A Dualistic Interpretation of the Incommensurate Modulated Structures of NbTe4

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Abstract

The incommensurate/discommensurate modulated structures of NbTe4 are described using the dualistic approach, an alternative to the superspace group formalism of de Wolff [*Acta Cryst.* (1984), A40, 34-42]. On the basis of electron diffraction experiments, the room-temperature modulated structure is described dualistically as $^{P4/mcc}_{I4/mmm}$ which is equivalent to the previously reported superspace group notation $W^{P4/mcc}_{1\bar{1}11}$ determined from X-ray diffraction. On cooling NbTe4, changes occur in the electron diffraction patterns over the temperature range 100 to 50 K, before the lock-in transition to the commensurate $(2a \times 3c)$ superstructure. Although this low temperature modulation can formally be described with the same dualistic and superspace group symbols as the room-temperature one, but with the basic and modulation unit cells enlarged from a and $\sqrt{2}a$ to $\sqrt{2}a$ and $2a$ respectively, it in fact represents a discommensuration phase, where the low-temperature $(2a \times 3c)$ superstructure is nucleated within the incommensurate surrounding and accommodated to it with periodic discommensurations.

Introduction

There is currently considerable interest in the crystallographic description of incommensurately modulated structures. One such material is NbTe4. It was first reported by Selte & Kjekshus (1964) who determined by X-ray diffraction that the structure was tetragonal with $a = 6.499$, $c = 6.837$ Å and space group $P4/mcc$. They also noted that a superstructure resulted in a tripling of the c axis and a doubling of the a axes. The structure is formed of chains of square antiprisms of Te atoms with Nb atoms approximately centred in them (Fig. 2). The crystals usually grow as short fibres parallel to the c axis, thus apparently exhibiting a quasi-one-dimensional character. NbTe4 was examined more recently by Boswell, Prodan & Brandon (1981, 1983), who showed using X-ray and electron diffraction methods that, in fact, the superlattice was strongly incommensurate. Further experi-

mental work which confirmed the incommensurate nature of the distortion in NbTe4 was carried out by Mahy, van Landuyt & Amelinckx (1983), Böhm & von Schnering (1983, 1985), Boswell & Prodan (1984), Mahy, Wiegers, van Landuyt & Amelinckx (1984), Eaglesham, Bird, Withers & Steeds (1985), and Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen (1986).

A schematic diagram of the room-temperature (RT) reciprocal lattice of NbTe4 as observed by electron diffraction is shown in Fig. 1. There has been considerable variation in the notation used by different workers to label the distortion wave q vector(s) that define the incommensurate superlattice spot positions and care should be taken when different results are compared. Until recently (*e.g.* Boswell, Prodan & Brandon, 1981, 1983; Boswell & Prodan, 1984; Eaglesham, Bird, Withers & Steeds, 1985) at least two independent q vectors have been used to describe the modulation, although all superlattice spots can be accounted for in terms of a single q vector if certain spots are attributed to second-order satellites. This was shown recently, as part of a detailed X-ray structure determination, carried out by van Smaalen (1985), Bronsema (1985) and van Smaalen, Bronsema & Mahy (1986), who described the RT modulated structure of NbTe4 using the superspace group approach of de Wolff (1974, 1977), Janner & Janssen (1977, 1979, 1980a,b), de Wolff, Janssen & Janner (1981), Yamamoto (1982) and Janner, Janssen & de Wolff (1983). These workers used a single q vector to describe the modulation, which means that a series of satellites is associated with each subcell reflection in reciprocal space, arising from the distortion and not as a result of dynamical multiple scattering.

Other aspects of the NbTe4 structure, first observed by Boswell, Prodan & Brandon (1981) and not explained until recently (Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen, 1986), are the structural changes which occur below RT before the commensurate superstructure locks-in at about 50 K. Recently Boswell & Prodan (1986) have submitted further details concerning these low-temperature

(LT) structural changes. Although the superspace group approach of van Smaalen (1985) and van Smaalen, Bronsema & Mahy (1986) gives a complete description of the RT modulated structure of NbTe₄, it appears difficult to extend it to obtain physical insight into the LT structural changes. In this paper we apply the dualistic interpretation of modulated structures, recently proposed by de Wolff (1984), to provide an alternative description of the RT structure of NbTe₄ and also to elucidate the LT structural changes that occur prior to the commensurate $(2a \times 3c)$ superstructure.

The dualistic approach to the structure description of NbTe₄

In an incommensurately modulated crystal, such as NbTe₄, there is a periodic distortion whose wavelength is not a simple multiple of the periodicity of the basic structure. Thus in the direction of the modulation, the translational periodicity is lost. The classical definition of a crystal as a solid with three-dimensional periodicity must be modified if modulated structures are to be included and the structure cannot be completely described in terms of a three-dimensional space group. However, it has been shown
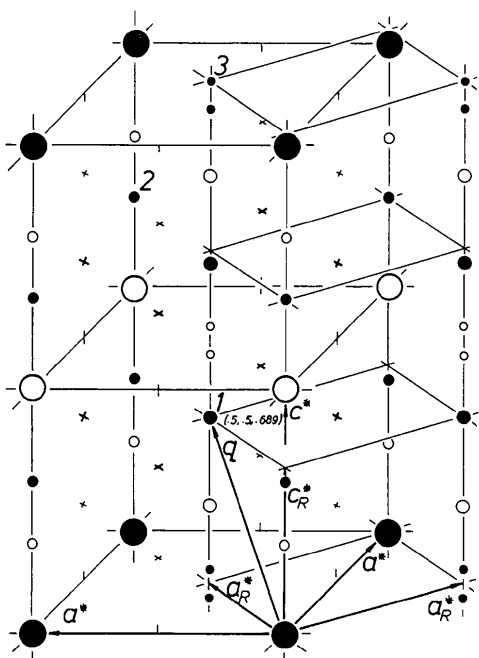

Fig. 1. Schematic diagram of the NbTe₄ room-temperature reciprocal space. The large circles, small circles and crosses indicate the subcell reflections, the room-temperature modulation satellites and the positions of the diffuse spots, which turn on cooling into streaks and low-temperature satellites, respectively. Both large and small empty circles and the light crosses show positions forbidden in the ⟨100⟩ and ⟨110⟩ sections. Unit cells for the basic structure (a^*, c^*) and the room-temperature modulation (a_R^*, c_R^*) as well as the corresponding modulation vector $q = (0.5, 0.5, 0.689)$ with the first- (1), second- (2) and third-order satellites (3) are indicated.

by de Wolff, Janssen & Janner (1981), that the symmetry of a one-dimensionally modulated incommensurate structure can be described in terms of a superspace group and this is the formalism used by van Smaalen (1985), Bronsema (1985) and van Smaalen, Bronsema & Mahy (1986).

De Wolff (1984) has described an alternative dualistic interpretation of incommensurate structures. In this approach the symmetries of the basic structure (B) and modulation (M) are described separately in terms of two conventional space groups. These groups together characterize the total symmetry of the structure and de Wolff (1984) has described the principles involved in the determination of these two space groups. The space group of the basic* structure of NbTe₄ (G_B) has been determined by a number of workers to be $P4/mcc$ using X-ray methods and this was confirmed by van Smaalen, Bronsema & Mahy (1986) who also determined the superspace group that describes the complete RT modulated structure. However, the additional diffraction effects, observed between the RT incommensurate and the LT commensurate structures present further problems associated with the very weak and mostly diffuse reflections. Recently, using electron diffraction methods, we have concluded that the resulting superstructure is not homogeneous (Boswell & Prodan, 1986), in which case the X-ray methods give only an average result.

In order to develop the dualistic interpretation we have investigated the information that can be deduced regarding the RT and LT modulations in NbTe₄ from a consideration of the reciprocal lattice derived from electron diffraction data. We explain in the following sections how this approach has been used to determine the space groups, G_{MR} and G_{ML} of the RT and LT modulations.

The incommensurate modulation structure of NbTe₄ at room temperature

A consideration of the periodicity and extinctions in the RT reciprocal lattice of NbTe₄ leads to $G_{MR} = I4/mmm$. Both G_B and G_{MR} belong to the same point group $4/mmm$ in accord with one of the dualistic principles described by de Wolff (1984). There are only four possible space groups G_{MR} which fulfill the body-centred condition. From that four, only $I4/mmm$ can be accepted, since $I4/mcm$ and $I4_1/acd$ have an additional condition: $0kl\,k,l = 2n$, which is certainly not obeyed, while $I4_1/amd$ as well as the already mentioned $I4_1/acd$ can be deleted also, since

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* To avoid confusion, it should be noted that we use the expression basic structure for the undeformed structure in agreement with the paper of de Wolff (1984). Böhm & von Schnering (1985) and Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen (1986) use the expression average structure. The last authors use the expression basic structure for the LT $(2a \times 3c)$ superstructure.

they generate too many equivalent positions along the Nb chains. Thus, the dualistic two-line symbol for the RT modulated structure is $_{I4/mmm}^{P4/mcc}$. This symbol can also be deduced directly from the NbTe$_4$ superspace group $W_{1}^{P4/mcc}$T$\bar{1}$1 previously proposed by van Smaalen, Bronsema & Mahy (1986). The reciprocal unit cells for the basic structure and the RT modulation as well as the RT modulation vector $q = (0.5, 0.5, 0.689)$ corresponding to the given superspace group, are shown in Fig. 1. The modulation axis coincides with the fourfold axes c and c_R, as required by de Wolff (1984) for an axial modulation.

It is of interest to examine symmetry relations of the modulation in the RT NbTe$_4$ structure. Positions of equivalent phasing of the modulation, generated by the symmetry operations of $G_{MR} = I4/mmm$, occupy the 16l positions along the Te chains and the 2a special positions along the Nb chains. All eight Te atom chains in a NbTe$_4$ column are modulated in phase with the Nb chains. On the other hand, the Te chains in the empty columns, form pairs which alternate in phase by π. These relationships are shown in
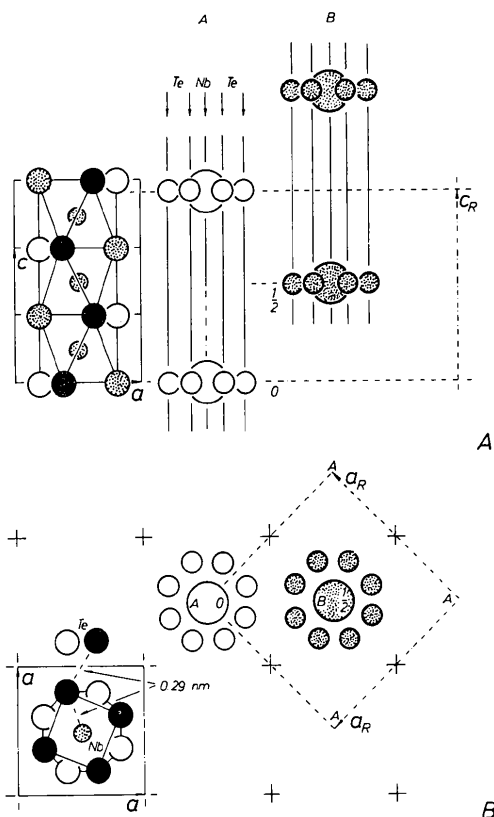

Fig. 2. Projections of the NbTe$_4$ basic structure (a, c) and room-temperature modulation (a_R, c_R) along the a axis (A) and c axis (B). The circles in the two right-hand columns A and B show equivalent positions of the modulation where the sizes of the circles indicate the atomic displacement amplitudes along Nb and Te chains. The origin of the modulation along column A and that of the basic structure have arbitrarily been chosen to coincide to clearly show the incommensurability.

Fig. 2. The resulting intrinsic phase in all NbTe$_4$ columns is the same, but two columns (A and B in Fig. 2) with a relative phase shift of π form the body-centred cell of the RT modulation with $a_R = \sqrt{2}a$ and $c_R = \gamma c$ with γ equal to the reciprocal value of the c^* component of the modulation vector q, i.e. $\gamma = 1/(1-0.311) = 1/0.689 = 1.451$.

The discommensurate low-temperature modulated structure of NbTe$_4$ and the lock-in transition

It has been shown (Mahy, van Landuyt, Amelinckx, Uchida, Bronsema & van Smaalen, 1985; Eaglesham, Bird, Withers & Steeds, 1985) that, on cooling below 50 K, NbTe$_4$ undergoes a sluggish transition to a commensurate superstructure. However, before this transition, significant changes occur in the electron diffraction patterns in the temperature range from RT to 50 K. Very weak, diffuse spherical regions in the RT reciprocal lattice of NbTe$_4$ (Fig. 1) gradually develop into streaks, extended along the c^* axis, as the temperature is lowered. At about 100 K, regularly spaced spots develop along the streaks and these spots become stronger as the temperature is decreased (Mahy, van Landuyt & Amelinckx, 1983; Boswell, Prodan & Brandon, 1981, 1983). It has been shown recently (Boswell & Prodan, 1986) that these LT spots occur in two sets which are commonly superimposed, but can arise independently in different regions of the same crystal. These two sets, labelled LT_1 and LT_2 are shown schematically, superimposed, in Fig. 3. The spot spacing is the same in each set but the two sets are displaced relative to one another along the c^* axis by exactly half the interspot distance of either set. The positions of the LT_1 and LT_2 spots in relation to the RT commensurate positions are shown in the inset of Fig. 3. When these LT spots develop there is no perceptible change in the RT superlattice spots.

We propose that the LT spots arise from small regions in the crystal with a new LT modulation. From a consideration of the reciprocal lattice we conclude that the LT modulation is body-centred and discommensurate, while the corresponding tetragonal unit cell is enlarged into $a_L \times c_L$ (i.e. $2a \times \frac{3}{2}c$). Note that although apparently incommensurate, the LT unit cell is commensurate, but the satellites are displaced as explained below (see the inset in Fig. 3, where the parameter δ indicates the relative displacements of the satellites from the commensurate position). The corresponding equivalent modulation vectors $q = (\frac{1}{2}0\frac{3}{2})$ and $(0\frac{1}{2}\frac{3}{2})$ are also shown. A possible doubling of the unit cell base ($a \times c$) is not taken into account either in the superspace group approach of de Wolff, Janssen & Janner (1981) or in the dualistic interpretation of de Wolff (1984). However, the problem can formally be resolved if the tetragonal base of the basic structure unit cell is enlarged to

$\sqrt{2}a$. The simplest dualistic interpretation is then described by $\frac{P4/mcc}{I4/mmm}$ and the corresponding super-space group is $W^{P4/mcc}_{1\bar{1}\bar{1}1}$, as for the RT modulated structure, but with both the basic and the modulation unit cells accordingly enlarged. We propose that the LT structure is composed of nucleation domains of the commensurate $(2a \times 3c)$ LT superstructure, which are accommodated to the surrounding RT incommensurate modulation by periodical discommensurations. The structure is thus discommensurate and the dualistic set goes over to the ordinary space group $P4/mcc$ with axes $(2a \times 3c)$.

The origin of the two sets of spots, LT_1 and LT_2, displaced relative to one another, can be explained using the theory of diffraction from shear structures (van Landuyt, de Ridder, Gevers & Amelinckx, 1970). According to this theory the dot product between the displacement vector $\mathbf{R}$ and the reciprocal vector $\mathbf{H}$ is equal to m – the fractional shift of the satellite from the position of the undistorted crystal. In this way $\mathbf{R}$ can be determined by measuring this displacement for three reflections belonging to two reciprocal space sections. Further, the direction of the narrow-spaced satellites and their spacing determine the defect planes and their periodicity. Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen (1986) have worked out this theory for the case of NbTe₄ and found that all satellites (only RT and LT_1 were considered) are described by vectors $\mathbf{R}_1 = \frac{1}{6}[032]$ and

$\mathbf{R}_2 = \frac{1}{6}[302]$ and their equivalents related by $\mathbf{T} = \frac{1}{2}[110]$, if the discommensuration periodicity is equal to $8c = 54 \cdot 7$ Å.

Contrary to our preliminary report (Prodan & Boswell, 1986) we note that the above explanation is correct except for the LT_2 satellites. These can be attributed to discommensurations with the same vectors, but spaced by a $16c$ periodicity. It should be pointed out that while the given vectors and the $8c$ periodicity explain the LT_1 set, with all other spots falling exactly into the positions of the RT satellites, the situation is somewhat complicated for the case where the same vectors and the $16c$ periodicity explain the LT_2 set of satellites. Some additional weak reflections (e.g. 110 or 112, indexed on the basis of the LT unit cell) would have to be observed besides the RT satellites, but they seem to be either too weak or too far from their origin. The $8c$ periodicity, which explains the LT_1 spots, contradicts the expectations since the c components of the given vectors accommodate 11 incommensurate RT modulation periods to 10 commensurate LT ones, i.e. $11 \times 1 \cdot 451c \simeq 10 \times 1 \cdot 5c + c = 16c$. Thus, the real discommensuration periodicity must be $16c$ in that case as well, but it can be averaged out to half that distance, owing to two equivalent positions shifted by $8c$ where the discommensurations can be placed as compared to the RT modulation and the basic structure (see Fig. 5).

The modulation corresponding to $G_{ML} = I4/mmm$ is shown in Fig. 4. The space groups at RT and LT are the same, but the unit cell of the first is incommensurate $(2a \times \gamma c)$ while that of the second is commensurate $(2a \times \frac{3}{2}c)$, giving together with the basic structure $(a \times c)$ the overall LT superstructure $(2a \times 3c)$. A- and B-type columns (similar to those in Fig. 2) exist, but they are now separated by $\sqrt{2}a$. The nearest neighbour columns to both A and B loose the modulation along the metal-atom chains (column C in Fig. 4). Thus, an alternative description is that columns A and B are extended laterally. There is a weak modulation in the Te chains of the C columns, due to their coupling with the Te atoms of adjacent A and B columns, such that alternating pairs of Te atoms belonging to the same original Te square undergo opposite c displacements. The equivalent positions of the modulation are the same as those for the RT modulation, i.e. $16l$ for the Te chains and $2a$ for the Nb chains. The actual atomic displacements in columns A and B are different, since the equivalent positions of the modulation along A and B are shifted in phase by π. Although it may seem somewhat surprising that different modulations would arise along originally equivalent NbTe₄ columns (this applies especially for columns C, where according to the proposed picture the modulation along the Nb chains should be lost), it should be noted that Wilson (1984) came to similar conclusions on the basis of theoretical considerations.
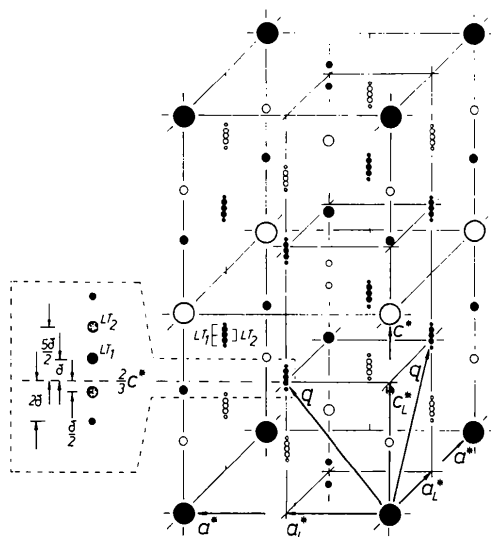

Fig. 3. Schematic diagram of the reciprocal space of low-temperature NbTe₄. The closely spaced rows of spots formed along the streaks and composed of the LT_1 and LT_2 sets are shown. The displacements of these spots relative to the commensurate position are shown in the inset, where the parameter δ has a value of about $0 \cdot 022c^*$, whereas the full and dotted circles indicate the LT_1 and LT_2 satellites. Unit cells for the basic structure (a^*, c^*) and the low-temperature modulation (a_L^*, c_L^*), as well as the equivalent modulation vectors $\mathbf{q} = (0 \cdot 5, 0, \frac{2}{3})$ and $\mathbf{q} = (0, 0 \cdot 5, \frac{2}{3})$ are indicated. The subcell reflections and the satellites forbidden in the $\langle 100 \rangle$ and $\langle 110 \rangle$ sections are indicated by empty circles.

At about 50 K NbTe$_4$ undergoes a slow lock-in transition to the commensurately modulated structure with a $(2a \times 3c)$ supercell (Mahy, van Landuyt, Amelinckx, Uchida, Bronsema & van Smaalen, 1985; Eaglesham, Bird, Withers & Steeds, 1985). During the transition the commensurate superlattice spots appear superimposed on the RT and LT satellite arrays. It appears that below 50 K the RT modulation changes directly into the commensurate phase, while the discommensurations move out of the LT modulation already present. Thus, the commensurate superstructure, which nucleates with the help of discommensurations during the LT discommensuration phase, spreads over the whole crystal.*

Discussion

In Figs. 5(a) and 5(b) a schematic diagram is given for the subcell (A), the RT incommensurate modula-

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* *Note added in proof:* Prior to this work and in agreement with it, Walker (1985) concluded on the basis of a theoretical analysis that interacting CDWs on different columns should cause a phase shift of π and that a rearrangement during the RT/LT phase transition is possible, which would enlarge the $\sqrt{2}a \times \sqrt{2}a$ basal plane into a $2a \times 2a$ one.
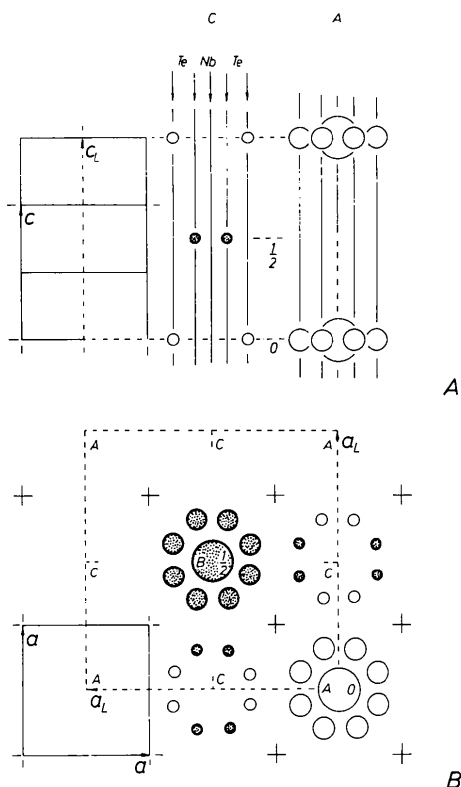
tion (or possibly long-period commensurate, since 11 modulation periods fit almost exactly to $16c$ – see Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen, 1986) (B), the suggested LT discommensurate modulation (C) and the locked-in LT superstructure (D). It is shown how the c components of the possible **R** vectors enable the nucleating LT $(2a \times 3c)$ superstructure to be adjusted to $16c$ in two possible ways, resulting in the average $8c$ or the real $16c$ periodicity. This fits the observations of Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen (1986), who noted arrays of lines separated by $8c$, and the results of Eaglesham, Bird, Withers & Steeds (1985) who found that, on heating NbTe$_4$ out of the commensurate state, faults evolved into multiple lines with an average separation of about 100 Å.

There is a composition-dependent change of the **q** vector incommensurability and the transition temperatures in mixed crystals Nb$_x$Ta$_{1-x}$Te$_4$ (Boswell & Prodan, 1984). The only difference between the two compounds is, that contrary to NbTe$_4$, in TaTe$_4$ all phases are commensurate, resulting in higher transition temperatures without any broader transition regions. There is also a high-temperature (HT) TaTe$_4$ phase with a $(\sqrt{2}a \times 3c)$ unit cell, which corresponds to the incommensurate RT NbTe$_4$. This phase was not observed until recently, since the RT/HT transition in TaTe$_4$ takes place close to the temperatures where the crystals disintegrate. Consequently, the same space group given for NbTe$_4$ seems to apply for the basic structure and for the superstructures of
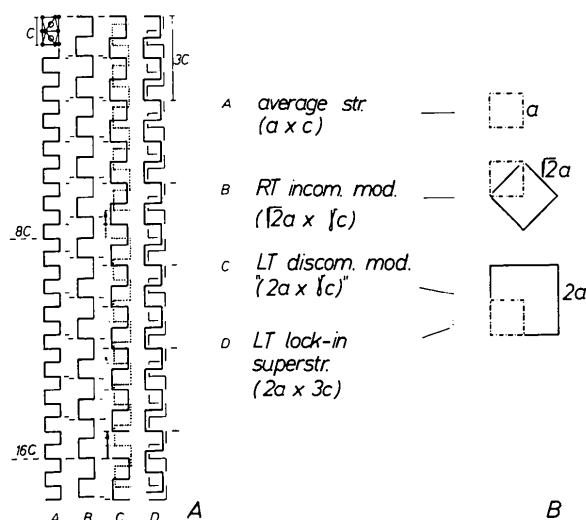
Fig. 4. Projections of the NbTe$_4$ basic structure unit cell (a, c) and the suggested low-temperature modulation (a_L, c_L) along the a axis (A) and c axis (B). The circles in the right-hand columns A, B and C show equivalent positions of the modulation where the sizes of the circles correspond to the displacement magnitudes. Note that there is no modulation along the Nb chains inside the C columns.

Fig. 5. A schematic diagram of the periodicities of the basic structure (A), the room-temperature incommensurate modulation (B), the suggested low-temperature discommensurate modulation (full or dotted lines) (C) and the low-temperature locked-in superstructure with the superimposed subcell and modulation periodicities (D) as seen perpendicular to (A) and along the c axis (B). The misfit between the periodicities of columns A and B as well as the one between columns B and C is also indicated. The two arrows along column C indicate the c components of the possible discommensuration vectors.

TaTe$_4$ as well. This is in agreement with the work of Eaglesham, Bird, Withers & Steeds (1985) who concluded from convergent-beam electron diffraction experiments that the RT TaTe$_4$ space group was $P4/mcc$, but differs from the work of Bronsema, van Smaalen, de Boer, Wiegers, Jellinek & Mahy (1987) and from that of Sahu & Walker (1985) who, on the basis of X-ray experiments and Landau-theory calculations, respectively, suggested $P4/ncc$.

We note in this connection that the present dualistic representation of the RT NbTe$_4$ modulated structure agrees with the superspace group given by van Smaalen, Bronsema & Mahy (1986), but not with their refinement, where the two mirror planes, parallel to the c axis are lost. Also, the only possible phasing between the modulations in neighbouring columns A and B, compatible with the space group $I4/mmm$ is π and not $2\pi/3$ as suggested by Mahy, van Landuyt, Amelinckx, Bronsema & van Smaalen (1986).

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