Memo Report C-55-3

CHEMISTRY RESEARCH DEPARTMENT

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A SUCCESSFUL DIAMOND SYNTHESIS

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Abstract: Diamonds have been grown, using graphite as a source of carbon, at 95,000 atmospheres and temperatures near 1700ºC. The crystals grow rapidly (two or three minutes). The process has been duplicated. The apparatus used has been described elsewhere. •

• The "Belt" Ultra-High-Pressure, High Temperature Apparatus, H. Tracy Hall, G.E. Research Laboratory Report No, 1064.

Diamonds were first synthesized by the process described below on December 16, 1954, At this writing, the process has been successfully repeated twelve times by the author, Dr. H. H. Woodbury on December 31, 1954 duplicated the author's results for patent purposes.

The diamonds were identified by the following means:

(a) Crystals are unequivocally diamond by X-ray,
(b) burn in oxygen leaving no residue,
(c) scratch polished boron carbide plate (only diamond does this),
(d) sink in methylene iodide (diamond dens. = 3.51, methylene iodide density = 3.32),
(e) are isotropic (as diamonds must be),
(f) have characteristic diamond habit, (tendency to grow as octahedra with very characteristic triangular faces, triangular pits and growth Patterns),
(g) are transparent by transmitted light,, have characteristic adamantine luster and give almost metallic reflection (because of high refractive index) from direct incident light,

The diamonds grow as polycrystalline masses. These masses have approached 1/4 carat in weight. Individual crystal faces protruding from the polycrystalline mass have edges up to 300 microns long. Some crystals are shown in the photograph accompanying this report.

In the diamond synthesis the "Belt" ultra-high-pressure, high temperature apparatus was employed.[1]

A pressure of 95,000 atmospheres was used. Temperatures between 1600*C. and 1800*C. were satisfactory. A graphite tube 0.450" long, 0.125" 0.D. and 0.085" I.D. was filled with iron sulfide. Graphite plugs were placed in the ends of the tube, Tantalum end disks were used to carry heating current to the tube (see ref. 1). After holding at temperature for three minutes, temperature was lowered to room temperature followed by reduction of the pressure to one atmosphere. Examination disclosed diamonds growing in the ends of the tube next to the tantalum end disks.

The Process of Diamond Growth

Previous work on compressing graphite to 100,000 atmospheres at temperatures up to more than 3000*C showed that diamond would not form by direct transformation.[2]

When a process will not proceed at a measurable rate even though thermodynamic conditions are favorable, a catalyst is sought. In the diamond synthesis reported here, experiments performed so far indicate that TaC (formed from the tantalum end disks) in conjunction with either iron or iron sulfide act as catalyst for the conversion of the graphite to diamond. A larger yield of diamond is obtained when iron sulfide is used than when iron is used. If sulfur alone is substituted for the iron sulfide no diamonds are grown.

Now that this catalyst combination has been found, obvious extensions become apparent. All the heavy metals that form carbides and the transition metals (particularly around iron) and their compounds, become candidates for investigation as catalysts. Further work will proceed in this direction and should lead to an elucidation of a detailed mechanism of the diamond growth.

The extremely high pressure and the high temperature used in this synthesis may not be necessary. Experiments are planned in which the pressure and the temperature will both be reduced until diamonds are not made. This work will then place bounds on the region of synthesis.

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HTH/gab

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[1] H. Tracy Hall, General Electric Research Laboratory Report No, 1064.

[2] Only a portion of this work has been published. See "Attempt to Synthesize Diamond," H. Tracy Hall, Memo Report C-54-51.

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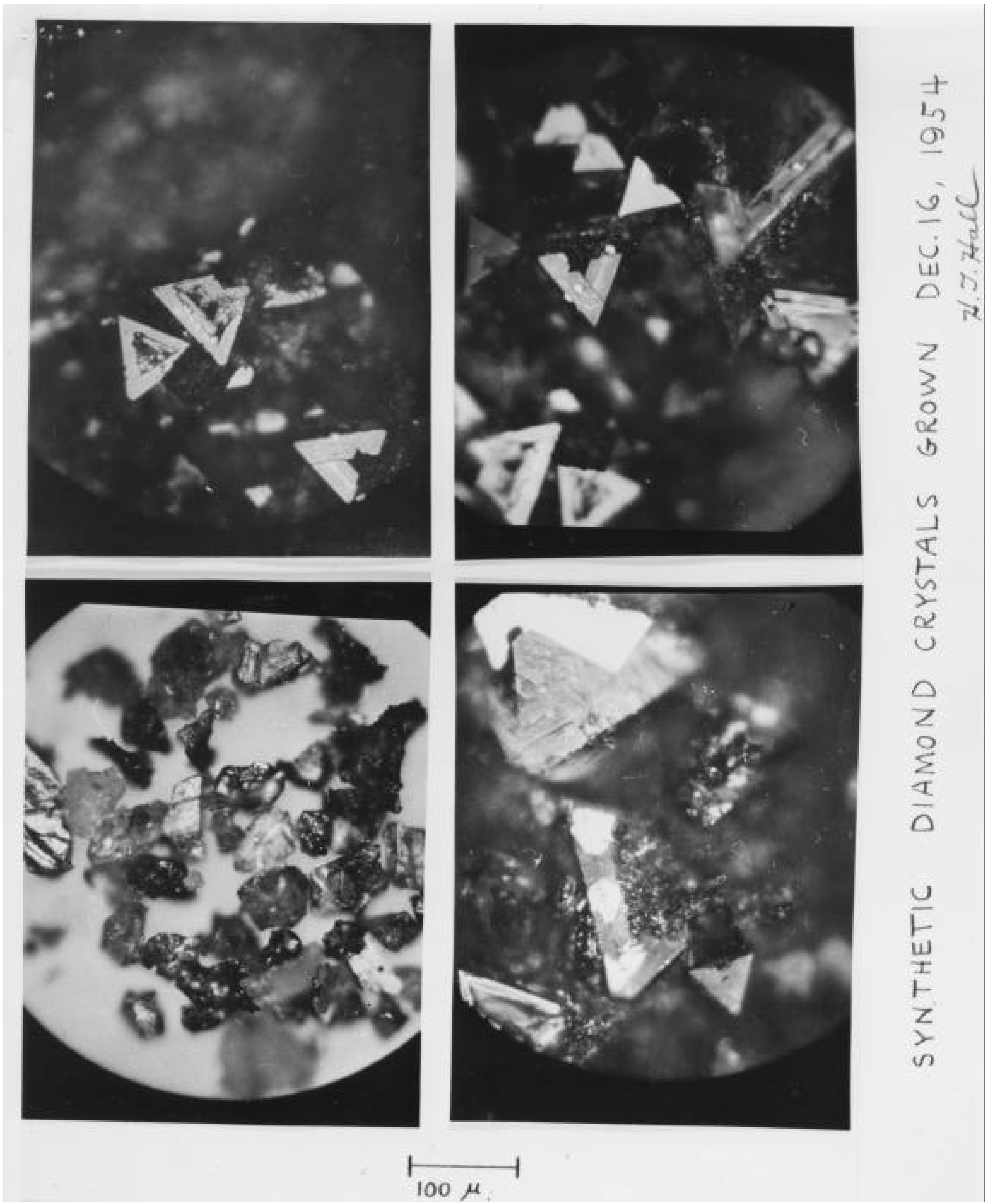
SYNTHETIC DIAMOND CRYSTALS GROWN DEC. 16, 1954
H. T. Hall
100 μ.