ON THE GEOMETRY OF THE QUANTUM REFLECTION OF X-RAYS IN DIAMOND

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1. *Introduction*

THE Raman effect in crystals, as is well known, arises from an exchange of energy and momentum between the crystal and a photon traversing it, the spectroscopic results of such exchange differing greatly in the two cases in which the vibrations of the crystal lattice following the encounter are respectively in the acoustic and optical ranges of frequency. In a series of papers which appeared in April and May 1940,[1, 2, 3, 4] Raman and Nilakantan put forward evidence indicating that such quantum exchanges of energy and momentum occur also when X-rays pass through a crystal. If the excited vibrations of the lattice are of the acoustic class, that is, in the nature of elastic solid waves traversing the crystal, we have a *diffuse* scattering of the X-rays with relatively small alterations of frequency. On the other hand, when the vibrations are of the optical class, their frequencies lying in the infra-red region, we have a reflection of the incident monochromatic X-rays by the lattice planes of the crystal with altered frequency in specific directions which are, in general, different from those of the classical or Laue type of reflection. The theory of these effects has been discussed by Raman and Nath in two papers, 1940.[5, 6] The geometric law obeyed by these dynamic reflections has been shown to be

$$2\,d \sin\psi \sin(\vartheta \pm \epsilon) = n\,\lambda \sin\vartheta \qquad (1)$$

$$(n = 1, 2, 3 \cdots)$$

where, d = the crystal spacing,

2ψ = the angle between the incident and the reflected X-rays,

ϵ = the inclination of the dynamic stratifications to the static crystal planes,

and ϑ = the inclination of the phase waves (planes of constant phase) of the lattice oscillations to the static crystal planes.

Though in the earlier X-ray literature references are found to " extra-spots " appearing in Laue diagrams, the true nature of the phenomenon as stated above was first indicated and established by Sir C. V. Raman and his collaborators. In view of this and especially of the formal analogy between the new reflections and the Raman effect in crystals, they will be referred to in the course of this paper as " modified " or " quantum " or " Raman " reflections to distinguish them from the well-known unmodified or classical reflections of the Laue and the Bragg types.

2. *The Case of Diamond*

Though all crystals exhibit the phenomenon of modified X-ray reflection, the case of diamond is of extraordinary importance as it furnishes several crucial tests of the Raman-Nath theory of the phenomenon. In the first place, as has been shown by Raman and Nilakantan (1940[7], and 1941[8]), the directions in which the modified reflections of the (111) planes of diamond are observed satisfy equation (1) in a very exact manner over a wide range of settings of the crystal, while they deviate widely from other formulæ which have been proposed. Further, the angle ϑ comes out as identical with the angle between the octahedral and cube faces of diamond. In other words the phase waves for the (111) reflection are parallel to the (100) planes, a most interesting result of which the physical significance becomes evident on an examination of a model of the diamond crystal. While the (111) planes are normal to the valence bonds joining the carbon atoms, the (100) planes bisect the angles between them, whereas both sets of planes contain the atoms belonging to the two interpenetrating lattices in separate layers interleaved with each other. Thus it is not surprising that an oscillation of these two lattices, relative to each other, normal to the (111) planes should have its phase waves parallel to the (100) planes.

The present paper deals with the purely geometrical aspects of the modified reflections by the (111) planes in diamond on the basis of formula (1) above. Granting that $\vartheta = 54°44'$, in other words that the phase waves are parallel to the (100) planes, it follows immediately from considerations of symmetry that there should be three sets of phase waves, instead of one, namely, sets parallel to the (100) planes, the (010) planes and the (001) planes. The consequences of this idea are followed out in the paper. It is shown that they furnish a complete explanation of the phenomena of the streamers and the subsidiary spots accompanying the modified reflections (Raman and Nilakantan,[3, 7] 1940, 1941), as also the tripling of the spots observed in certain settings of the diamond by Jahn and Lonsdale[9] (1941). Various other consequences of the formulæ are also worked out and found to be in accord with experimental facts.

3. The circular shape of the Raman spots

Before considering the phenomena of the streamers and subsidiary spots on the basis that there are three sets of phase waves as indicated above, we shall first consider the explanation of the fact that the modified reflections by the (111) planes appear in general as *round* spots instead of having an elliptic shape as in the case of the Laue reflections. We may put aside the suggestion—certainly not true in the case of diamond—that the round shape arises from any inherent "diffuseness" of the modified reflection. If we accept formula (1) as rigorously correct, *the modified reflections should be as sharp as the unmodified ones*, and the difference in shape of the spots should be merely a consequence of the difference in the geometric laws obeyed by them. We shall now proceed to show that this is actually the case.

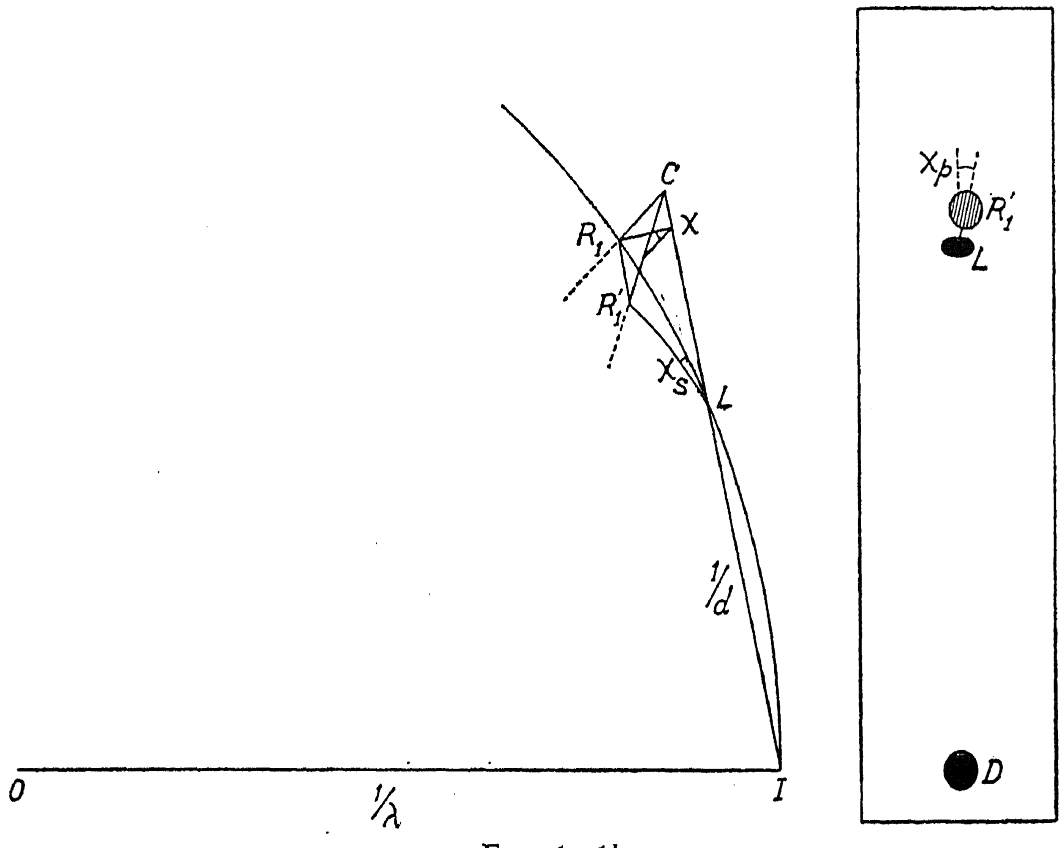
While a Laue reflection occurs with 'white' radiation, the Bragg and the Raman reflections occur with monochromatic radiation. In the Laue reflections neither the wave-length nor the angle of incidence is restricted; for the Raman reflections, owing to intensity considerations the wave-length must be considered as restricted while the angle of incidence is variable. For the Bragg reflections, both the wave-length and the angle of incidence are completely defined. Consequently when the incident pencil possesses a conical divergence only a narrow diametral section of the cone, normal to the incident plane, is operative for a Bragg reflection, while the entire cone is operative for the Laue and the Raman reflections. For the Laue reflection it is easily seen that the beam divergence in the incident-plane gets changed into a convergence while the divergence in the orthogonal plane is unaffected, thus rendering the spot elliptical. For a Raman reflection the total deviation (2ψ) of the ray remains sensibly a constant for small changes in the angle of incidence so that the original divergence of the beam remains unaffected by the reflection. Hence the Raman spot is circular with its diameter equal to the major axis of the Laue spot. Of course, this can happen only if the cone be of uniform intensity or if the exposure is sufficiently long. However, in practice the cone has a central intense core the intensity of the rest rapidly diminishing towards the edges. Therefore, in general, the modified spot is not crisp at the edges and its size is less than that of the unmodified spot.

The above discussion holds good only for an infinitely thin crystal. The effect of a small finite thickness 't' of the crystal is to elongate the spots through a distance $t \tan 2\psi$, towards the direct spot.

4. *Reflection not in the plane of incidence*

The direction of the quantum reflection is found with the help of the sphere of reflection first introduced by Ewald. The vector $OI = \overrightarrow{1/\lambda}$ represents the direction of incidence and the vector IC represents the reciprocal lattice vector $\overrightarrow{1/d}$ drawn normal to the crystal spacings concerned. When the point C lies just on the sphere of reflection drawn with O as centre and OI as radius, the Bragg reflection takes place and the angle OIC is the complement of the Bragg angle θ_B.

When θ, the glancing angle of incidence, is less than θ_B, the end C of the vector $\overrightarrow{1/d}$ lies outside the sphere. No reflection is possible unless the phase waves creating the appropriate dynamic stratifications operate. As mentioned in § 2, Raman and Nilakantan have shown for diamond that these phase waves are transverse to the plane of incidence and inclined at 54° 44′ to the crystal spacings when the incident plane is a plane of symmetry—a (110) plane. For such an incident plane the reciprocal phase vector τ_1 lies in it, and OR_1 the direction of the modified reflection, is also in the same plane (Fig. 1).



FIGS. 1*a*–1*b*

FIG. 1*a*. OR_1 is in the direction of the Raman reflection when the plane of incidence is a (110) plane. OR_1' is the direction when the plane of incidence makes an angle χ with the 110 plane. FIG. 1*b* shows the modified spot R_1' out of the incident plane as would appear on a photographic film normal to the incident X-ray pencil. L is the Laue spot.

However, when the crystal setting is slightly changed so that the plane of incidence is no longer a plane of symmetry, the reciprocal phase vector τ_1 cuts the sphere of reflection at a point R_1', which is not in the incident plane. Hence the Raman reflection whose direction is given by OR_1' goes out of the plane of incidence.

The angle $R_1 L R_1'$ on the sphere is the angle between the intersections of the planes ICR_1 and ICR_1' (the original and the final positions of the appropriate 110 plane of the crystal) with the surface of the sphere. χ is the angle between the planes ICR_1 and ICR_1'. If the angle $R_1 L R_1'$ is denoted by χ_s, we have

$$\tan \chi_s = \tan \chi \sin \theta,$$

where θ is the inclination of the tangent plane at L to the plane orthogonal to both the incident planes. This θ happens to be the glancing angle of incidence itself. The gnomonic projection χ_p of χ_s on the photographic plate normal to the incident pencil is given by

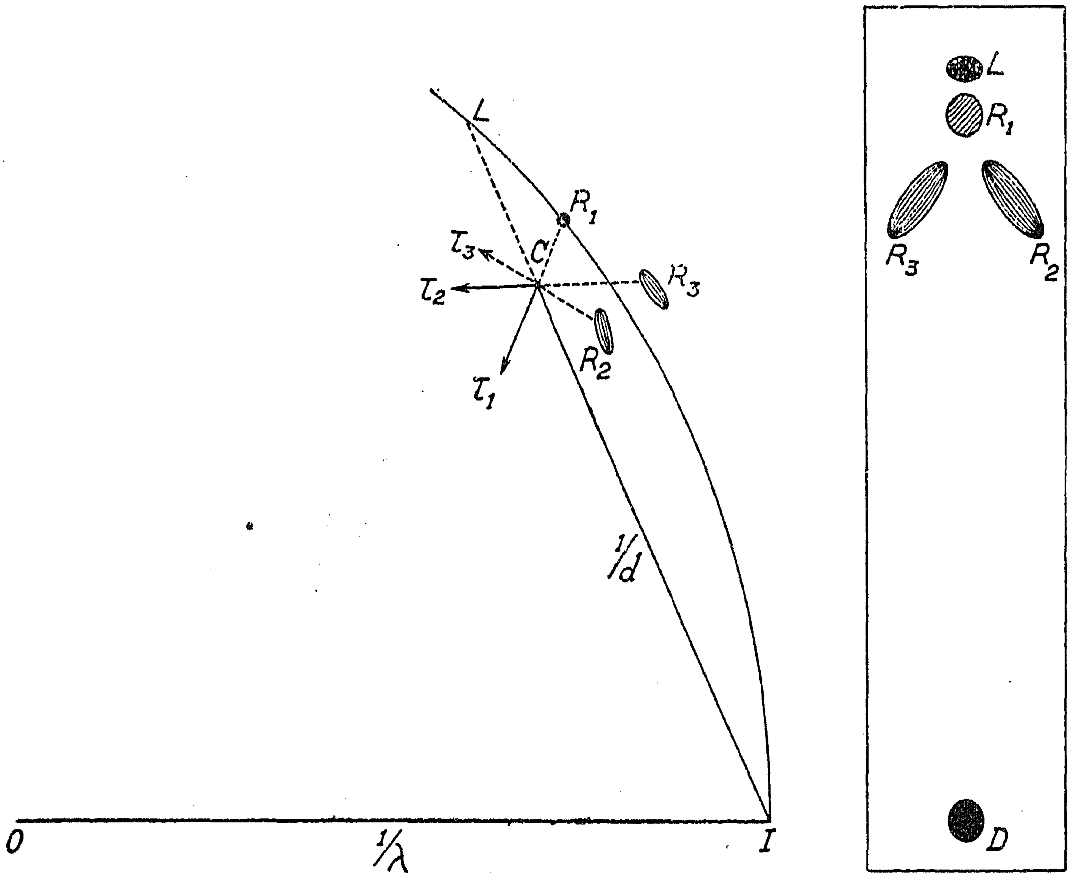
$$\tan \chi_p = \tan \chi_s \cos 2\psi = \tan \chi \sin \theta \cos 2\psi.$$

Raman and Nilakantan actually looked for such a change in the position of the modified spot with change of the incident plane and their observations are in agreement with the above formula.

The occurrence of such a modified reflection out of the plane of incidence in agreement with the above geometric formula is a striking confirmation, at least in the case of diamond, of the idea that the phase waves have a definite inclination and azimuth with respect to the crystal spacings giving rise to modified reflections.

5. *Explanation of streamers and subsidiary spots*

That the 'streamers' and the subsidiary spots are consequences of the existence of three sets of phase waves parallel respectively to the (100), (010) and (001) planes was mentioned in § 2. When θ the glancing angle of incidence is different from θ_B, the three reciprocal phase vectors τ_1, τ_2, τ_3 corresponding to the three sets of phase waves naturally operate giving rise to modified reflections. τ_1, τ_2 and τ_3 are mutually perpendicular to each other and all are inclined at an angle of 54° 44′ to the reciprocal lattice vector $\overrightarrow{1/d}$. To begin with we will consider the case $\theta > \theta_B$ and when one set of the phase waves is transverse to the plane of incidence their inclination being towards the incident pencil. This happens when the incident pencil is nearly along a trigonal axis, the reflection taking place from a set of (111) spacings normal to one of the other three trigonal axes. In this setting τ_1 is in the plane of incidence and points nearly towards the centre of the sphere of reflection. Let τ_1, τ_2, τ_3 cut the sphere of reflection at points R_1, R_2, R_3. The directions of the modified spots are given by OR_1, OR_2 and OR_3. If IC produced cuts the sphere of reflection at L, the direction of the Laue spot is OL. R_1 is in the plane of incidence and is the primary Raman reflection. The small finite divergence present in the incident beam makes IC sweep over a small angle in all directions so that R_1 is a nearly circular area while R_2 and R_3 are two very elongated ellipses. The appearance of these spots as will be recorded on a photographic plate normal to the incident pencil is represented in Fig. 2 *b*.

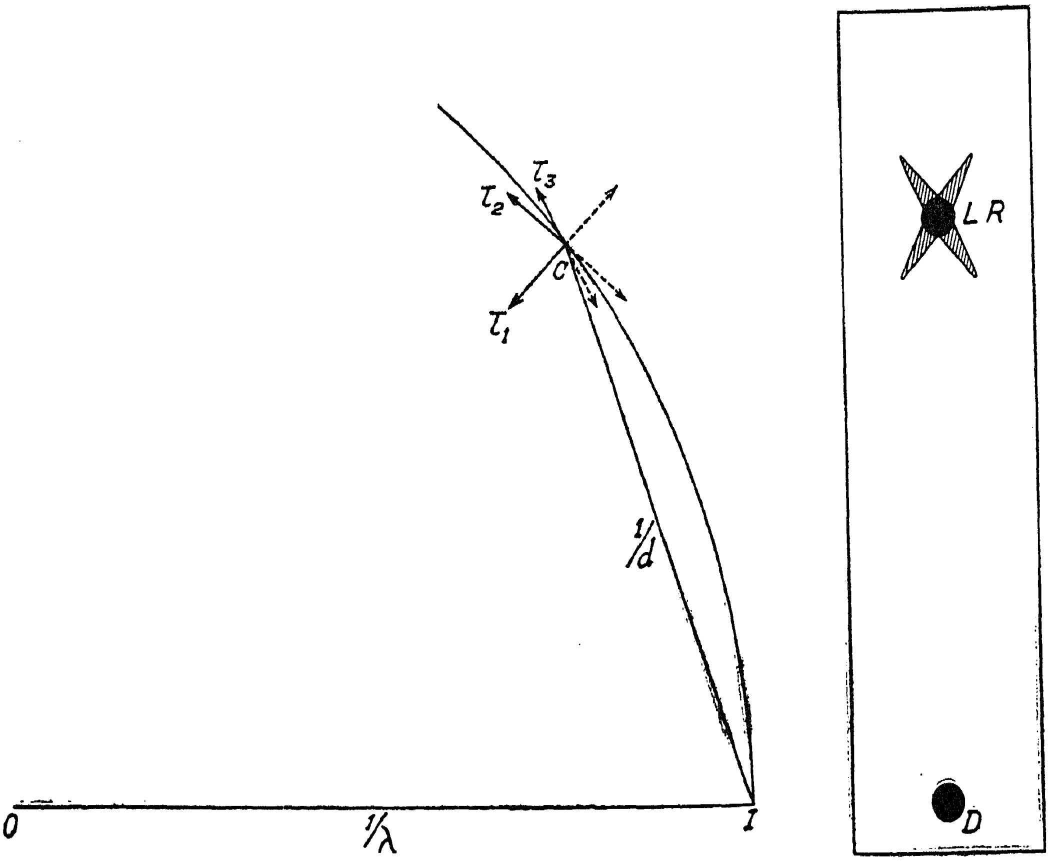


FIGS. 2*a*–2*b*

FIG. 2*a*. τ_1, τ_2, τ_3 are the three reciprocal phase vectors, τ_1 being in the plane of incidence. The diagram represents the case $\theta > \theta_B$. FIG. 2*b* indicates the primary Raman spot R_1 and the two subsidiary spots R_2 and R_3 as will be recorded on a photographic film normal to the incident X-ray pencil. L is the Laue spot.

When the angle θ is further increased, C moves away from the surface of the sphere and the distance LR_1 as well as the size of the triangle R_1 R_2 R_3 increases.

When the glancing angle θ becomes equal to the Bragg angle θ_B, C lies just on the surface of the sphere and the plane containing τ_2 and τ_3 is only slightly inclined to the surface of the sphere at C. This inclination is nearly 13° for the particular setting of diamond we are considering, when copper radiations are used. The small divergence of the incident beam makes IC sweep over a small angle in all directions so that the vectors τ_2 and τ_3 cut the sphere of reflection over long arcs nearly at right angles to each other. Every point on these arcs correspond to a possible direction of reflection so that we get four ' streamers ' when the modified spot is just on the Laue spot. The appearance of the ' streamers ' on the photographic plate is represented in Fig. 3 *b*.

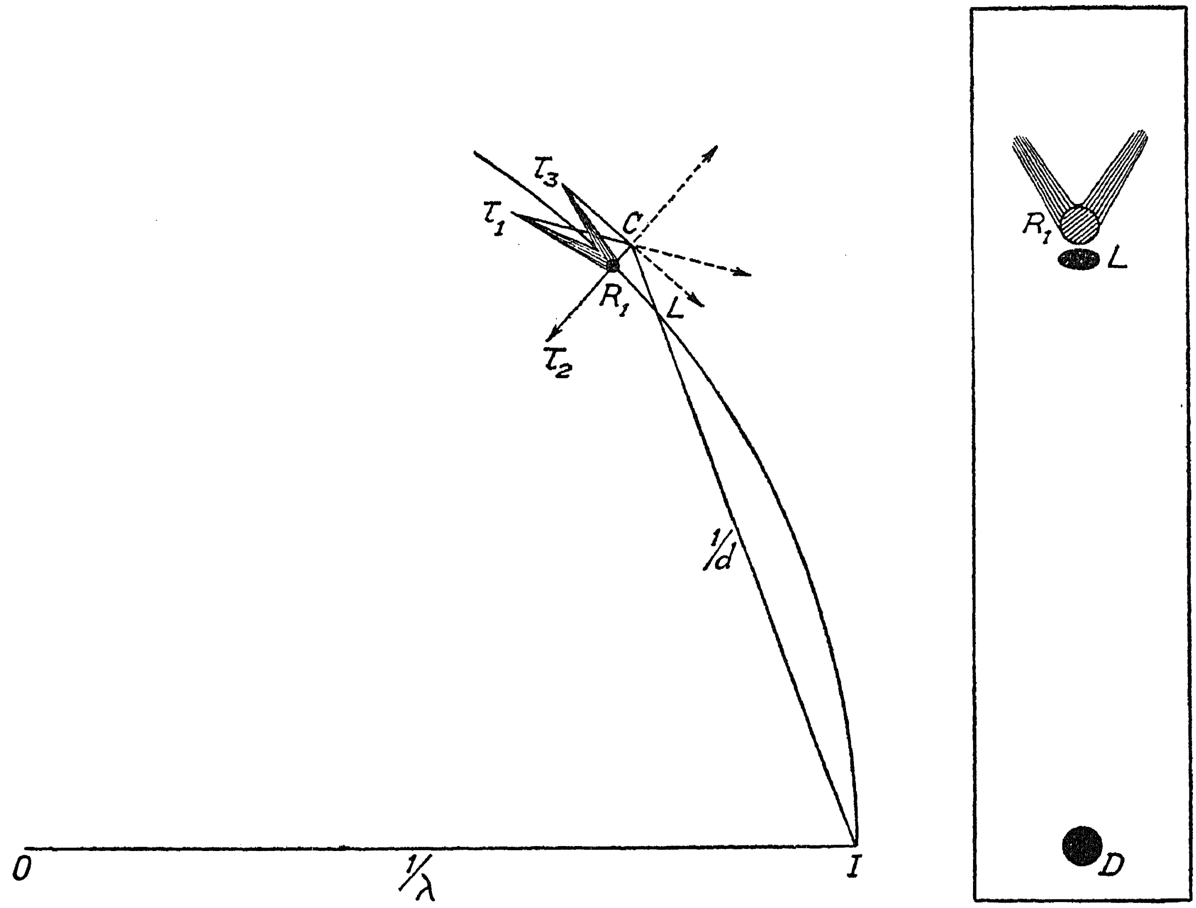


FIGS. 3*a*–3*b*

FIG. 3*a*. Case $\theta = \theta_B$. τ_2 and τ_3 which are nearly on the surface of the sphere of reflection give rise to the streamers. FIG. 3*b* shows the streamers as they appear on the photographic film normal to the incident pencil. The Laue and the Raman reflections coincide.

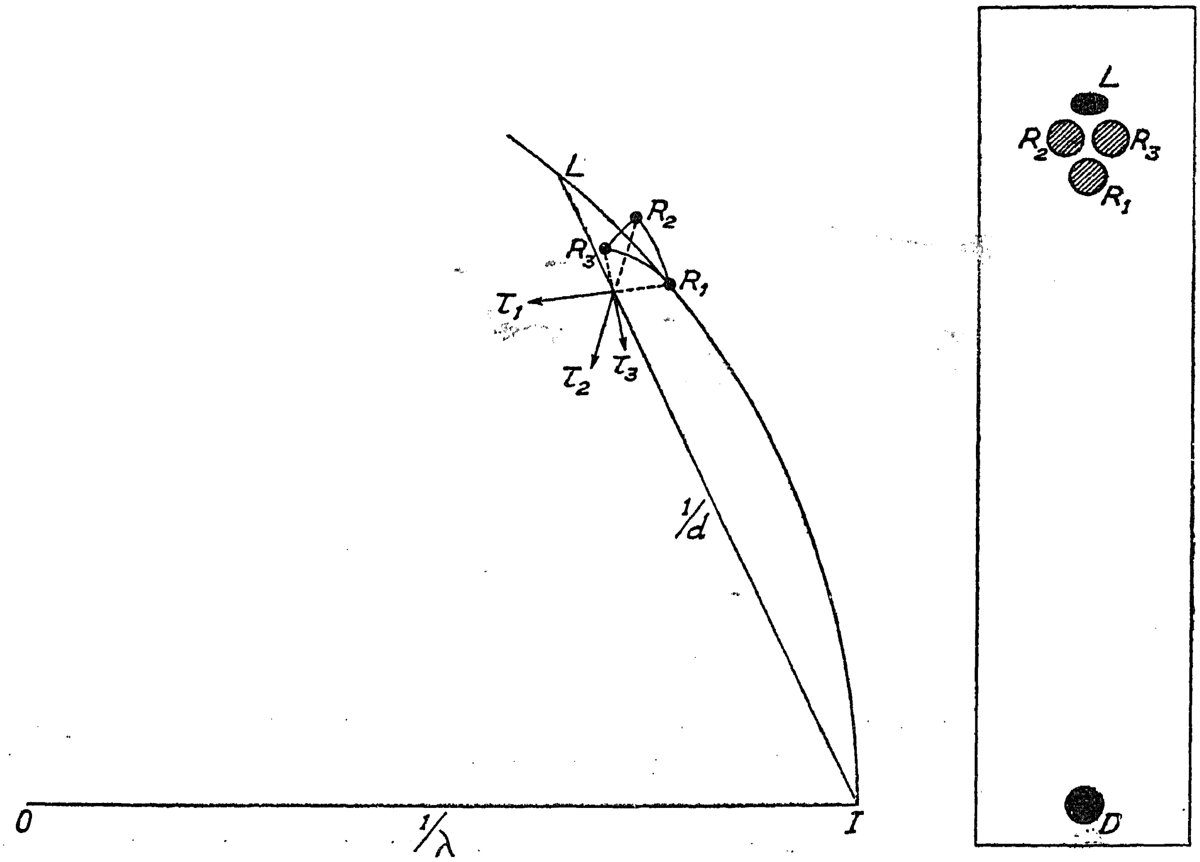
On decreasing the angle of incidence θ so that it is just less than θ_B, the end C goes out of the sphere of reflection. The Raman spot in the plane of incidence is given by R_1, L being the position of the Laue spot. τ_2 and τ_3 cut the surface in R_2 and R_3 provided the distance of C from the surface of the sphere is very small. Since the inclination of τ_2 and τ_3 to the surface of the sphere is small, the divergence of the incident beam moves R_2 and R_3 over long arcs thus giving rise to the ' streamers '. When C is fairly distant from the surface of the sphere the vectors τ_2 and τ_3 leave off the surface of the sphere and therefore the ' streamers ' disappear completely. This explains why the streamers are so sensitive to the crystal setting.

The disposition of the reciprocal phase vectors in the setting of Jahn and Lonsdale is different from what we have been considering so far. From



FIGS. 4*a*–4*b*

FIG. 4*a*. Case $\theta < \theta_B$. FIG. 4*b* shows the streamers starting from the modified spot R_1.

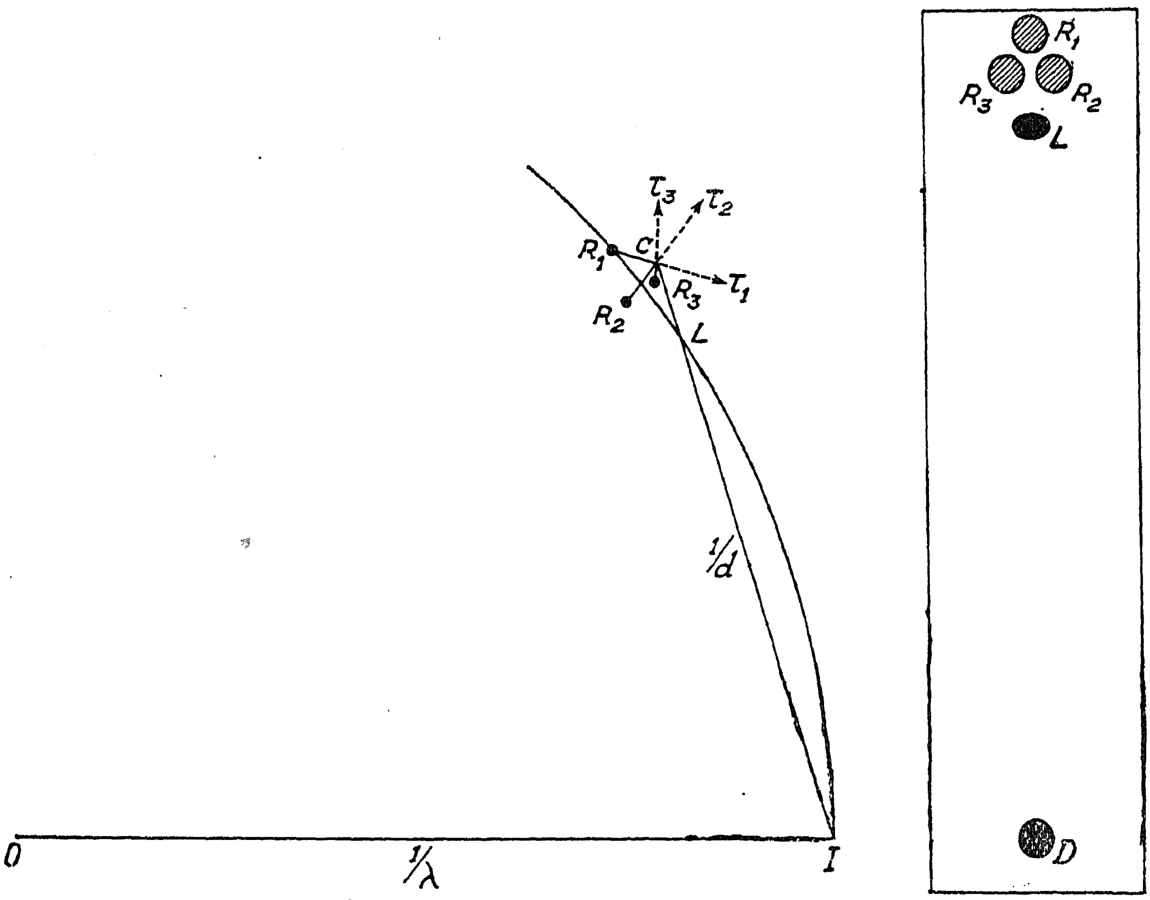


FIGS. 5*a*–5*b*

FIG. 5*a*. Shows the relative disposition of the reciprocal phase vectors in the Jahn-Lonsdale setting for $\theta > \theta_B$. τ_1 is in the plane of incidence and meets the sphere of reflection at R_1 farther from the Laue direction L, than R_2 and R_3. FIG. 5*b* indicates the triple spots as they appear on the photographic film.

a crystal model of diamond it is easily seen that the Jahn-Lonsdale setting is obtained from the setting, so far considered, by a rotation of the whole configuration through 180° with I C as axis.

For $\theta > \theta_B$, C is inside the sphere and τ_1, τ_2, τ_3 cut the surface at the three points R_1, R_2, R_3, R_1, the modified reflection in the plane of incidence being farther away from the Laue spot than the secondary spots R_2 and R_3. The small uncertainty in the direction of O C due to the divergence of the incident pencil smears out the three reflections into partly overlapping spots, as all the three vectors τ_1, τ_2, τ_3 are having practically the same inclination to the surface of the sphere. (This is not so for the setting previously considered.) No streamers can occur when C is just on the surface of the sphere ($\theta = \theta_B$); for, all the vectors are considerably inclined (nearly 35°) to the surface, while for the other setting, two of the vectors are inclined at very small angles (about 9°) to the surface of the sphere. When $\theta < \theta_B$, C goes out of the surface and the disposition of R_1, R_2, R_3 changes



FIGS. 6a–6b

FIG. 6a. Shows the reciprocal phase vectors in the Jahn-Lonsdale setting when $\theta < \theta_B$. τ_1 is in the plane of incidence. FIG. 6b indicates the triple spots as they appear on the photographic film.

such that R_1, the modified reflection in the plane of incidence, is again farther from the Laue reflection than the subsidiary reflections. The size of the triangle R_1 R_2 R_3 increases in either case with the distance of C from the surface of the sphere, *i.e.*, as $\theta \sim \theta_B$ is increased. These are just the phenomena mentioned in the Jahn-and-Lonsdale note referred to before.

6. *Quantitative Formulæ*

(*a*) *The secondary spots appearing in the first setting.*—We will denote the modified spot in the plane of incidence by R_1 and the other two by R_2, R_3 (Fig. 2 *a*). When R_1 is near the Laue spot the normal to the plane R_1 R_2 R_2 from C is inclined at an angle

$$A = \frac{\pi}{2} - \theta - \vartheta$$

with the reciprocal phase vector τ_1 giving rise to R_1. Since CR_1, CR_2 and CR_3 are mutually perpendicular, the angle $R_2\hat{R}_1R_3 = 2\,\alpha$ is given by

$$\cos 2\,\alpha = \frac{1 - \cos^2 A}{1 + \cos^2 A}.$$

As the spots are recorded on a photographic plate held normal to the incident ray, we are interested in the gnomonic projection of this angle on the plane tangent to the sphere at I. If $2\,\alpha_p$ be the value of this angle we have

$$\tan \alpha_p = \tan \alpha \cdot \cos 2\,\psi,$$

where $2\,\psi$ is the angle between OI and OR_1. For diamond $\vartheta = 54°\ 44'$ for the (111) reflection of copper K_α radiation $\theta_B = 21°\ 58'$. When the crystal is near the Bragg setting $\theta + \phi$ is practically $2\,\theta_B$. Substituting these values $2\,\alpha_p$ works out to be nearly 71°. As θ increases $2\,\psi$ also slightly increases so that the angle $2\,\alpha_p$ decreases as we move more and more from the Bragg setting.

The distance R_1 R_2 or R_1 R_3 is easily calculated in terms of the distance of C from the surface of the sphere of reflection. Let the directions of the vectors τ_1, τ_2, τ_3 be taken as the co-ordinate axes. Then the direction cosines of p, the normal drawn from C to the plane $R_1R_2R_3$ are cos A, $\sin A \cos \frac{\pi}{4}$, and $\sin A \sin \frac{\pi}{4}$. Hence,

$$R_1R_2 = p\left(\frac{2}{\sin^2 A} + \frac{1}{\cos^2 A}\right)^{\frac{1}{2}}.$$

Now
$$p = \frac{1}{d}(\theta - \theta_B) \cos \theta.$$

Therefore the angular distance, between the main Raman spot R_1 and one of the subsidiary spots, is

$$R_1\,R_2\Big/\frac{1}{\lambda} = \frac{\lambda}{d}(\theta - \theta_B) \cos \theta \left\{\frac{2}{\sin^2 A} + \frac{1}{\cos^2 A}\right\}^{\frac{1}{2}}.$$

From equation (1) it can be shown with a little manipulation, that the angular distance of the main Raman spot R_1 from the Laue spot is given by

$$L\hat{O}R_1 = 2(\theta - \theta_B)(1 + \cot \vartheta \tan \theta)^{-1}.$$

Hence the ratio of the angular distance between the main spot and a subsidiary spot to that between the main spot and the Laue spot simplifies on substituting the values involved in the copper K_α reflections from the (111) planes of diamond to the approximate value

$$0 \cdot 68 \operatorname{cosec} A.$$

A is as before $(90° - 54° \ 44' - \theta)$ and is taken as small in the simplification. When $\theta = 23° \ 12'$ for which the subsidiary spots are distinct, this ratio is nearly 3, a value which agrees very well with the experiments of Raman and Nilakantan. The formula also shows how the size of the triangle formed by the three spots increases as the distance of the main spot from the Laue spot is increased.

(*b*) *The streamers.*—When θ is just less than θ_B the vectors τ_2 and τ_3 are very little inclined to the surface of the sphere (Fig. 4). The inclination of the plane containing τ_2 and τ_3 to the surface of the sphere at R_1 is as before

$$A = \frac{\pi}{2} - \vartheta - \theta.$$

Due to the small divergence $\delta\theta$ in the plane of incidence the end point C of the vector I C moves through a distance I C. $\delta\theta$. Consequently the vectors τ_2 and τ_3 sweep out arcs on the sphere of reflection and these arcs determine the streamers. The angle 2α between the streamers is as before given by

$$\cos 2\alpha = \frac{1 - \cos^2 A}{1 + \cos^2 A}.$$

The gnomonic projection of this angle on the photographic plate is given by

$$\tan \alpha_p = \tan \alpha . \cos 2\psi.$$

Substituting the appropriate data for diamond, we get $2\alpha_p$ to be nearly 71° which is in good agreement with the observed value of about 69°.

The length of the streamers for an uncertainty $\delta\theta$ in the orientation of the reciprocal lattice vector,—*i.e.*, for a divergence of $\delta\theta$ in the incident beam is calculated as follows. The angle β made by the vector τ_1 or τ_2 with the surface of the sphere at R_1 is given by

$$\sin \beta = \sin A \cdot \sin \frac{\pi}{4}.$$

When the end of the vector I C is lifted above the surface of the sphere of reflection, the vectors τ_2 and τ_3 cut through the great circles containing them. The angle γ of the arc cut on one of them as a result of this lift is given by

$$\gamma\left(\beta - \frac{\gamma}{2}\right) = \frac{\lambda}{d} \cos \theta_B \, \delta\theta,$$

where $\delta\theta$ is the angle through I C is rotated, λ and d being the wave-length and the crystal spacing respectively. Substituting the values for the reflection of Cu K_α radiation from the (111) planes of diamond, we obtain

$$\gamma \simeq 4{\cdot}5 \, \delta \, \theta.$$

Hence a total divergence of one degree in the incident beam will produce a streamer about 4·5 degrees long. The height of the segment of the great circle containing τ_2, above the vector τ_2, in the correct Bragg setting is only

$$\frac{1}{\lambda}\left(1 - \sin^2 \frac{\beta}{2}\right).$$

Therefore when $(1/d) \cos\theta \cdot (\theta_B - \theta)$ is equal to this quantity, the vectors τ_2 and τ_3 are lifted cleanly off the surface of the sphere and the streamers disappear completely. The magnitude of $(\theta_B - \theta)$—the angular tilt of the crystal from the correct Bragg setting—required to bring about this disappearance of the streamers works out to be nearly 2° for the diamond case we are dealing with.

(*c*) *The spots in the Jahn-Lonsdale setting.*—In this setting the triangle formed by the components of the triple spot has its apex in the plane of incidence and always away from the Laue spot (Fig. 5). The angle made by τ_1 with the normal to $R_1 \, R_2 \, R_3$ is given by

$$B = \frac{\pi}{2} + \theta - \vartheta,$$

(while for the other setting it is $\frac{\pi}{2} - \theta - \vartheta$). The angle $R_2\hat{R}_1R_3 = 2\,\alpha'$ is given by

$$\cos 2\,\alpha' = \frac{1 - \cos^2 B}{1 + \cos^2 B}.$$

If $2\,\alpha'_p$ be the gnomonic projection of this angle on the photographic plate,

$$\tan \alpha'_p = \tan \alpha \cos 2\,\psi,$$

where $2\,\psi$ is the angle between the incident pencil and the modified reflection in the incident plane. Substituting the data for the particular case of diamond we are dealing with, $2\,\alpha'_p$ works out to be about 42°, when $\theta \sim \theta_B$ is small. As $\theta - \theta_B$ changes sign there is no change in the angle $R_2 \, R_1 \, R_3$, but however, the triangle gets inverted.

The angular distance R_1R_2 is given by

$$\frac{\lambda}{d}(\theta-\theta_B)\cos\theta\left\{\frac{2}{\sin^2 B}+\frac{1}{\cos^2 A}\right\}^{\frac{1}{2}},$$

and the angular distance $L R_1$ is nearly equal to

$$2(\theta-\theta_B)(1-\cot\vartheta\tan\theta)^{-1}.$$

Therefore the ratio of R_1R_2 to LR_1 is, to a first approximation,

$$0\cdot24\left(\frac{2}{\sin^2 B}+\frac{1}{\cos^2 B}\right)^{\frac{1}{2}}.$$

For settings near the Bragg setting this works out to be nearly 0·60. The photograph appearing in the *Nature* note by Jahn and Lonsdale suggests this value.

My sincere thanks are due to Sir C. V. Raman, F.R.S., for his invaluable help in the course of this work. In addition, I wish to record my thanks to Dr. P. Nilakantan for his very useful suggestions.

7. *Summary*

The paper deals with the purely geometrical aspects of the Raman reflections from the (111) planes of diamond based on the Raman-Nath formula. The round shape of the spots obtained over a wide range of the setting of the crystal as contrasted with the elliptic shape of the Laue reflections is explained as a purely geometrical consequence of the divergence of the incident pencil and the special law of the reflection. The appearance of quantum reflections outside the plane of incidence in cases when it is not a plane of symmetry is worked out quantitatively and the agreement with observation is a striking evidence of the definite orientation of the phase waves associated with the lattice vibrations in this case. From considerations of symmetry *three* sets of phase waves, parallel respectively to the three (100), (010) and (001) planes, are postulated instead of the only one transverse to the plane of incidence. The streamers and the two subsidiary spots accompanying the modified reflection (Raman and Nilakantan), their behaviour with changes of the crystal setting, the tripling of the spots in a particular setting (Jahn and Lonsdale), its changes with changes in the angle of incidence—all these different phenomena are shown to be geometrical consequences of the above idea. The various quantitative formulæ derived are found to be in accord with experimental facts.

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