. The experimental profiles, used in the article were obtained with the participation of Krinary G.A., Eskina G.M., and Sabirova N.U.

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## ОСОБЛИВОСТІ ДИФРАКЦІЇ НА НЕХІРАЛЬНИХ НАНОТРУБКАХ

***Анотація.*** *Запропоновано кількісну теорію дифракції будь-якої хімічної композиції на азимутально впорядкованих кругових нанотрубках. Розглянуто псевдоортогональний ефект, знайдений експериментально нами раніше. Проведено порівняння отриманих результатів з рентгенодифракційними моделями орієнтованих препаратів хризотилових нанотрубок.*

***Ключові слова:*** *кругова нанотрубка, дифракція, електронна мікродіфракція, шарова лінія, зигзаг, хризотил.*