

*An X-ray study of diamonds artificially prepared by  
J. B. Hannay in 1880.*<sup>1</sup>

(With Plate X.)

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**D**URING the course of a general investigation by one of us (K. L.) of diamonds from various sources, using X-ray analytical methods, the inquiry arose as to whether so-called artificial diamonds had ever been examined in this way. It was found that there was, in the Mineral Department of the British Museum, a glass slide bearing 12 minute specimens, labelled as being diamond, artificially prepared and presented by Mr. J. B. Hannay in 1880, presumably the remainder of those investigated by Prof. N. Story-Maskelyne, and referred to in a letter to the Editor of *The Times* which appeared in that paper on February 20, 1880, as follows:<sup>2</sup>

‘ARTIFICIAL PRODUCTION OF THE DIAMOND.

Sir,—A few weeks since I had to proclaim the failure of one attempt to produce the diamond in a chemical laboratory. To-day I ask a little space in one of your columns in order to announce the entire success of such an attempt by another Glasgow gentleman.

That gentleman is Mr. J. Ballantine Hannay, of Woodbourne, Helensburgh, and Sword-street, Glasgow, a Fellow of the Chemical Society of London, who has to-day sent me some small crystallized particles presenting exactly the appearance of fragments of a broken diamond.

In lustre, in a certain lamellar structure on the surfaces of cleavage, in refractive power, they accorded so closely with that mineral that it seemed hardly rash to proclaim them even at first sight to be diamond. And they satisfy the characteristic tests of that substance. Like the diamond, they are nearly inert in polarized light, and their hardness is such that they easily scored deep grooves in a polished surface of sapphire, which the diamond alone can do. I was able to measure the angle between the cleavage faces of one of them, notwithstanding that the image from one face was too incomplete for a very accurate result. But the mean of the angles so measured on the goniometer was 70 deg. 29 min., the correct angle on a crystal of the diamond being 70 deg. 31·7 min. Finally, one of the particles, ignited on a foil of platinum, glowed and gradually disappeared exactly as mineral diamond would do.

There is no doubt whatever that Mr. Hannay has succeeded in solving this problem and removing from the science of chemistry an opprobrium so long adhering to it; for, whereas the larger part of the great volume recording the triumphs of that science is occupied by the chemistry of carbon, this element has never been crystallized by man till Mr. Hannay achieved the triumph which I have the pleasure of recording to-day. His process for effecting this transmutation, hardly less momentous to the arts than to the possessors of a wealth of jewelry, is on the eve of being announced to the Royal Society.

I am, Sir, your obedient servant,  
Mineral Department, British Museum, Feb. 19.’  
N. Story-Maskelyne.

A previous letter, which appeared in *The Times* less than two months before this, on December 31, 1879, shows how thoroughly Prof. Story-Maskelyne

<sup>1</sup> A preliminary note ‘Laboratory synthesis of diamond’ appeared in *Nature*, London, 1943, vol. 151, pp. 334-335.

<sup>2</sup> Reprinted in *Chem. News*, 1880, vol. 41, pp. 97-98; *Nature*, 1880, vol. 22, p. 404; *Journ. Soc. Arts*, 1880, vol. 28, p. 289.

investigated another sample of 'artificial diamond' sent to him from a different source,<sup>1</sup> and how downright he was in his rejection of the spurious claim, which was subsequently withdrawn.<sup>2</sup> The text of this earlier letter is as follows:<sup>3</sup>

'THE SUPPOSED ARTIFICIAL PRODUCTION OF THE DIAMOND.

Sir,—I should be obliged if you would accord me space in *The Times* in order that I may answer a great number of letters and applications which have pursued me during the past few days on a subject of some little public interest, that subject being the asserted formation of diamonds by a gentleman at Glasgow.

Some ten days ago I had heard nothing whatever of the claim of Mr. MacTear, of the St. Rollox Works, Glasgow, to the artificial production of the diamond. My name, however, was already in several newspapers as that of a person in whose hands the asserted diamonds had been placed for a decision as to their true nature. Ultimately a small watch-glass, with a few microscopic crystalline particles, came into my hands for this purpose from Mr. Warington Smyth, and subsequently a supply came to me direct from Mr. MacTear. I shall proceed to state the results I have obtained from the examination of these.

Out of the first supply I selected by far the largest particle, one about 1-50th of an inch in length, and it may be that I wasted some time in experimenting on this particle, as it might not have been an authentic example of the "manufactured diamond", since it differed in some respects from the specimens I have since received direct from Mr. MacTear.

Now, firstly, the diamond excels all substances in hardness. Secondly, its crystals belong to the cubic system, and should not, therefore, present the property of doubly refracting light. Frequently, however, from the influence of strain within the crystal, caused by enclosed gas-bubbles or other causes, diamonds are not entirely without action on a ray of polarized light sent through them. Finally, the diamond is pure carbon, and, as such, burns entirely away when heated to a sufficiently high temperature in the air, and more vividly so burns or glows away when heated in oxygen gas.

The specimens I had to experiment upon were too light to possess appreciable weight, too small even to see unless by very good eyesight or with a lens, yet were, nevertheless, sufficiently large to answer the three questions suggested by the above properties.

A few grains of the dust, for such the substance must be termed, was placed between a plate of topaz—a cleavage face, with its fine natural polish—and a polished surface of sapphire, and the two surfaces were carefully "worked" over each other, with a view to the production of lines of abrasion from the particles between them. There was no abrasion. Ultimately the particles became bruised into a powder, but without scratching even the topaz. They are not diamond.

Secondly, some particles, more crystalline in appearance than the rest, were mounted on a glass microscope slide, and examined in the microscope with polarized light. They acted each and all powerfully in the manner of a birefringent crystal. It seemed even in one or two of them that when they lay on their broadest surface (it can scarcely be called a "crystal face"), a principal section of the section of the crystal was just slightly inclined to a flattish side of it in a manner that suggested its not being a crystal of either of the orthosymmetrical systems. Be that as it may, it is not a diamond.

Finally, I took two of these microscopic particles and exposed them to the intense heat of a table blowpipe on a bit of platinum foil. They resisted this attempt to burn them. Then, for comparison, they were placed in contact with two little particles of diamond-dust exceeding them in size, and the experiment was repeated. The result was that the diamond particles glowed and disappeared, while the little particles from Glasgow were as obstinate and unacted on as before. I had previously treated the specimen I have alluded to as the first on which I experimented, by making a similar attempt in a hard-glass tube in a stream of oxygen, and the result was the same. Hence I conclude that the substance supposed to be artificially formed diamond is not diamond and is not carbon; and I feel as confident in the results thus obtained from a few infinitesimal particles that can hardly be measured, and could only be

<sup>1</sup> Chem. News, 1879, vol. 40, p. 306.

<sup>2</sup> A. H. Allen, On the artificial production of precious stones. Chem. News, 1880, vol. 41, pp. 68-69.

<sup>3</sup> Reprinted in Chem. News, 1880, vol. 41, pp. 4-5; Nature, 1880, vol. 21, pp. 103-104; Journ. Soc. Arts, 1880, vol. 28, p. 105.

weighed by an assay balance of the most refined delicacy, as if the experiments had been performed on crystals of appreciable size.

Not content with merely proving what these crystalline particles are not, I made an experiment to determine something about what they are.

Heated on platinum foil several times with ammonium fluoride they became visibly more minute, and a slight reddish-white incrustation was seen on the foil. At the suggestion of Dr. Flight, assistant in this department, a master in the craft of the chemical analyst, these little particles were left for the night in hydrofluoric acid in a platinum capsule. This morning they have disappeared, having become dissolved in the acid, and on evaporation there is seen a slight white incrustation, on the capsule, of the residuary fluoride. I have, therefore, no hesitation in declaring Mr. MacTear's "diamonds", not only not to be diamonds at all, but to consist of some crystallized silicate, possibly one resembling an augite, though it would be very rash to assert anything beyond the fact that they consist of a compound of silica, possibly of more than one such compound.

The problem of the permutation of carbon, from its ordinary opaque black condition into that in which it occurs in nature as the limpid crystal of diamond, is still unsolved. That it will be solved no scientific mind can doubt, though the conditions necessary may prove to be very difficult to fulfil. It is possible that carbon, like metallic arsenic, passes directly into the condition of vapour from that of a solid, and that the condition for its sublimation in the form of crystals, or its cooling into crystal-diamond from the liquid state, is one involving a combination of high temperature and high pressure present in the depths of the earth's crust, but very difficult to establish in a laboratory experiment.

I am, Sir, your obedient servant,

Nevil Story Maskelyne.

Mineral Department, British Museum, Dec. 30.'

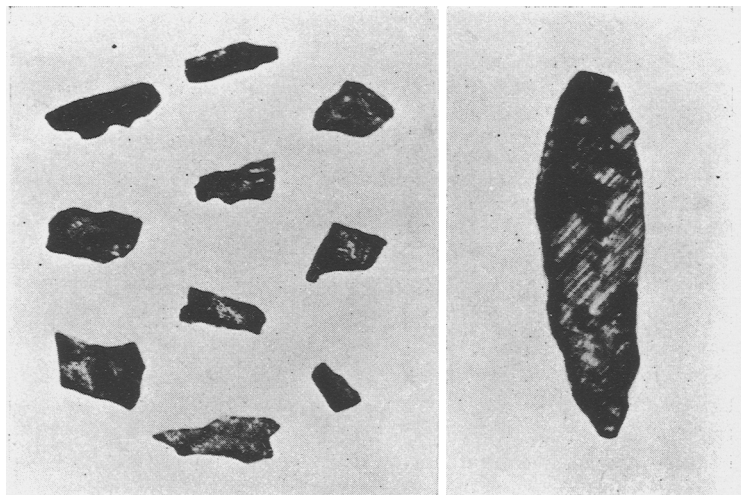
In view of the interest aroused in the subject at about that time, Story-Maskelyne's acceptance of the specimens submitted by Hannay as being genuine diamonds has all the more authority, since his investigation of them was certainly as searching as possible.

The twelve particles that remain from Story-Maskelyne's work in 1880 were found mounted in Canada balsam on a glass slide. They are of irregular shape averaging in size  $0.4 \times 0.2 \times 0.1$  mm. (text-fig. 1). Unfortunately no account of their identification by Story-Maskelyne is to be found in the records of the Mineral Department, and his correspondence with Hannay has not been traced. The glass slide is accompanied by a card label 'Hannay's artificial diamonds' in the handwriting of Thomas Davies, assistant in the department from 1862 to 1892. The first examination of Hannay's specimens in London must have been carried out a few months before the removal of the Mineral collections from the British Museum, Bloomsbury, to the new building in South Kensington, in June 1880. It is indeed fortunate that a few fragments mounted on glass should have survived removal and that they were eventually catalogued, registered (B.M.87756), and labelled by Dr. L. J. Spencer in 1901. Our recent re-examination of the fragments not only places their identity beyond doubt but adduces new evidence for the authenticity of their origin.

Examination of each particle in turn under the polarizing microscope prior to an X-ray rotation photograph, showed that all but one are isotropic, colourless, and have the lustre and appearance of diamond. The X-ray photographs, as will be shown in detail later, confirm that eleven of the twelve are indeed diamond. The remaining buff-coloured birefringent particle has not yet been identified with certainty; possibly it is a fragment of some ceramic material. Several of the diamond particles have curiously striated cleavage faces (text-fig. 2) which in our opinion identifies the specimens satisfactorily with Hannay's material, for it is

precisely this feature which Story-Maskelyne commented upon in his description quoted above. That being so, it is of interest to consider Hannay's work from a historical point of view and to find out, if possible, why he has never received the credit due to him for his success.

The experiments which led Hannay to take up this research are described in two papers to the Royal Society.<sup>1</sup> His principal interest at this time was in the nature of the fluid state, as later papers show,<sup>2</sup> and he and a colleague, James Hogarth, had been engaged on the study of the solubility of solids in fluids above their critical point. It occurred to him that carbon, hitherto so recalcitrant as a



Hannay's artificial diamonds.

FIG. 1. Ten of the twelve fragments (B.M.87756) remaining from Story-Maskelyne's tests.  $\times 33$ .

FIG. 2. One of the fragments (the eleventh), which proved to be a mosaic type II diamond, showing cleavage striations. Photographed in methylene iodide.  $\times 100$ . (See pl. X, figs. 4 and 5.)

solute, might dissolve in some fluid above its critical point, and might then crystallize out as diamond rather than as graphite when the temperature was gradually reduced. Many experiments showed him that ordinary carbon (charcoal, lampblack, or graphite) was not affected by the most probable solvents, chemical action taking the place of solution. He noticed, however, that when a gas containing carbon and hydrogen was heated under pressure in the presence of Li, K, Na, or Mg, the hydrogen combined with the metal and the carbon was left free, being deposited in a particularly hard, scaly form. Then began a series

<sup>1</sup> J. B. Hannay and J. Hogarth, On the solubility of solids in gases. *Proc. Roy. Soc.*, 1879, vol. 29, pp. 324-326; 1880, vol. 30, pp. 178-188.

<sup>2</sup> J. B. Hannay, On the state of fluids at their critical temperatures. *Ibid.*, 1880, vol. 30, pp. 478-484. On the solubility of solids in gases, II. *Ibid.*, 1880, vol. 30, pp. 484-489. On the limit of the liquid state. *Ibid.*, 1881, vol. 31, pp. 520-522; 1882, vol. 33, pp. 294-321. On the absorption of gases by solids. *Ibid.*, 1881, vol. 32, pp. 407-408. On the states of matter. *Ibid.*, 1881, vol. 32, pp. 408-413.

of heart-breaking experiments 'On the artificial formation of the diamond', described in a preliminary note and in great detail in a later paper presented to the Royal Society,<sup>1</sup> which were communicated and read by Professor G. G. Stokes, then a Secretary of the Society.

Only three out of eighty experiments were at all successful; Hannay scrupulously describes those that were unsuccessful as well as those in which he believed he had succeeded in obtaining diamond. In the experiment which gave him the largest amount of solid diamantiferous material he used a mixture of 'paraffin spirit boiling at 75°' (a mixture of light paraffins) 90%, together with 10% of carefully rectified bone-oil<sup>2</sup> ('Bone oil . . . was distilled, and the portion boiling between 115° and 150° was taken and rectified over solid caustic potash, and latterly over sodium [until] free from moisture, oxygen, and sulphur'); these were placed, with 4 gm. metallic lithium, in a tube constructed on the gun-barrel principle (a wrought iron coil-tube) made of Lowmoor iron which he describes as 'very pure and strong'. The dimensions of the tube were 20 inches long  $\times$  4 inches external diameter,  $\frac{1}{2}$  inch bore, and the mixture three-quarters filled it. After many efforts at obtaining an effective seal, he found that the best method of closing the tube was by welding, although this was a most difficult performance and, as he says, may occasionally have introduced a little silica. The completely closed tube was then placed in a tilted position in a large reverberatory furnace (which had to be rebuilt after some shattering explosions) the construction of which is minutely described, and heated to a 'dull red-heat' for fourteen hours, after which it was allowed to cool slowly. Many of his tubes exploded under this treatment; of those that did not explode, 90% were quite empty when they were opened, although a test by hydraulic pressure showed that there had been no leakage of any ordinary kind. What did happen, as he himself realized, of course, was that at the dull red heat attained, the iron coil-tube became porous to gases (Proc. Roy. Soc., 1881, vol. 32, p. 407), and although Hannay tried various ways of lining his tubes (electroplating with Cu, Ag, Au; siliceous linings of fusible enamels and glass) he only succeeded in retaining any liquid or condensed gaseous matter in the tubes in 4 out of 34 subsequent experiments.

However, in his few successful experiments the tubes, for some reason or other, *did* hold the pressure, for he writes:

'On opening the tube a great volume of gas was given off, and only a little liquid remained. *In the end of the tube which had been the upper end in the furnace* [our italics], the tube lying obliquely, there was a hard smooth mass adhering to the sides of the tube, and entirely covering the bottom. As I had never obtained all the solids in one piece before, I wished to examine it, and so had the other end of the tube cut off, exposing the hard mass. It was quite black, and was removed with a chisel, and as it appeared to be composed principally of iron and lithium it was laid aside for analysis. I was pulverising it in a mortar, when I felt that some parts of the material were extremely hard—not resisting a blow, but hard otherwise. On looking closer, I saw that these were mostly transparent pieces imbedded in the hard matrix, and on triturating

<sup>1</sup> J. B. Hannay, On the artificial formation of the diamond. Proc. Roy. Soc., 1880, vol. 30, pp. 188–189 (preliminary notice); 1880, vol. 30, pp. 450–461. Reprints or summaries of these papers appeared in Chem. News, 1880, vol. 41, pp. 106, 111; Nature, 1880, vol. 21, pp. 421–423, vol. 22, pp. 255–257.

<sup>2</sup> In Hannay's second paper these quantities are reversed, and have been so quoted subsequently by others, but it is clear from the context that only a relatively small percentage of bone-oil was actually used and that Hannay intended to give the figures as we have given them above.

them I obtained some free from the black matter. They turned out to be crystalline carbon, exactly like diamond.' (Proc. Roy. Soc., 1880, vol. 30, p. 458.)

'The carbon obtained in the successful experiments is as hard as natural diamond, scratching all other crystals, and it does not affect polarised light. I have obtained crystals with curved faces belonging to the octahedral form. . . . The crystals burn easily on thin platinum-foil over a good blowpipe, and leave no residue, and after two days' immersion in hydrofluoric acid they show no sign of dissolving, even when boiled. On heating a splinter in the electric arc, it turned black—a very characteristic reaction of diamond.' (Proc. Roy. Soc., 1880, vol. 30, p. 189.)

A combustion conducted in pure oxygen, the crystals being laid on a thin piece of platinum foil which was ignited by an electric current, showed that the sample used contained 97.85 % carbon, a very close approximation considering the small quantity (14 mg.) available for test. The specific gravity, determined by flotation in a mixture of bromide and fluoride of arsenic, ranged as high as 3.5. Hannay was not satisfied with the results of what he afterwards referred to as 'that most troublesome investigation' (Proc. Roy. Soc., 1881, vol. 32, p. 407), and stated finally: 'I intend, when my other work—which I laid aside for the diamond experiments—is finished, to begin a series of experiments on the decomposition of carbon compounds by metals, to find whether a more easily controlled reaction may not be discovered' (Proc. Roy. Soc., 1880, vol. 30, p. 461). Apart from the 34 additional experiments mentioned above, in which he used tubes with various linings, it does not appear that Hannay ever carried out this intention.

It is noteworthy that Hannay regarded the presence of *nitrogen* as being essential to the success of the experiment, though whether the nitrogen was believed by him to be an essential constituent of diamond (since he says that his final crystals contained a very small amount of nitrogen chemically combined) or whether, as he says elsewhere, the carbon after being set free from the hydrocarbon has to be acted upon by a stable nitrogen compound in order that it may be obtained in the clear, transparent form of the diamond, is not obvious from his description.<sup>1</sup>

In spite of Hannay's careful tests of his final material and of the confirmation provided by Story-Maskelyne, his claim to have made diamond by a laboratory method, seems never to have been taken seriously except, perhaps, by Moissan. In 1902 we find Hannay complaining (Chem. News, 1902, vol. 86, p. 173) of a statement made in the new volumes of the 'Encyclopaedia Britannica' (10th edition, article 'Gem, Artificial'), that the hard substance he found in his tubes was really carborundum. 'Had the original paper or the correspondence in *The Times* been consulted,' he writes, 'this could hardly be asserted, as it was fully tested at the time, and was found to be converted entirely into carbon dioxide when burnt in air or oxygen.'

In his Bakerian Lecture in 1918 Sir Charles Parsons<sup>2</sup> claims to have repeated Hannay's experiments but without success. He says (p. 77):

'Hannay's experiments were repeated, where paraffin and dipple-oil with the alkali metals, especially potassium, were sealed in steel tubes and subjected to a red heat for several hours. The analysis gave no diamonds; in fact it became apparent that when hydrocarbons or water

<sup>1</sup> Compare W. H. Hudleston, *Min. Mag.*, 1883, vol. 5, p. 209.

<sup>2</sup> C. A. Parsons, *Experiments on the artificial production of diamond*. *Phil. Trans. Roy. Soc., Ser. A*, 1919, vol. 220, pp. 67-107. [M.A. 1-232.]

were relied on to produce pressure, the latter could only exist for a short time at the commencement, for when a red heat was reached the hydrogen escaped through the metal, and the oxygen combined with the steel. We did not analyse the steel tubes themselves. Many experiments were, however, tried with central heating under the press at 6000 atmospheres, and nothing was obtained of interest with the substances used by Hannay, unless, as previously mentioned, some iron was present.'

We have tried unsuccessfully to trace any of the products of Sir Charles Parsons's experiments, but it is clear from his own statements<sup>1</sup> that Parsons was satisfied that in no case had he himself really succeeded in preparing diamond artificially, and he appears to have been equally convinced that no one else had done so either.<sup>2</sup> The article in 'Nature' referred to below states:

'Sir Charles Parsons and Mr. [H. M.] Duncan have also repeated the experiments of other workers who have claimed to have produced diamonds by artificial processes, but in every case the results have been negative. The conclusion seems inevitable that diamonds have not yet been produced in the laboratory, and that investigators have been misled into regarding as diamonds various transparent, singly-refracting minerals which happen to be very resistant to chemical reagents.'

In the same article the writer suggests that spinels, which are formed at high temperatures from magnesia and alumina are exceedingly resistant to repeated treatment with concentrated hydrofluoric acid and boiling sulphuric acid, and that being colourless and cubic in structure they may easily be mistaken for diamonds; only the combustion test, which would leave such crystals unchanged, could satisfactorily distinguish the genuine diamond.

X-ray analysis does, however, provide the means of making an even more stringent test. It is well known that every substance gives certain typical and unique diffraction patterns when irradiated by X-rays, the nature of the pattern depending upon the particular technique adopted. These patterns are most conveniently examined by means of photography. If, for instance, the substance is available in powder form only, the pattern recorded when a suitably prepared specimen of the powder is irradiated by X-rays of a particular wave-length, consists of a number of fine lines, the relative positions and intensities of which, by comparison with a card-index of patterns of known substances, can provide a positive means of identification of the powder. The analogy with the 'finger-print' method of criminal identification has actually led to the use of the term 'crystal finger-print' for a pattern of this kind. When the unknown substance is available in the form of small single crystals, other methods of identification are also available. Firstly, the crystal may be mounted in a random orientation and then rotated or oscillated during irradiation by X-rays of a particular wave-length. This will give, for diamond, a pattern of the kind shown in pl. X, fig. 5, which is obtained using a circular camera with copper radiation. The spots correspond to reflections from particular planes of the crystal, their radial distance from the trace of the undeviated beam gives a measure of the spacing of the planes, and their intensity indicates the relative atomic densities of the planes. Thus by

<sup>1</sup> Quoted in an article on 'The problem of artificial production of diamonds'. *Nature*, 1928, vol. 121, pp. 799-800, signed C. H. D[esch]. [M.A. 4-68.]

<sup>2</sup> In the biography of Sir Charles Parsons by Rollo Appleyard ('Charles Parsons', Constable & Co. Ltd., London, 1933) it is stated: 'At a meeting of the Royal Microscopical Society on April 23, 1924, he [i.e. Parsons] said he had for twenty years been trying to make diamonds, and had spent on his experiments £20,000, yet he had come to the conclusion that nobody had ever made a diamond.'

means of X-rays not only the crystal form is ascertained, but also the distances apart of the atoms, the arrangement of the atoms, and their actual atomic weights. No two known substances are composed of atoms of the same weights, arranged in the same way, and having the same interatomic distances, and any substance is therefore capable of unique identification by this means. Such a test shows that eleven of the twelve particles (B.M.87756) labelled as being the products of Hannay's experiments are indeed diamond. In the case of four specimens, reflections foreign to the diamond pattern are present and these, though insufficient in number for positive identification of the impurity giving them, might be due to adherent or included magnetite and graphite, while one buff-coloured, birefringent specimen, already mentioned, is not diamond at all and has not yet been identified.

A second method of using single crystals for identification purposes is that of the Laue photograph, for which white radiation (covering a range of wavelengths) is used with the crystal specimen stationary. Only one of the Hannay specimens (text-fig. 2 and pl. X, fig. 4) has been examined in this way, but the results are completely satisfactory. By mounting the crystal on a pair of arcs it was found possible to orientate it (by calculation from random Laue photographs) so as to place a dyad axis vertical and thus to have four octahedral planes also vertical. The Laue photographs obtained with diamonds in this setting have recently been examined with the greatest thoroughness,<sup>1</sup> and the photographs of the particular Hannay specimen examined are typical of those found for any natural diamond, allowance being made for the difference of crystal size.

It is possible, however, by means of such Laue photographs, not merely to identify diamond as such, but also to distinguish ordinary diamonds from the rarer kind which has been classed by Robertson, Fox, and Martin<sup>2</sup> as type II, 'transparent' diamond. Ordinary type I diamonds show, in addition to the usual Laue spots, various combinations of spots and streaks whose exact origin is not yet fully understood.<sup>3</sup> These extra spots and streaks are absent from Laue photographs of type II diamonds. The difference between the two types of diamond is shown in pl. X, figs. 1 and 2. In order to take similar photographs of the exceedingly minute Hannay diamond within a reasonable time, it was necessary to use an intensifying screen placed behind the 'duplitzed' (i.e. sensitized on both sides) X-ray film, and photographs of a small natural diamond, similarly intensified, were taken for comparison purposes. It was quite clear from these photographs, two of which are shown in pl. X, figs. 3 and 4, that the Hannay diamond examined is of the rare, 'transparent' type II. Further confirmation of this fact was obtained by taking a Laue photograph of the Hannay diamond, without intensification, in such a position that the (111) plane was exactly set for selective reflection of  $\text{CuK}\alpha$  radiation. Previous comparison of a number of ordinary and rare type diamonds photographed in this position had shown that although the (111) plane of an ordinary diamond reflects strongly, yet the

<sup>1</sup> K. Lonsdale, *Proc. Roy. Soc., Ser. A*, 1942, vol. 179, p. 315. [M.A. 8-284.]

<sup>2</sup> R. Robertson, J. J. Fox, and A. E. Martin, Two types of diamond. *Phil. Trans. Roy. Soc., Ser. A*, 1934, vol. 232, pp. 463-535. [M.A. 6-6.] Further work on two types of diamond. *Proc. Roy. Soc., Ser. A*, 1936, vol. 157, pp. 579-593. [M.A. 6-494.]

<sup>3</sup> K. Lonsdale, *loc. cit.*; *Proc. Physical Soc.*, 1942, vol. 54, p. 314.



reflection from a rare type diamond is very much stronger. The selective reflection from an ordinary type diamond is subject to what is known as 'extinction', whereas in the rare type diamond 'extinction' is small or absent.<sup>1</sup> One consequence of this fact is that the 111 selective reflection from a rare type diamond, as recorded on a photographic film, is a spot surrounded by a number of small concentric rings, the result of the diffraction of the intense reflected beam by the black paper cover of the film-holder. These rings are much more difficult to observe in the case of an ordinary type diamond, and it is seldom that more than one is visible. The Hannay diamond, in spite of its extreme smallness, gave a 111 selective ( $\text{CuK}\alpha$ ) reflection of such intensity that not only had photographic reversal occurred, but the first *four* black paper rings were easily observable. These indications that Hannay's artificially prepared diamond is of the more unusual 'transparent' type, are consistent with the observation that these diamonds are optically isotropic, whereas ordinary type diamonds generally show restoration of light, often to a considerable degree.<sup>2</sup>

The rarity of these 'transparent' diamonds (so-called because of their unusual transparency in certain regions of the infra-red and ultra-violet spectrum) may be judged from the fact that Robertson, Fox, and Martin (*loc. cit.*), having obtained one such diamond, examined between two and three hundred diamonds without finding another, although on further search they obtained five large and some small diamonds of the rare type, some from the Cape and others from Brazil. Raman,<sup>3</sup> after examining several hundred diamonds at the Panna mines in Central India, obtained four plates of the 'transparent' type. Chesley,<sup>4</sup> in the course of an investigation of the minor elements in diamonds from various sources, found one 'transparent' diamond, and it is noteworthy that it was the purest of all the diamonds he examined. The absence of 'extinction' suggests that the 'transparent' diamonds are of a more 'mosaic' structure than ordinary type diamonds, and it is certain that crystals formed under laboratory conditions in which only small amounts of material are available and slow uniform rates of change cannot be maintained for any considerable lengths of time, are much more likely to be 'mosaic' than 'perfect' in crystallographic structure. It is not surprising, therefore, that the Hannay diamond should prove to be of the 'mosaic', type II variety. Whether any of these artificial diamonds show anomalous spectroscopic effects cannot, unfortunately, be determined, as they appear to be much too small for this kind of examination. No fluorescence could be observed when they were irradiated by X-rays (colourless diamonds of both types examined by one of us (K. L.) have always shown a blue-grey fluorescence under irradiation), but this was possibly also due to their smallness, though it might signify the absence of some impurity normally present in natural diamonds.

<sup>1</sup> The existence of extinction is the result of what the X-ray crystallographer calls crystal 'perfection'. By this is meant a uniformity of internal structure, without the slight changes of orientation or position that exist between the component crystallites of a 'mosaic' crystal. A diamond may be 'perfect', in this sense, and yet show optical anisotropy, because of the existence of a uniform internal strain; or it may be 'mosaic', and yet optically isotropic because the individual crystallites are unstrained.

<sup>2</sup> Compare G. Friedel, *Bull. Soc. Franç. Min.*, 1924, vol. 47, p. 60 [M.A. 4-292]; Robertson, Fox, and Martin, *loc. cit.*; C. V. Raman, *Current Sci.*, Bangalore, 1943, vol. 12, p. 41.

<sup>3</sup> C. V. Raman, *Current Sci.*, Bangalore, 1942, vol. 11, p. 261.

<sup>4</sup> F. G. Chesley, *Amer. Min.*, 1942, vol. 27, p. 20. [M.A. 8-268.]

Many of the Hannay diamonds show the (111) striations remarked on by Robertson, Fox, and Martin, as being typical of type II diamond. The interval between the striations is about  $5\mu$ . The diamond shown in text-fig. 2 when examined by transmitted light reveals the cross-hatched appearance of two sets of (111) cleavage striations.

We have definitely proved, therefore, that all but one of the specimens labelled as having been made artificially by J. B. Hannay in 1880 and presented by him to the British Museum are genuine diamond, one at least being a type II ('transparent' or 'mosaic') diamond. There seems to be no reason to doubt the genuineness of the specimens themselves; they correspond to Story-Maskelyne's description of them, as given in his letter to *The Times*; and various inquiries that we have made indicate that Hannay himself was a scientific man of integrity and that he genuinely believed that he had succeeded in his most difficult investigation. A suggestion made to us that these diamonds may have been fragments thrown into the tube by a workman who was tired of continual explosions, or 'seeds' put in to start crystallization, is negatived by the fact that Hannay obtained the diamonds from the solid mass at the *upper end* of the tilted tube and it is also rather improbable in that case that any of the 'seeds' would have chanced to be of the rare type II. We believe that J. B. Hannay did indeed make diamonds by a laboratory method and that the method he used is capable of giving the 'transparent' type which is apparently rare among natural diamonds, perhaps because the latter are so seldom really pure.

On the other hand, the difficulties which Hannay encountered, the low percentage of success in his numerous experiments, the failure of Sir Charles Parsons to repeat Hannay's method, and the minute amount of diamond which even the most successful experiments yielded, show that any hope of making artificial diamond on a large scale is hardly likely to be realized without considerable patience, perseverance, and expense.

#### EXPLANATION OF PLATE X.

##### *X-ray study of Hannay's artificial diamonds.*

The first four figures have been reproduced from Laue photographs comparing Hannay's diamond with types I and II of natural diamond. The details of film and crystal setting for figs. 1-4 are: crystal to film distance 3 cm.; film-normal inclined  $68^\circ$  with the forward direction of the X-ray beam, the beam inclined  $23.5^\circ$  with the (111) plane and  $11.75^\circ$  with the (110) plane, the axis [110] of the diamond being vertical.

FIG. 1. Natural diamond type I. Photograph not intensified, 30 minutes exposure. Specimen D(2) of K. Lonsdale (loc. cit.). Weight 1 mg.

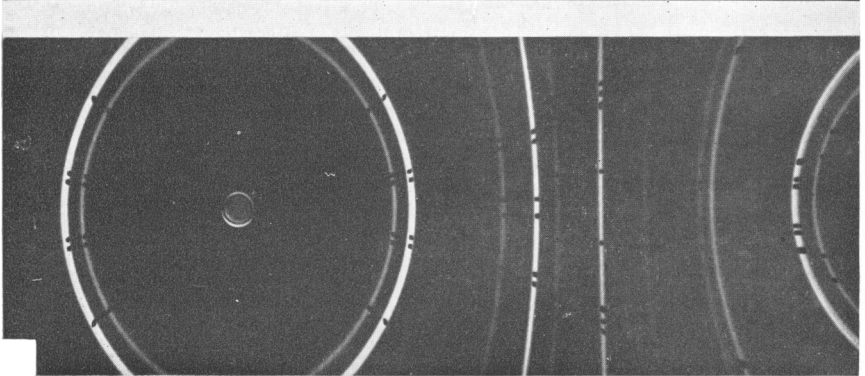
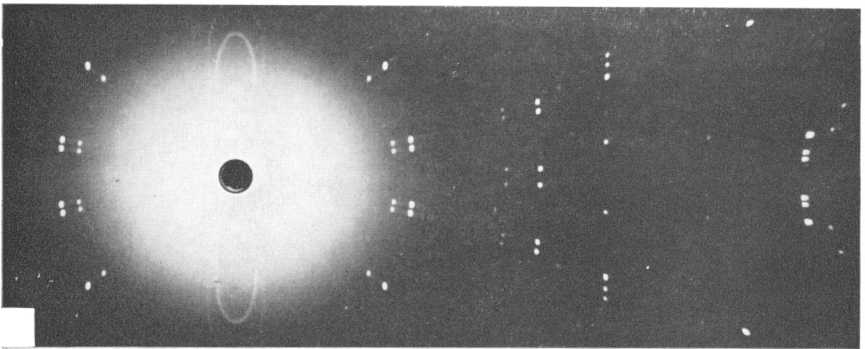
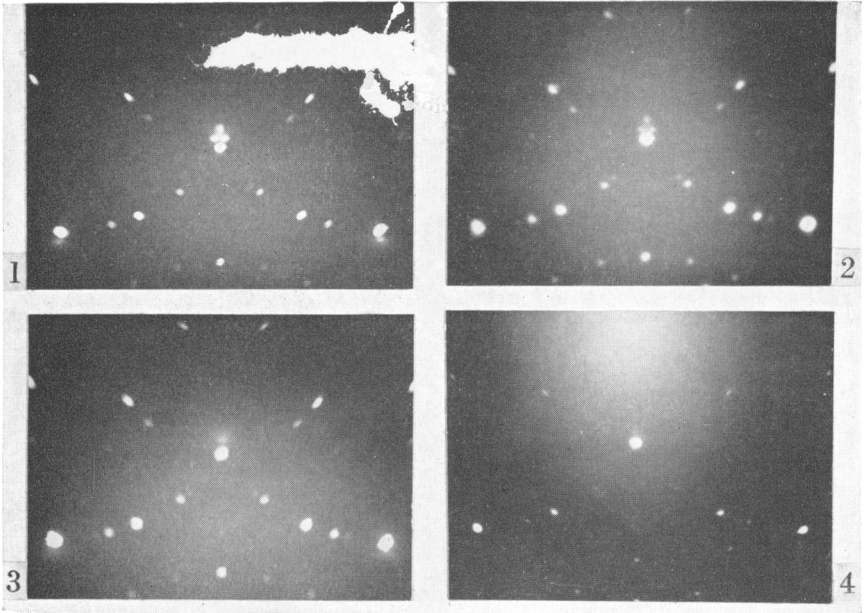
FIG. 2. Natural diamond type II. Photograph not intensified, 5 minutes exposure. Specimen D16 of Robertson, Fox, and Martin. Weight 0.6149 gm.

FIG. 3. Natural diamond type I. Photograph intensified by fluorescent screen, 5 minutes exposure. Specimen D(2). Weight 1 mg.

FIG. 4. Hannay's diamond (text-fig. 2). Photograph intensified, 30 minutes exposure. Weight less than 0.05 mg.

FIG. 5. Rotation photograph of Hannay's diamond (text-fig. 2) mounted in random orientation. Unfiltered copper radiation, camera diameter 6 cm. Actual size.

FIG. 6. Spots of fig. 5 reversed and superposed upon a powder photograph of natural diamond (commercial powder kindly supplied by Mr. B. W. Anderson).  $\text{CuK}\alpha$  and  $\text{CuK}\beta$  lines are present for the diffractions 111, 220, 311, 400, and 331.



F. A. BANNISTER AND K. LONSDALE: X-RAY PHOTOGRAPHS OF HANNAY'S ARTIFICIAL DIAMONDS