Diamonds in NATURE

A recently rediscovered recipe for synthesizing diamonds at low pressures may actually work

By IVARS PETERSON

An eccentric inventor, a private laboratory and a secret recipe for making diamonds seem more like the ingredients for an alchemical tale or a gothic mystery. Yet these elements provide the background for an intriguing scientific puzzle.

Eighty years ago, in a brief letter published in the Aug. 24, 1905 NATURE, physicist Charles V. Burton claimed he had synthesized microscopic diamonds. His unusual method involved dissolving carbon in a molten lead-calcium alloy, then forcing the carbon to crystallize out of solution—in the form of diamonds.

Although Burton promised "shortly to return to the subject of these experiments," he published nothing more on this topic. No abstracts of his original letter appeared in the three major journals that in those days monitored chemical research. His name practically disappears from later books and papers on carbon and synthetic diamonds.

One of the few references to Burton's chemical work appears in the recent book *Diamond* (Adam Hilger Ltd., Bristol, 1984), written by Gordon Davies of King's College in London, England. Davies lumps Burton with several other turn-of-the-century investigators who all tried various ways of getting diamond crystals out of solution—apparently unsuccessfully.

A few years ago, Felix Sebba, a chemical engineer at the Virginia Polytechnic Institute and State University in Blacksburg, Va., stumbled across Burton's work. "I found it by accident," says Sebba. "I was intrigued by it. It struck me that here was a thing that had been completely lost."

More as a joke than anything else, Sebba applied to the National Science Foundation (NSF) for a small grant to see if Burton's method really worked. To his surprise, NSF provided funds, and together with graduate student Neal Sugarman, Sebba tried to duplicate the process.

"There were certain difficulties because there was so little information given in [Burton's] letter to NATURE," says Sebba. "We had to do some research on our own to solve the problems." In the end, the researchers managed to obtain small quantities of microscopic crystals, a few micrometers in size, that looked like diamonds.

"I'd say I'm about 90 percent certain that they're diamonds," says Sebba. The black powder in which the crystals are found can scratch glass, and the crystals themselves have a high refractive index. X-ray diffraction studies also point to the presence of diamond.

Sebba's method, as outlined last month in a letter in the July 18 NATURE, involves dissolving a small amount of calcium carbide in molten lead. When steam at 550°C is passed over the alloy, it reacts with the calcium (but not with the lead) to form calcium hydroxide, which is not soluble in lead. The calcium hydroxide comes out to form a layer of scum on the metal's surface.

But carbon by itself is less soluble in lead than in a lead-calcium alloy. The removal of calcium forces some of the carbon present to crystallize. When the resulting gray crust is treated to remove all traces of lead and calcium salts, the product is a considerable amount of black powder containing tiny diamondlike crystals.

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Artificial Diamonds.

Of the two phases, diamond and graphite, diamond is the denser, and has also the less internal energy. It follows that, if carbon can be crystallised at comparatively low temperatures, the minimum pressure sufficing to determine the diamond form will be lower than that employed in M. Moissan's experiments.

For estimating the transformation temperature corresponding to low pressures, the data available are incomplete; it is here suggested, however, as a tentative result from experiments which are still in a preliminary stage, that the transformation temperature corresponding to atmospheric pressure lies somewhere between 550° C. and 700° C., or not far outside those limits, temperatures having so far been judged only by eye.

A molten alloy of lead with about 1 per cent. calcium appears to be capable of holding in solution some small proportion of carbon, which exists either as free carbon or as calcium carbide; and if the calcium is eliminated from the molten mass, some carbon crystallises out. Steam, for example, converts the calcium into hydrate without attacking the lead. If the reaction has occurred at a full red heat, graphite is found in the crust of lime; if only a very low red heat has been attained, no graphite is found, but a number of very small or microscopic crystals, which have many of the properties of the diamond. The illustration is from a pencil drawing of a very minute crystal, viewed under a magnification of 80 diameters, and drawn on a greatly enlarged scale.

FIG. 1.—Supposed diamond from lead-calcium-carbon solution.

The crystals obtained exhibit mostly faces of the octahedron, modified by the cube and dodecahedron; in no case has any internal flaw or lack of perfect transparency been detected in them. The refractive index is clearly very high, and an attempt to determine it by displacement of focus gave 2·43 (instead of 2·47), any convexity of the refracting surface tending to give too low a value. The crystalline faces are, in fact, generally if not always convex, in many cases strongly so. The crystals adhere tenaciously to clean, dry glass; they are unacted upon by ordinary acids (hot or cold), by cold hydrofluoric acid, and by fused [illegible] at a red heat.

Although almost all current processes for making synthetic diamonds involve high temperatures and high pressures, Sebba says his experiment shows that diamonds may form under other conditions. Applying an old chemical idea that "metastable" forms of a substance sometimes appear before the final stable form is achieved, Sebba argues that at 500°C, the conversion of metastable diamond to stable graphite is so slow that diamond crystals can be detected.

"This principle doesn't appear in any modern textbooks, so people don't know about it," says Sebba. "I'm pretty ancient now, so I do know about it. It struck me at once that there was no scientific reason why diamond shouldn't come out first."

Sebba now wants to redo his experiments more carefully under better controlled conditions. "I want to see exactly what we've got," he says, "and I would like to see if we can make the diamonds bigger."

Equally intriguing to Sebba is "the fact that what could have been a very important and certainly very romantic discovery was so completely ignored and so completely forgotten." Very little is known about the circumstances that led Burton, who was already recognized as an ingenious inventor, to do his diamond-making experiments, apparently on his own time at a friend's laboratory. Then, shortly after the publication of his letter, Burton mysteriously left the instrument company where he was employed and devoted his time, until his death in a 1917 automobile accident, to theoretical studies of gravity and the aether, a light-carrying medium once thought to permeate all space.

"I'm very keen to give him some sort of posthumous recognition," says Sebba. "It was a very ingenious idea. I often wonder how many other useful discoveries have been lost, simply because they have been neglected or haven't been properly abstracted." □

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