

The Early History of Manufactured Diamond

By James Evans, EG

The first manufacture of diamond is widely credited to Erik Lundblad and the Swedish company ASEA for their success in 1953. Less well-known are the incremental steps taken by scientists over the previous 250 years...

The Adamantine Matter

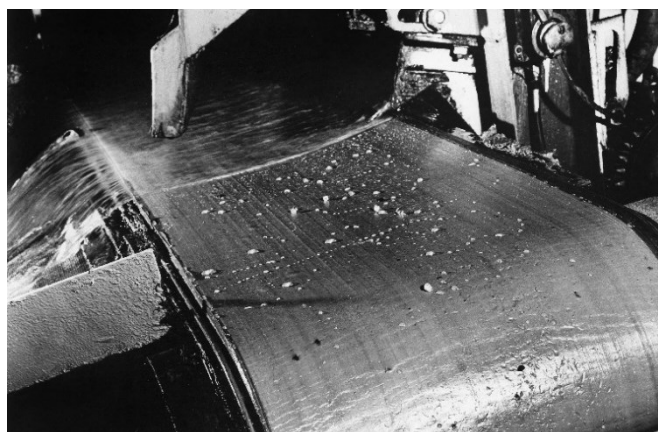
Isaac Newton took an early step to identify the constituent matter of diamond, which would soon be known as “adamantine matter”. In his treatise on optics from 1704, Newton wrote:

[...] the refraction of Camphire, Oil-Olive, Lintseed [sic] Oil, Spirit of Turpentine and Amber, which are fat sulphurous unctuous [i.e. oily] Bodies, and a Diamond, which is probably an unctuous substance coagulated, have their refractive powers in proportion to one another as their densities without any considerable variation. [...]

It seems rational to attribute the refractive power of all Bodies chiefly, if not wholly, to the sulphurous parts with which they abound. And as Light congregated by a Burning-glass acts most upon sulphurous Bodies, to turn them into fire and flame; so, since all action is mutual, Sulphurs ought to act most upon light. (Newton, 1704, pp.75-76)

In reality, there is little causal relationship between a material’s refractivity and its combustibility. But in other respects, Newton was correct in surmising that:

1. if diamond were viscous, it would likely be considered an oil (given its high carbon content, hydrophobicity, and attraction to other oils); and
2. diamond is combustible (although not to the extent Newton imagined).



Diamonds being separated on a grease belt.¹

One of the first demonstrations of diamond’s vulnerability to heat was provided in 1694-95, when the Italians Giuseppe Averani and Cipriano Targioni placed a diamond beneath the Bregans Lens (a large burning-glass that was owned, along with the diamond, by Cosimo III de’ Medici).² However, rather than turning into ‘fire and flame’ as Newton had predicted, the diamond was merely ‘exhaled away’ (Averani; Targioni, 1711, p.232).

The possible fate of Cosimo's diamond was further investigated by the French chemist Jean Darcet in 1768. Darcet had placed two small diamonds in separate porcelain crucibles: one with a tightly-fitting lid; the other with a pierced lid to permit the flow of air. He then subjected the two crucibles to the violent fire of a porcelain furnace, after which neither diamond remained. Darcet concluded, quite reasonably, that his diamonds had evaporated – for combustion should not have been possible without a supply of air (as in the closed crucible).

Two years later, Darcet repeated his experiment at the request of the French Academy of Sciences. This time, the closed crucible was replaced by a ball of porcelain paste encasing one of the diamonds. The results were the same. Nevertheless, as Darcet's experiments were reworked by others in the scientific community, some curious results emerged. Most curious of all were the findings of Pierre-Joseph Macquer, who employed a 'muffle furnace' in his experiments. This apparatus had separate compartments for the burning fuel and the specimen, allowing Macquer to open his 'muffle' at the moment of the diamond's destruction to observe the stone surrounded by flame!

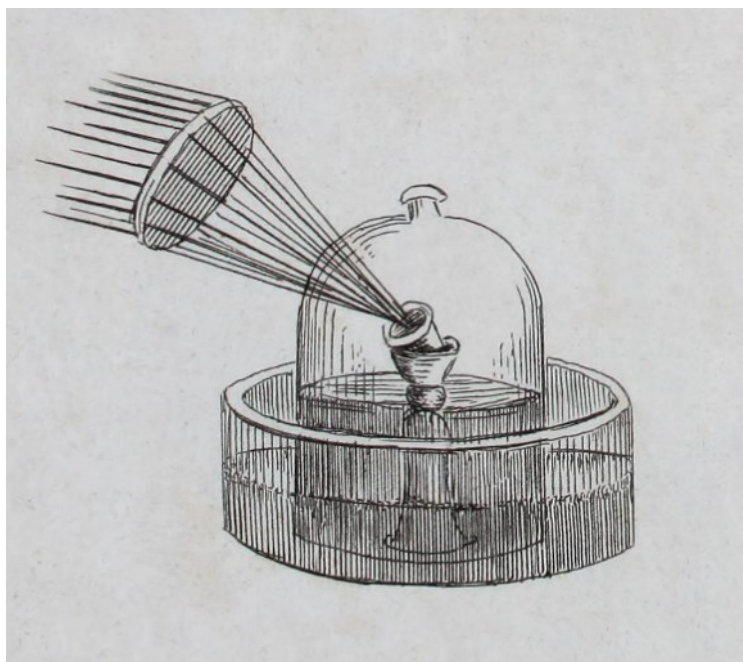
Despite Macquer's observation, the nature of diamond's destruction continued to be debated. The Academy's policy on such topics was for investigations to be carried out co-operatively (Guerlac, 1961). And so it was that, in 1772, Macquer began collaborating with Louis Claude Cadet de Gassicourt (Cadet) and Antoine-Laurent Lavoisier. The group set themselves the challenge of distilling vapours from the evaporation of diamond. But, after placing their diamonds in a ceramic retort and subjecting this to a very violent fire for three hours, no vapours were obtained. Instead, *'the diamonds were found in the retort, almost as they had been put in, but nearly all frosted, coated with a kind of black-brown varnish like the inside of the retort; but they had lost more than two grains of their weight and had begun to melt'* (L'Académie Royale des Sciences, 1772, pp.18-19).

The manner of diamond's destruction was finally settled with the assistance of a Parisian jeweller, Monsieur Maillard. Not satisfied with Darcet's original closed crucible (with the tightly fitting lid), Maillard placed three diamonds in a ceramic pipe-head filled with powdered charcoal (which would have absorbed any oxygen that reached the pipe-head). This was then sealed with a strip of sheet metal, surrounded by silica sand, placed in a crucible coated with chalk, then sealed between two further crucibles! Not all of the protective layers survived two hours in Macquer's large furnace, but the pipe-head and its contents did, proving beyond doubt that diamond could withstand extremely high temperatures when sheltered from air.

What had caused the initial confusion? Further experiments by Macquer demonstrated that porcelain paste was not impermeable, as had been assumed. Given that diamond had survived in a ceramic retort, its destruction in Darcet's closed crucible was presumably due to some small gap between the crucible's base and its lid (however tightly-fitting the lid was reported to be).

A further contribution from Antoine Lavoisier was then to place several diamonds in a porcelain crucible, which sat atop a glass pedestal, in the middle of an earthenware bucket filled with distilled water. A glass bell-jar was then placed over the diamonds before the Lens of the Royal Palace was focussed upon them. The point of the experiment was seemingly to smell the odours arising from diamond's destruction. But no odours were produced. Nevertheless, after the apparatus had cooled, Lavoisier noticed that the water level had risen inside the bell-jar. This led to further investigation. Lavoisier poured 16 ounces of limewater (calcium hydroxide) into the jar and found a cloudy solution (calcium carbonate) arose, as would be expected from air after the combustion of charcoal. He therefore concluded:

According to this experience, it is difficult not to believe that the air in which one has evaporated diamond, acquires at least in part the properties of fixed air [now known as carbon dioxide]. (L'Académie Royale des Sciences, 1772, p.26)



**The apparatus used by Lavoisier
in his combustion of diamond.³**



The Lens of the Royal Palace.⁴

An Englishman, Smithson Tennant, took the final step to identify ‘adamantine matter’ in 1796 (three years after Lavoisier’s execution during the French Revolution). Replicating Lavoisier’s quantitative approach, Tennant weighed the fixed air produced from diamond’s combustion and found it equal to that produced by the same weight of charcoal, concluding that:

[...] the nature of the diamond [...] consists entirely of charcoal, different from the usual state of that substance only by its crystallized form. (Tennant, 1797, p.124)

The Creation of Graphite

Having discovered the composition of diamond to be carbon, at the time known as charcoal, the next challenge was to manufacture it. But before getting to this, we must first consider another form of carbon: graphite.

Carl Scheele identified graphite as a form of carbon in 1779. Scheele had no access to the extravagant laboratory equipment enjoyed by Lavoisier. Instead, he burnt a mixture of ‘plumbago’ (as both graphite and the ores of lead were known) and arsenic acid within a small retort of thick glass, upon which a large ox bladder was bound. The role of the arsenic acid was to provide sufficient oxygen for the combustion of graphite. Or, as Scheele would have understood it, to absorb phlogiston, for he believed, like others at the time, that combustion released phlogiston rather than absorbing oxygen. But regardless of the prevalent theory of combustion, Scheele’s finding was that the heated ‘plumbago’ (graphite) had given off pure ‘aerial-acid’ (as carbon dioxide was known).



Scheele’s apparatus.⁵

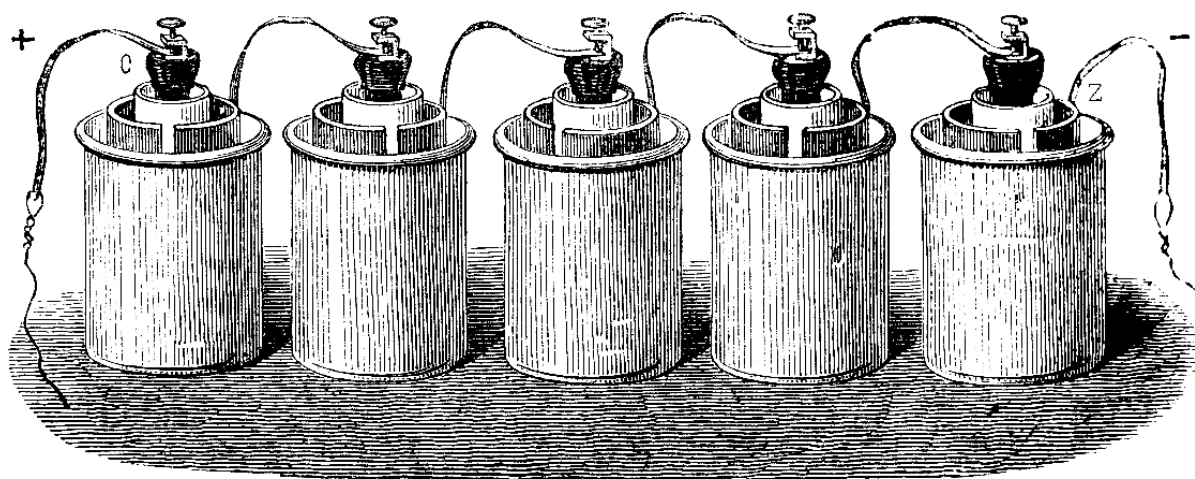
I am convinced that plumbago is a kind of mineral sulphur or carbon, the constituents of which are aerial-acid, combined with a large amount of phlogiston.

(Scheele, 1779, p.243)

Seven years later, three Frenchmen, Messieurs Vandermonde, Berthollet and Monge, found that blades of plumbago would form from molten cast iron as it cooled, and that ‘the quantity of this substance is always greater when the cast iron is greyer, that is to say, when more charcoal has been placed in the charge of the furnace’ (Vandermonde et al., 1786, p.136). The trio correctly identified the blades as being the same material used in

England to make pencils. But it wasn't until the end of the 1780s that the German mineralogist, Abraham Gottlob Werner, labelled the material as 'graphit' (from the Greek 'graphein', to write).

By the late 1840s, César-Mansuète Despretz (another Frenchman) was investigating the manufacture of diamond with an influential new tool: the battery. At the start of the decade, the German chemist Robert Wilhelm Bunsen had improved upon the existing Grove Cell by replacing its expensive platinum cathode with carbon. The lower price of the new Bunsen Cell meant many more cells could be joined together to form a powerful battery (also known as a 'pile'). Despretz used his Bunsen Pile to pass electricity through numerous varieties of carbon, at pressures of up to three atmospheres, to find that *'diamond itself changes, like the other species of coal, into graphite'* (Despretz, 1849, pp.46-47).



A Bunsen Pile.⁶

It had therefore been established that charcoal, graphite and diamond were three forms of the same material. But whilst graphite had been created, first from charcoal and then from diamond, a method to manufacture diamond remained elusive. Despretz had already employed a Bunsen Pile with 600 cells in his attempts to form diamond. Having failed, he concluded:

[...] diamond is not like the graphite that can be produced artificially in blast furnaces, the product of the action of intense heat on organic or carbonaceous matter. Rather, it would appear [...] to have a vegetable origin and to have been initially in a state of softness and to have hardened gradually as one sees a gum harden [to amber].
(Despretz, 1849, pp.46-47).

The Creation of Diamond

Despretz was following the work of Scottish physicist David Brewster when he suggested a vegetable origin for diamond. Although this now seems a fanciful notion, Brewster supported his argument with reference to ‘mellite’, a mineral with a distinct crystalline form that does emerge from vegetable matter (i.e. coal) *‘by the influence of time, and the slow action of corpuscular [i.e. molecular] forces’* (Brewster, 1835, p.457).

Brewster’s rationale was that diamond’s properties were, in some respects, similar to those of amber:

- they were found in similar localities;
- they were both based on carbon;
- they behaved similarly under a polariscope; and
- most significantly, both contained inclusions of air; the expansive force of which could induce strain patterns surrounding the inclusion (when viewed through a polariscope).

On this final point, Brewster believed that strain anisotropy could *‘arise only from the expansive force exerted by the included air in the diamond and the amber, when they were in such a soft state as to be susceptible of compression from so small a force. That this compressible state of the diamond could not arise from the action of heat is manifest from the nature and recent formation of the soil [i.e. the matrix of sedimentary rock] in which it is found’*. Brewster further stated: *‘It is obvious that such an effect cannot arise from any mode of crystallization; [...] I have never observed the slightest trace of it in more than 200 mineral substances which I have examined, nor in any of the artificial salts from aqueous solutions’* (Brewster, 1835, p.456).

Having first proposed a vegetable origin of diamond in 1820, Brewster updated his work in 1835 to consider new evidence. A certain Captain John Franklin had found diamond within a new, possibly igneous, matrix from Bundelkhand (India), and suggested it had withstood the heat required to produce its rocky matrix because of a great pressure. Furthermore, Franklin surmised that this pressure might also account for the gem’s original crystallisation. Brewster admitted this was quite conceivable, and that fusion under pressure could account for the gemstone being in a sufficiently soft state to produce strain anisotropy. But he did not believe it, for it was a fact that ‘compressive cavities’ did not occur in minerals of igneous origin.



A rough mellite crystal.⁷

By 1862, Brewster had changed his mind. Having re-examined several plates of topaz, he realised that his 'compressive cavities' were frequently seen in this mineral also (Brewster, 1862). As such, the most notable attempts at creating diamond would henceforth operate under increasing levels of pressure...

A compatriot of Brewster, James Hannay, claimed to have manufactured diamond in 1879. His method was later described by Gardner Williams, as follows:

A [wrought iron] tube twenty inches long by four inches in diameter was bored so as to have an internal diameter of half an inch [and thus walls of 1 ¾ inches]. In the tube was placed a mixture of ninety per cent of rectified bone oil, and ten per cent of paraffin spirit, together with four grams of the metal lithium. The open end of the tube was welded air-tight, and the whole mass was heated to redness for fourteen hours.

(Williams, 1906, p.138).

Hannay reported the results of his experiments as being small, transparent pieces of "crystalline carbon", stuck to the sides of the tube within a black mass of iron and lithium. However, the majority of his account focussed on the carnage wrought by numerous violent explosions – hardly encouraging for those looking to validate his work. Added to the significant risk of injury was the risk of failure, for Hannay reported just three successful experiments out of eighty. It was almost as if he was trying to put people off! Later tests would find that Hannay's diamonds were in fact natural (Davies, 1984), presumably added by Hannay at the start of his experiments.



**One of Hannay's diamonds
(approx. 0.4 mm long).⁸**

Henri Moissan claimed to have manufactured diamond in 1893. By the time of his experiments, diamonds had been discovered in the 'blue earth' (i.e. Kimberlite) of South Africa and in the meteorites of Canyon Diablo, Arizona. Both sources supported the role of high pressures in the stone's formation. To replicate these conditions, Moissan dissolved sugar charcoal (a pure form of amorphous carbon derived from sugar) in a crucible filled with molten iron, then plunged the crucible in water. Cast iron expands as

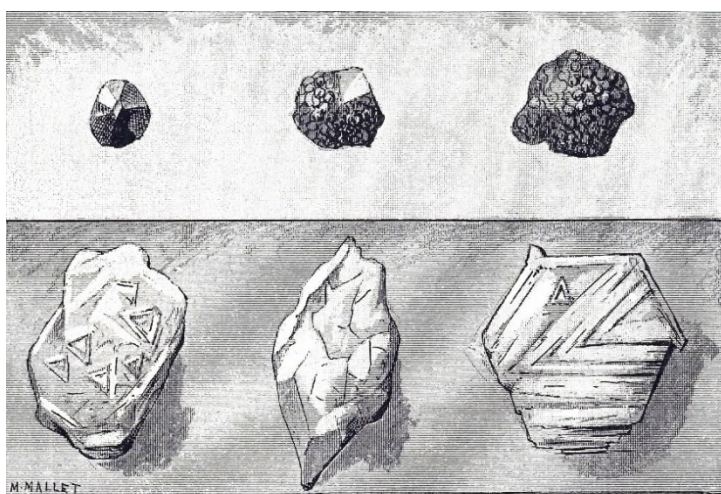
it solidifies then contracts as it cools. Moissan therefore believed the outside of the iron mass would be contracting whilst the inside was expanding, creating an immense pressure within the crucible. His approach was reported to produce graphite, brown charcoal, and *'a small quantity of a fairly dense carbon which, when separated from its iron matrix by a series of acid baths, displayed all the characteristics of diamonds'*. These characteristics were listed as:

- a specific gravity close to 3;
- being harder than ruby; and
- being entirely combustible in oxygen.

Of these, Moissan considered combustibility the most important, for the other characteristics were shared with a *'number of metallic silicides and carbides'* (Moissan, 1893, pp.221-224).



**Henri Moissan quenching cast iron
(next to the arc furnace he invented).⁹**



Some of the crystals produced by Moissan.¹⁰

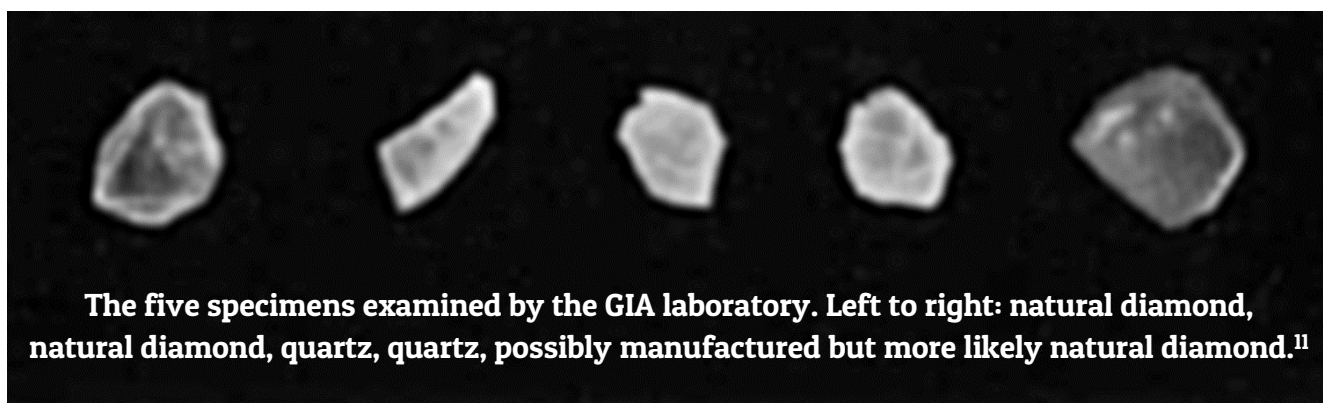
The crystals produced by Moissan were comprised of two groups, described by Moissan as having *'a chagrined appearance and a shade of grey black identical to that of certain carbonados'* (pictured, top row); and *'the transparent fragments which appear broken into small pieces have a greasy appearance [... and] possess a certain number of parallel streaks or triangular impressions'* (bottom row; Moissan, 1893, pp.221-222).

Moissan's crystals, however, were not entirely combustible. Those specimens which burned left an ochre-coloured ash that Moissan considered analogous to the combustion of low-grade diamond (Moissan, 1893). His experiments would later be replicated by Charles Parsons, who found that more crystals were formed when impurities (such as silicon, aluminium, magnesium and chromium) were present in the iron; and that few crystals would emerge from pure iron. The suspicion then, was that Moissan's diamonds were in fact either carbides (such as silicon carbide, now known as moissanite) or spinels (Desch, 1928).

An approach similar to Moissan's was later employed by J. Willard Hershey and his students at McPherson College, USA. In 1940 Hershey reported that:

Since we first began our experiments in their manufacture [in 1929] more than 50 diamonds have been made synthetically [...], ranging in size from the smallest, 1 millimeter in diameter, to the largest, which is 2 millimeters by one and one half millimeters by one millimeter, weighing 1/30 carat (Hershey, 1940, p.140).

As Moissan's largest specimen had measured 0.75 mm, Hershey believed he had produced the largest manufactured diamonds on record. However, by this point several of Hershey's diamonds had already been examined by the GIA laboratory and found to be a mixture of natural diamonds and quartz. The GIA rather charitably concluded that *'there may have been too much confusion of specimens in the system followed by Hershey and his assistants'* (GIA, 1938, p.198).



At the same time as Hershey's experiments at McPherson College, Percy Bridgman was working at Harvard University developing apparatus to maintain astonishingly high pressures. By 1947, he reported that at room temperature, not even 400,000 atmospheres of pressure was sufficient to transform graphite to diamond. Yet it had previously been calculated that diamond was the preferred form of carbon at pressures of just 15,000 atmospheres (Davies, 1984). The problem was that, without sufficient heat to break apart its carbon bonds, graphite's lustrous transformation to diamond was occurring at a negligible pace. But to maintain both high temperatures and pressures was a formidable engineering challenge.

Success would finally be achieved in 1953. On 15th February that year, Erik Lundblad and the Swedish company ASEA employed their ‘sphere’ apparatus – designed for the purpose by the Swedish inventor Baltzar von Platen – to produce the first ‘high pressure, high temperature’ manufactured diamonds.¹² Lundblad had replaced the cast iron employed by Vandermonde, Berthollet and Monge with cementite – an iron carbide with a much higher density. But his method was in some senses an evolution of their work, along with that of: Newton, Averani and Targioni, Darcet, Macquer, Cadet, Lavoisier, Maillard, Tennant, Scheele, Despretz, Brewster, Franklin, Hannay, Moissan, Bridgman and von Platen. The slow progress of science and engineering over a quarter of a millennium had finally revealed the secret to creating diamond.

Notes

¹ Image by Granger Historical Picture Archive / Alamy.

² The Bregans Lens was reported to have had a diameter of ‘*two thirds of a Florentine arm*’; equivalent to around 40cm (Averani; Targioni, 1711, p.225). However, as the burning-glass is still in existence, we know the actual diameter of its lens to be 45cm.

³ Image from Lavoisier (1789, plate IV). Edited by the author.

⁴ The Lens of the Royal Palace (then owned by the Royal Academy of Sciences) was made by the German mathematician Ehrenfried Walther von Tschirnhaus and had a diameter of 89cm (and thus a surface area nearly four times as great as the Bregans Lens). This lens is not to be confused with ‘The Great Burning Glass of Mr Trudaine’, which would have a diameter of roughly 260cm (and thus a surface area thirty-three times as great as the Bregans Lens). Image by Michael Lange, provided by the mathematics-physics department of Staatliche Kunstsammlungen Dresden (the museums of the Staatliche Kunstsammlungen, Dresden).

⁵ Image from Scheele (1777). Edited by the author.

⁶ Each cell of this Bunsen Pile contains a carbon cathode at its centre, immersed in nitric or chromic acid, and surrounded by a tubular zinc anode in sulphuric acid. The two acids are separated by a ceramic pot through which ions, and thus an electric charge, can pass. Image from Figuier and Nansouty (1911).

⁷ Image by Rob Lavinsky / iRocks.com, used under Creative Commons licence [CC BY-SA 3.0](https://creativecommons.org/licenses/by-sa/3.0/). Edited by the author.

⁸ Image by The Natural History Museum (London) / Alamy (collection number: BM.87756).

⁹ Image by Archive Pics / Alamy.

¹⁰ Image by World History Archive / Alamy.

¹¹ Although five stones were examined by the GIA, Hershey later acknowledged that the first specimen was natural. It might be questioned how the GIA laboratory could have identified manufactured diamonds – assuming none had ever been produced. The answer is that inclusions were observed within Hannay’s stones that were typical of natural specimens. The uncertain verdict on the fifth stone was explained as follows:

‘The surface is not sufficiently clear to allow a detailed study of the inclusions to be made, but from its appearance this stone might possibly be diamond produced by

Dr. Hershey's method. However, in view of the outcome of the tests on the other stones of this group, plus the appearance of the specimen, which likewise is quite similar to that of many South African stones, its synthetic [i.e. manufactured] origin may reasonably be doubted'. (GIA, 1938, p.197).

Image courtesy of GIA (GIA, 1938, p.195). Edited by the author.

¹² Although a significant achievement, Lundblad's creation of diamond was not actually the first. Just one month earlier, William Eversole and the Union Carbide Corporation had achieved new growth atop a seed of natural diamond, using a low-pressure technique of Chemical Vapour Deposition (Angus; Hayman, 1988). Lundblad and ASEA were, however, the first to create diamond without a natural diamond seed.

For a full account of Lundblad's work, see *Diamonds* by Gordon Davies.

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