Technical Staff Meeting – February 14, 1955 – The day before the announcement.
Converted from a Dictabelt in 11/08
(Portions of this meeting)

1st Dictabelt
(Moderator)
The next speaker will be Tracy Hall who is going to tell you how we made the diamonds.

(H. Tracy Hall)
Dr. Bundy has outlined for you the thermodynamic requirements to be met to place a system in the diamond stable region. He has also described equivalent for meeting these requirements. In connection with this equivalent I'd like to say something about Francis. You never know how cold the water is until someone jumps in and finds out. Well, Francis jumped in. His initial work in the high temperature/high pressure field was basic to the success to this project. Now, the ideal way to make diamond would be to start with a non-diamond carbon, such as graphite, and convert it in total to diamond by subjecting it to diamond-stable conditions. This should be done at a high enough temperature such that the reaction takes place in a reasonable time. The first high pressure experiments preformed on the super pressure project were attempts to transform graphite to diamond. These were conducted in November of 1951 by Dr. F.P. Bundy in operation hot shot. Bundy shot 1/120th second pulses of current through a thin disk of graphite and a Bridgman flat-faced anvil apparatus. The pressure was 150,000 atmospheres and the temperature 1,400 degree's C. Bundy had hoped to reach 2,500 degrees C, but the resistance of the sample turned out to be about ½ of that expected and because of equipment limitations this limited the temperature to 1,400 C. The next attempts to make diamond by direct conversion were made a year ago on the belt apparatus and a little latter the same sort of experiments were conducted in the belt at 100,000 atmospheres. In these experiments pressure and temperature were maintained for an hour or more, and temperatures were very slowly reduced while maintaining the pressure. Temperatures, by conservative extrapolation ranged to as high as 4,500 degrees Centigrade. No diamond formation was observed. When a chemist, or a physicist for that matter, cannot make a reaction proceed at a measurable rate in a thermodynamically stable region, he looks for a catalyst or an indirect system to provide a favorable reaction path. Actually, this was anticipated in advance of direct conversion experiments. Dr. Wentorf may well have been the first to find such a path. In May 1953, he found faint x-ray evidence of diamond formation in a lithium carbide iron system which had been subjected to 40,000 atmosphere's at about 1,100 degrees C. The evidence was not conclusive however. Faint x-ray evidence was at about this same period also found in systems in which carbonates were reduced at high pressure and temperature. Dr. Strong, who has had a great deal of interest in iron carbon systems, was about a year ago getting some x-ray evidence of diamond formation. Again however, this evidence was inconclusive. These are just a couple of citations of the sort of thing that has been going on. Actually we have preformed hundreds and hundreds of experiments in attempts to make diamond. If you can think of a system, we've already tried it. This is the laboratory's first diamond, at least when the evidence was conclusive and unequivocal that the material is diamond. The length of this crystal is about 1.2mm. It was grown by Dr. Strong in the first

apparatus described by Dr. Bundy. In comparison to the tip of a phonograph needle you can see that it would make several of them. Let me quote in part from Dr. Strong's synthesis report: "On December the 8th 1954, two diamond octohede were placed in separate envelopes of pure iron sheet these were surrounded with a steel carbonizing mixture called speco, a mixture of charcoal, coke, barium carbonate. In this experiment a pressure of 53 and a half thousand atmospheres and a temperature of 1300 degrees C was maintained on the sample for 16 hours. Upon opening the pressure chamber the two diamond octahedral seed crystals were recovered unchanged, the iron of one of the samples had partially melted and two knew distinct diamond crystals had grown out from it. The iron then was the catalyst in Dr. Strong's reaction. We are indebted to Dr. Horn for this photograph and the photographs of diamonds that follow. At the present time it has not been possible to grow additional diamonds under the original conditions used by Dr. Strong. That is 53 and a half thousand atmospheres and 1300 degrees C. However, with suitable catalysts, diamonds are readily and reproducibly grown at 95 thousand atmospheres and temperatures between 1400 degrees and 1900 degrees C. Those temperatures is the temperature measured midway between the ends of the capsule here, in this high pressure apparatus. The temperature at the ends is approximately 150 – 200 degrees cooler than it is at the center. The first catalyst employed under these conditions was iron sulfide. Iron sulfide was rammed in through this hole in a graphite tube. After heating three minutes at 1700 degrees C and 95 thousand atmospheres, temperature was reduced to room temperature, followed by a lowering of the pressure. Diamonds were formed on these end bits, which are made of candalum. Analysis of the current bulge relationships in the heating circuit indicated that the diamonds had grown in a period of about 15 seconds. These diamonds, although tiny, were first seen with the unaided eye upon opening the capsule. This was possible because the diamonds grew with many of the triangular face of the octahedron showing. Because of diamonds' high refractive index, an almost metallic reflection was obtained from these triangles. This slide gives an idea of what was seen, here are the triangular faces of the octahedra giving an almost metallic reflection. They grow as an aggregate, a group of crystals oriented in many directions. The masses of these crystals have weighed up to a tenth of a karat. Just the other day Hal Bovenkerk succeeded in growing a mass of these crystals that weighed a quarter of a karat. These fragments were broken from the polycrystalline mass that you saw in the previous slide. Note this triangular face of an octahedron. The triangular face in this case is about 200 microns on edge. Just this past week Dr. Wentorf succeeded in growing some of these that were 1,000 microns, or four hundredths of an inch on edge. Since iron sulfide served as a catalyst in 95 thousand atmospheres and 1700 degrees C, iron alone was tried and also worked. Sulfur alone did not work. Soon after the above conditions for diamond synthesis were discovered, Dr. Hugh Woodbury duplicated the iron sulfide experiment for patent purposes. A little later the task force, Doctors Marshall, Lawson, Heb and Hollowman had Dr. Woodbury and Dr. R.A. Orianni duplicate the iron sulphide experiment using independent sources of starting materials to which we did not have access. This they did approve to everyones satisfaction that diamonds could be reproducibly made. Diamonds have now been made over 75 times at 95 thousand atmospheres at temperatures in the region of 1400 and 1900 degrees C. Everyone on the project using his own pet method is growing diamonds under these conditions. Dr. Wentorf set himself the task of classifying materials that would act as catalysts. He has found sodium, candalum, aluminum, manganese, chromium, nickel, cobalt, and palladium to be effective. Dr. Bundy is studying the iron graphite system using various means including geometrical conditions to clearly understand the mechanism of this system. Dr. Strong, with J.E. Cheney and H.B. Bovenkerk is studying iron carbon and nickel carbon systems. Lets now view some additional slides of diamonds made in the laboratory. This large one of the triangular face of a previous slide, note the triangles within the triangles. This is characteristic of diamonds made in the laboratory as well as characteristic of diamonds found in nature. Here is a diamond octohedran, I believe that you can see a couple of the triangular face’s but actually the complete thing is an octohedran. Here is the water white crystals grown by Hal Bovenkerk. They’re about as transparent as any that we have seen as yet. I believe that you can see right through some of these. The dimensions of all these crystals are about the same. Somewhere in the range of 100 and 400th’s of an inch across their longest dimension. Here is a beautiful little specimen grown by Jim Cheney. You can see the triangular face of an octohedran sticking out from this polycrystal aggregate of diamond that is growing in this ring. Here is a roundish octohedran. They’re found the same way in nature and make a strong tendency to grow around the shape of an octohedran. I believe that you can see that these are also transparent crystals. Here is some more cystalamithy on the face of another crystal. Here is another triangular face, that I believe you can make out all the depth, the focus is not to great, but that is actually another octohedran.

Now, in order to try and make this seem a little more real to you, we have an apparatus here that is used in medicine and we would like to show you some actual diamond. Dr. Horn has got some of these on a slide behind me and he is going to view these through a microscope and you are going to see them projected via this color television microscope on the screen at my right. Now these should look to you just as if you were looking right in the microscope yourself.

Tony Nerad made the remark this morning that he was not sure that this apparatus was compatible with the color of our diamond.

These yellow crystals are the diamond and they’re going into this matrix as polycrystalline aggregates. All this yellow stuff here is diamond. This is one of those larger yield variety. Jim Cheney grew this one. What you see here is a section of an arm. This cylinder inside the apparatus—the diamonds actually grew around in a circle forming what you might call a silo. So you’re viewing here a section of this silo and the material in here, slightly yellow in color, is diamond. The yellowish color is quite characteristic of diamonds found in nature. Most of the diamonds we have grown are this yellowish color. It’s quite possible that diamonds that have this yellow color have the happen to be in the presence of a small amount of iron. I believe that all the diamonds that we have grown in the presence of iron have had yellowish cache with the exception of one. I don’t know what the exception is. Now this particle of diamond has been broken from this polycrystalline aggregate as they grow on a cable. Here’s a carbon based octahedra. These again have a yellow cache. Under the microscope we should get a good view of the various crystals. These crystals are about the same size as those in the sink. Some are around 50, 51, a couple hundred micron or more. The next you will see will be a very, very similar technique for you all to look at. Now, this is really quite a big crystal. Here is the triangle along here. Focus that up and down a bit and look at the triangles,…. The diamond will grow, and fit in the diamond barrel… seen it both ways.

2nd Dictabelt

(H. Tracy Hall, con't.)

Here's the triangle along here and I think if you could focus you would see it's really an octahedron. There's triangular growth on top of triangular growth. Very difficult…the triangles grow and shift in the diamonds and they are all raised up as as ___ in the surface of the diamonds—seen it both way's.

Now the next one we that we are to do will be immersed in a liquid and we hope in this next one that the heat from the lamp shining of the crystals will cause convection in the liquid, stir up the crystals so that they will turn over and over, and we hope you'll be able to catch a glimpse of more than one side of the crystal at the top. All of these things in here are crystals immersed in a liquid. I don't see much….you might have to wait a minute for things to get turned on. The same sort of slightly yellow tinged crystal which you've seen before on a colored slide and then the other video we made. All of the stuff that you're viewing is diamond. This has all been cleaned up and your not—all the crystals your looking at are diamond. This one here may be an part an octahedron. Looks like we should warm that one up a little bit before we put it on, I don't think we're getting much convection there to turn the crystal over. Thank you very much ____.

Now you have noted that the man made diamond shown to you this day are very small. You have no doubt wondered if diamonds this size are useful. The answer is yes. Over one half of the industrials, and that's 4 million carrots, of diamonds the size shown here and smaller, are used each year as industrial diamond powders for the sawing, grinding, and polishing of hard materials. Thank You.

(Moderator)

And now for the skeptics left in the audience Herb Strong is going to tell you the evidence we base the conclusion that these things you have been looking at in the audience are really diamond. Herb.

(Herb Strong)

My leg went to sleep there I guess, and that's no insult to the previous speakers. When you think of all the suspicions attached to early claims of diamond synthesis, and that at least one unwise fellow is languishing in jail you can understand what a bad case of jitters the first few diamonds induced. Of course we knew all along in our bones that we had made diamonds, but it's kind of hard to express that in the literature. To prove the crystals you have seen are diamond. One shown by x-ray diffraction that their crystal structure was identical with that of diamonds. And the...