# Quiz

(year)

- The first synthetic gems were rubies produced in (year)
- Mass production of synthetic rubies began in \_\_\_\_\_
- The first synthetic diamonds were produced in \_\_\_\_(year) by the \_\_\_\_\_ company
- The first synthetic diamonds were produced by the technique
- The yearly World production of synthetic diamonds is carats
- Natural quartz gems (amethyst and ) are too inexpensive to support a synthetic gemstone market (True or False)
- The first commercially successful synthetic emeralds were made by Carroll C. Chatham in 1939, when he was 25 (True or False)
- Synthetic, gem-quality diamonds are still years from being commercially available.

# **Gemstone Synthesis**

Paul F. Hlava Access to Gems & Minerals







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- Day One Aag found a very pretty piece of rock and tied it around his neck with a piece of string. (Gemstones were born.)
- Day Two Envious Oog couldn't find a piece of the "real" stuff so he made something to look like it. <u>Simulated</u> gemstones were born. (Why not synthetic?)





#### As I was Saying.....

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... Moreover, I have in my library certain books by authors now living, whom I would under no circumstances name, wherein there are descriptions as to how to give the color of smaragdus [emerald] to crystallus [rock crystal] and how to imitate other transparent gems: for example, how to make a sardonychus [sardonyx] from a sarda [carnelian, in part sard]: in a word to transform one stone into another. To tell the truth, there is no fraud or deceit in the world which yields greater gain and profit than that of counterfeiting gems.



Pliny [23-79 AD], from Ball, 1950, p. 19

#### As I was Saying.....

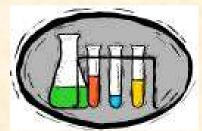
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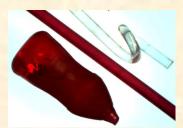
Nothing has changed!





Definitions

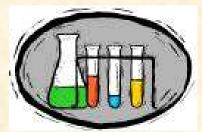








- Definitions
- The Keys to Success

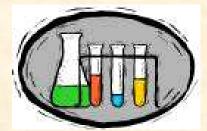


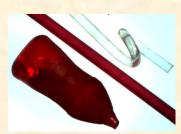






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  - Flame Fusion or Verneuil
  - Czochralski
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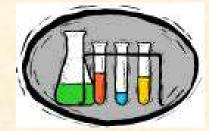








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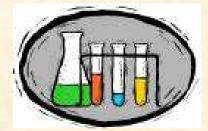








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  - High Pressure Synthesis
  - Recent Developments

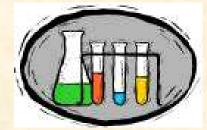


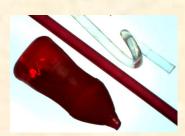






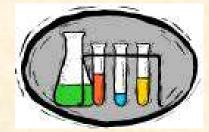
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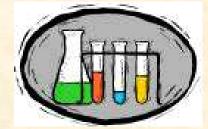
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- **Gemstone** a naturally occurring material that is desirable for its beauty, valuable in its rarity, and durable and stable enough to give lasting pleasure when worn as an ornament.







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  - Simulants any material with the wrong chemistry and physical properties masquerading as a gemstone. (plastic, glass, other minerals)









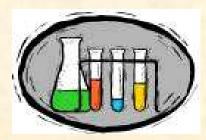
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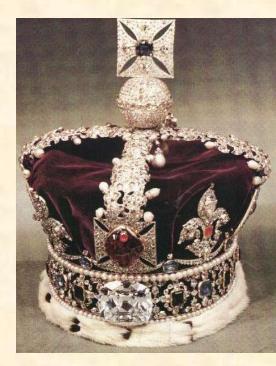
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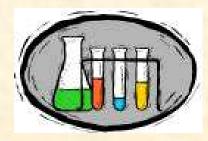






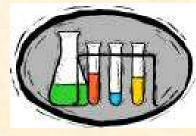
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- Advances in the science of chemistry by the end of the 18th century revealed the constituent elements and coloring impurities of many gems and their proper proportions.
   (diamond, 1797; emerald, 1798; ruby, by 1800)







#### **Other Keys**

- Heat required development of potent torches and furnaces
  - Rubies and sapphires made at 2200°C (4000°F)



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- Pressure not realized until late
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- Pure starting materials
   Ruby from alum

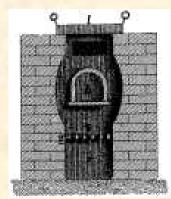


#### **Synthetic Rubies - Early Work**

• Nassau lists over 2 dozen people who worked on ruby synthesis in the early 1800's.





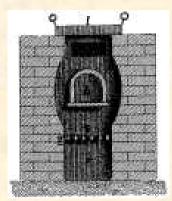


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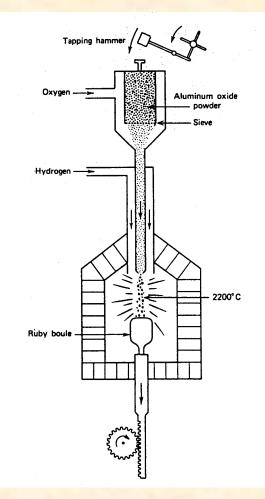
 Frémy, 1877 - large crucibles, Pb-oxide flux, small but commercial quality - too expensive to compete with naturals.





#### **Verneuil Succeeds**

 Verneuil (student of Frémy) perfected a viable furnace to make ruby, and later sapphire somewhere between 1888 and 1891. Commercial, mass production began in 1902. The technique is called **Flame Fusion or the Verneuil Process.** 



# Verneuil Furnace and Boules



The small attachment neck is crucial to reducing stresses.

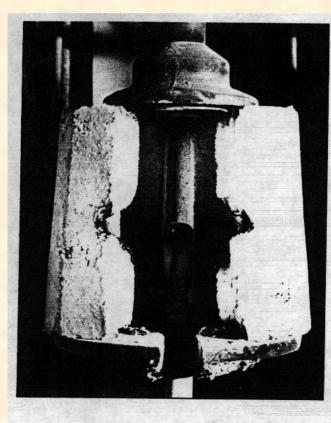
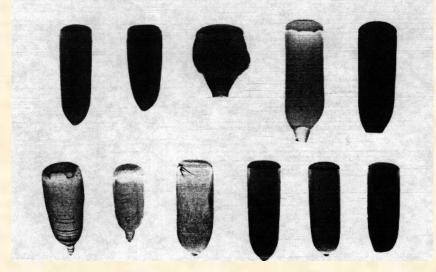
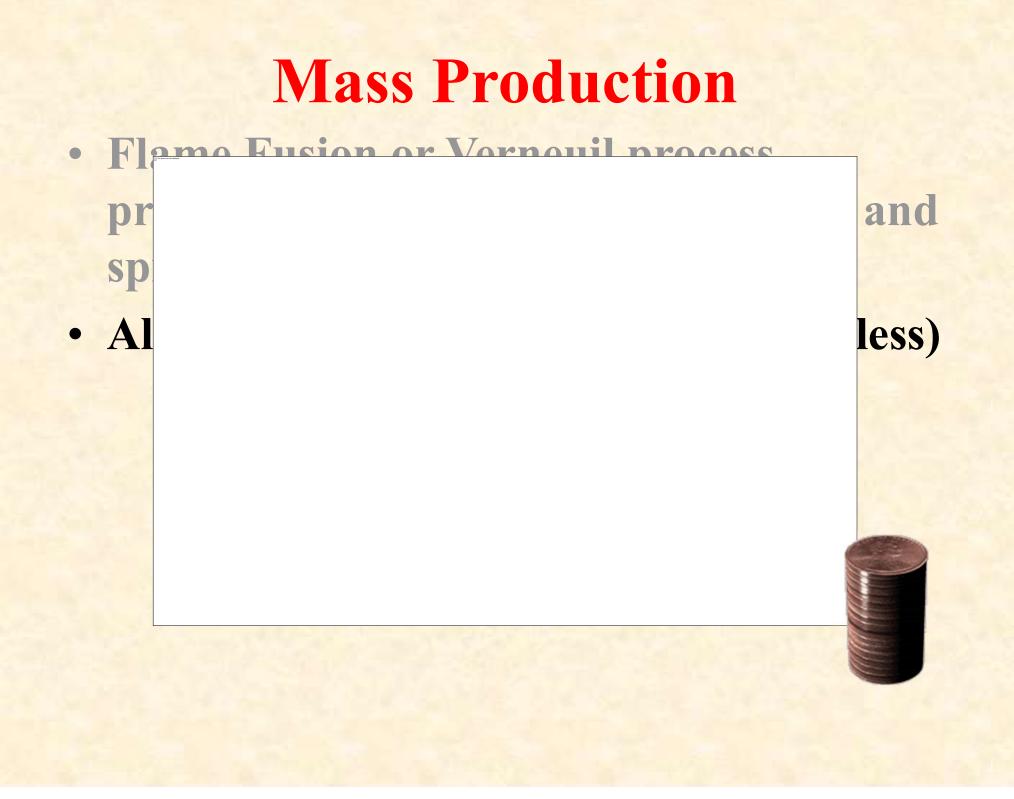


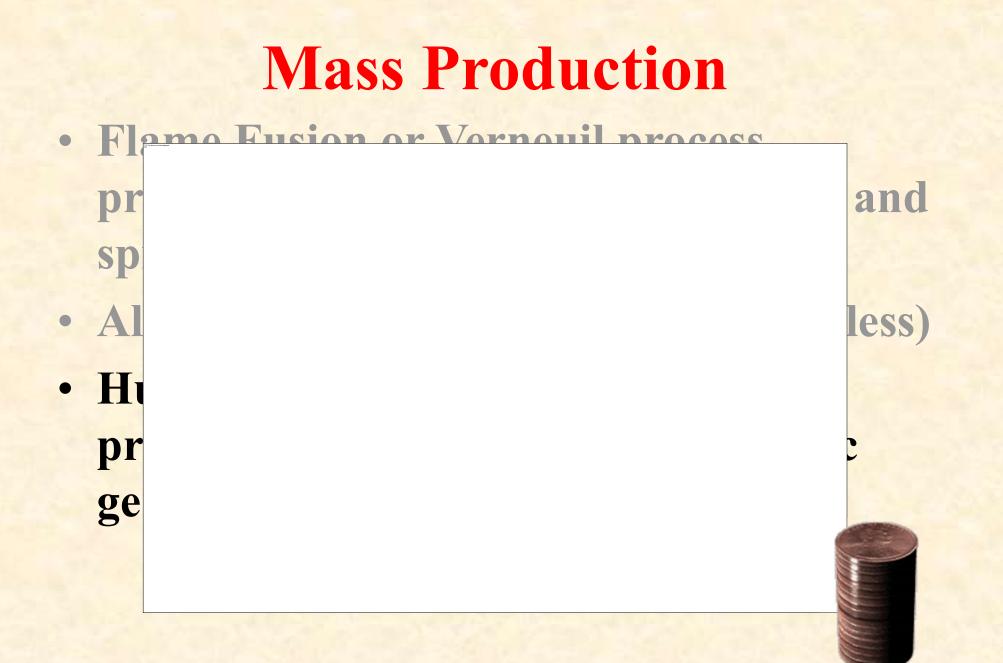
Figure 15.2 Inter furnace showing (D. Swarovski)

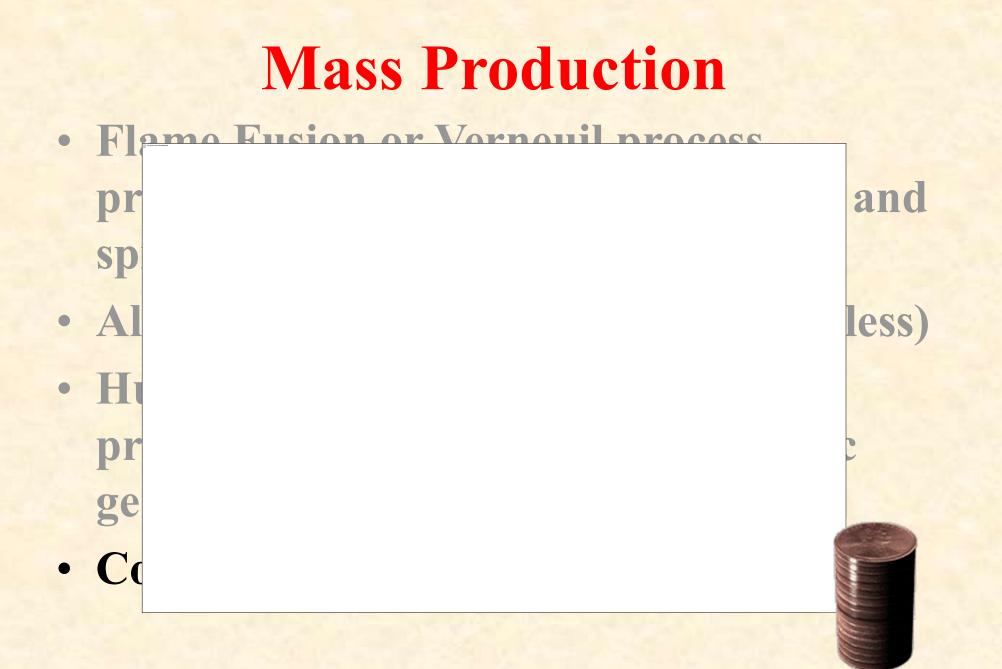


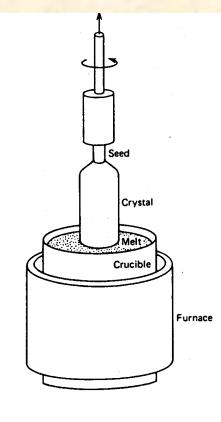
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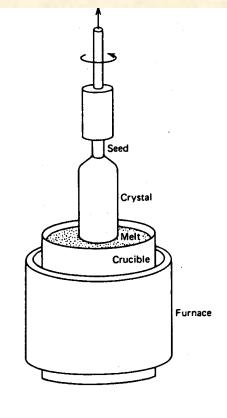






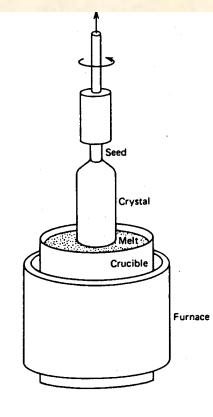






• A small seed on a rotating rod is dipped into a pool of molten ruby.

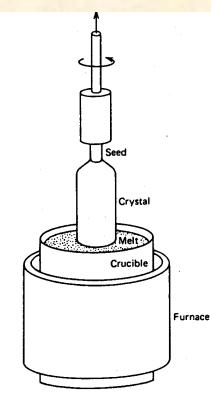




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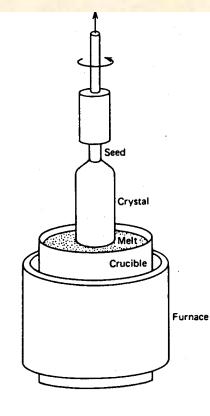


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• Product more expensive because technique is touchy and requires expensive Ir crucible.



## **The Emerald Problem**

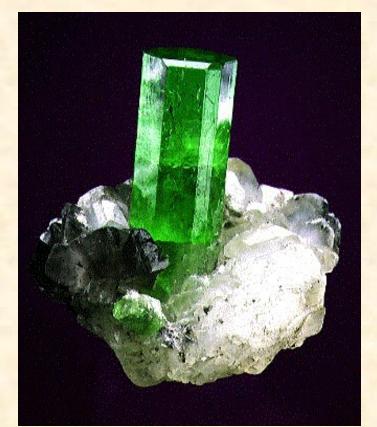
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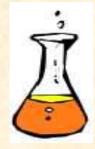
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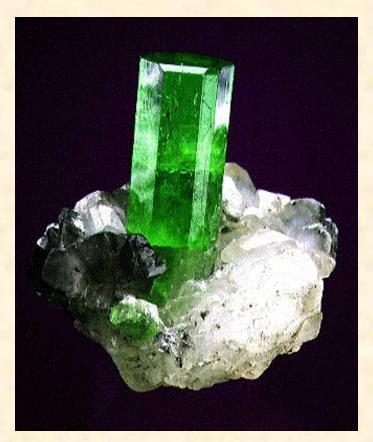




# **The Emerald Problem**

- Emeralds melt and crystallize incongruently (They decompose into other compounds before they melt or form these upon cooling from a melt.)
- The Verneuil technique does not work.
- They have to be crystallized from some solution or flux.





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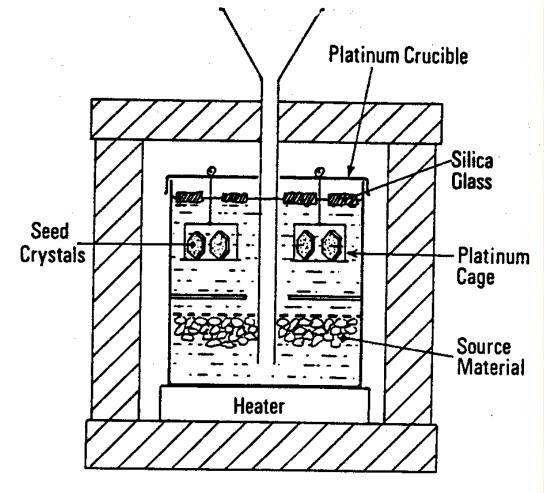
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- Gilson 1964 seeds, heterogeneous nucleation





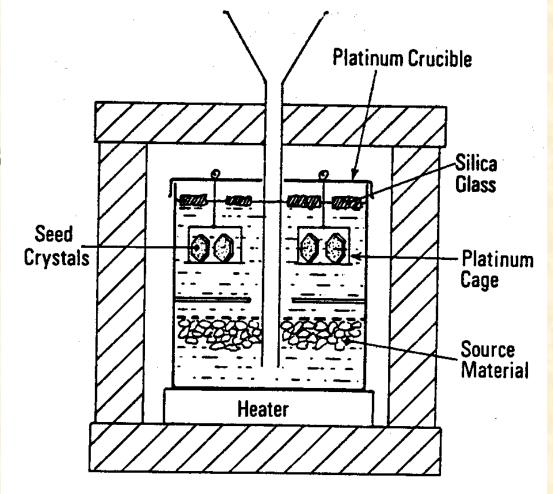
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 First successes with high temperature solvents called fluxes



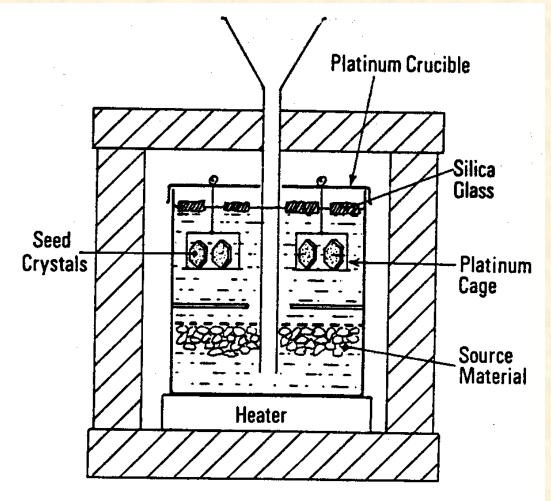
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- Pt crucible with flux and right chemicals (flux versus fluxreaction).
- May or may not use seed crystals.



Flux grown gem crystals require

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- All of these are expensive
- Product is pricey but excellent quality





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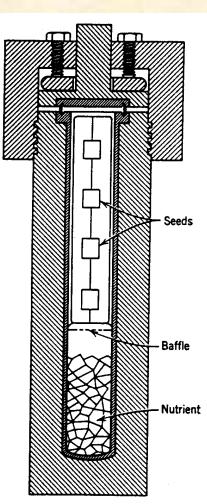
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- USA and Britain WW2 many successful, commercial processes by 1950.

## Hydrothermal Quartz

 Quartz synthesis requires an alkaline (NaOH), aqueous solution, modest temperatures (just a bit over 300°C)and pressures (1700 bars), a modest temperature gradient (~40°C) and pure feed for about 33 days.

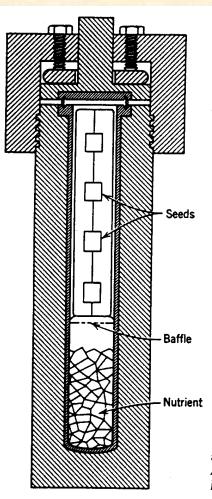






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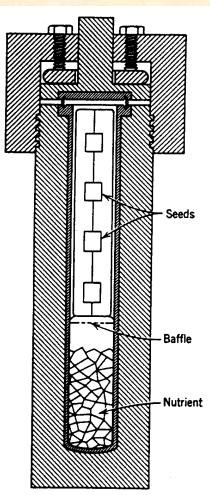


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- Part of this is smoky quartz, citrine, and amethyst







Emeralds grow from solutions just like quartz so we should be able to grow them hydrothermally, also.

Wyart and Scavinar - 1957?

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 Hydrothermalreaction process

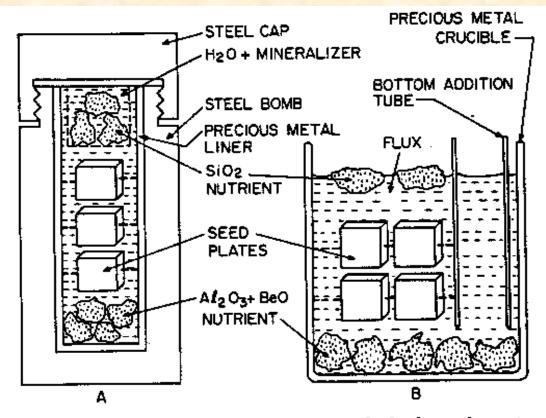




FIG. 13–2. Schematic diagrams comparing hydrothermal reaction growth (A) and flux reaction growth (B) for synthetic emerald.

- Hydrothermalreaction process
- Similar to fluxreaction process

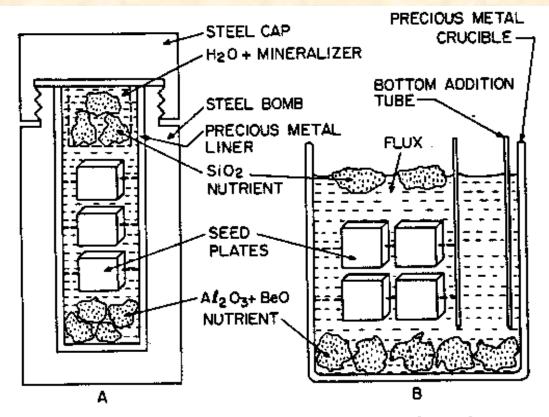




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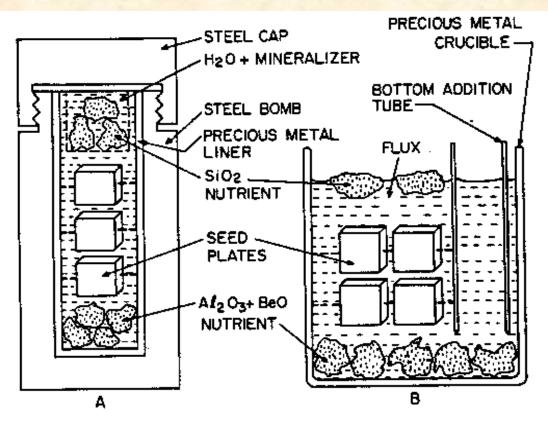


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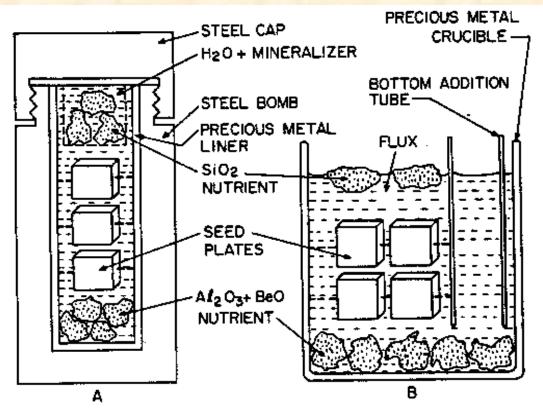


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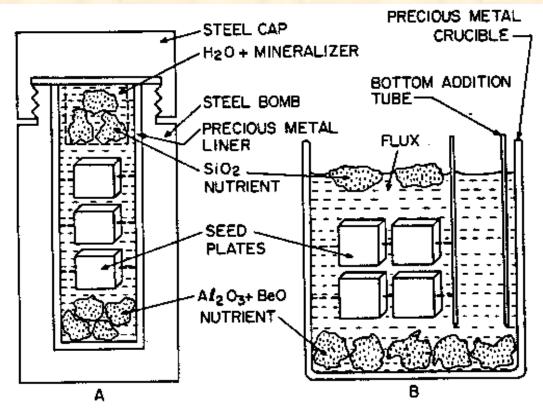
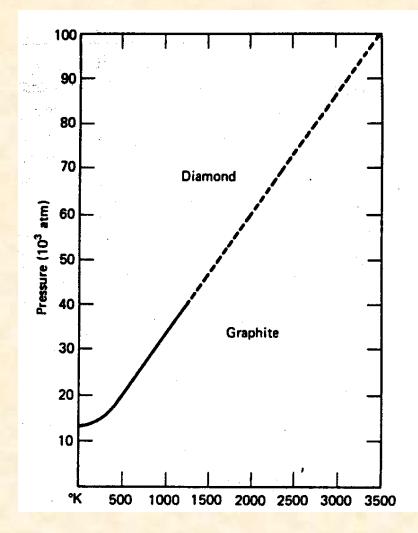


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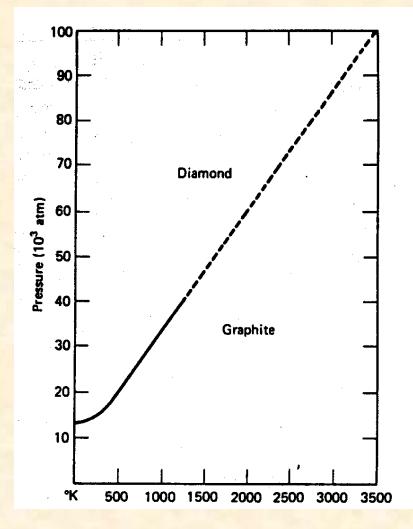
### **Diamond Synthesis Problem**

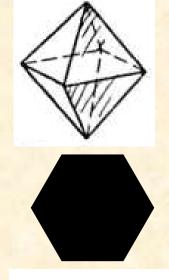
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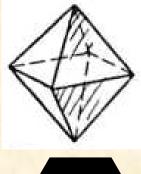
- Diamonds need more than heat to grow, they need tremendous pressures and a solvent.
- Pressures of 60,000 to 70,000 atm or about 1 million psi.



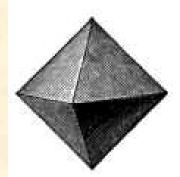




• Many Early workers claim to have made diamonds



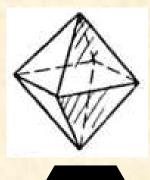


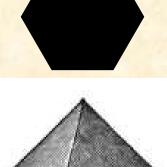


- Many Early workers claim to have made diamonds
- Most famous



– J. B. Hannay - 1880, iron'tubes (naturals)



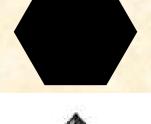


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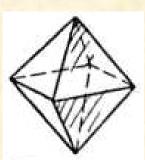




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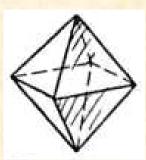


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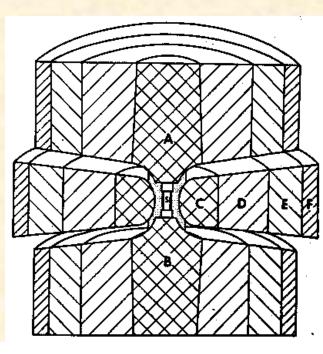
- F. F. H. Moissan iron rods (SiC)
- C. A. Parsons a variety of methods (spinel)
- All were found to have failed (pressures low)





### **High-Pressure Research**

The main problems stem from the need for materials that will continue to function at those extreme T-P conditions.

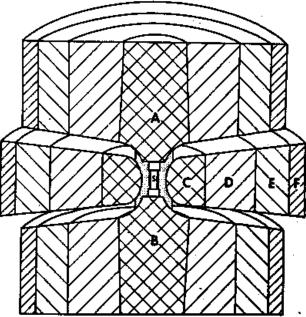


### **High-Pressure Research**

The main problems stem from the need for materials that will continue to function at those extreme T-P conditions.

- Bridgman Father of HPR, lots of papers

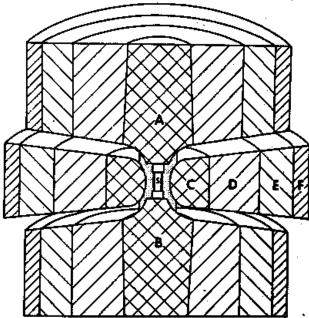
   Bridgman Anvil WC
  - Bridgman unsupported area seal



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The main problems stem from the need for materials that will continue to function at those extreme T-P conditions.

- Bridgman Father of HPR, lots of papers – Bridgman Anvil - WC
  - Bridgman unsupported area seal
- GE effort
  - Tapered pistons
  - Belt seal (Tracy Hall)



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- Tracy Hall was relegated to the leaky old 400 ton press and his new seal design.
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- Independent run by outsider on Dec. 31 confirmed technique



#### "First" Synthetic Diamonds

 First to actually synthesize diamonds - a team at ASEA (~General Electric Co. of Sweden) in 1953







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- Not published until after GE announcement
- Very similar process







 Most Synthetic diamonds used for abrasives



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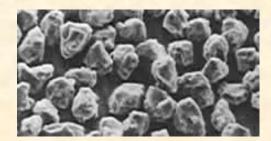


- Most Synthetic diamonds used for abrasives
- 2.5 billion carats/year
- Each machine gets 6 to 8 runs per hour.
- Gem diamonds would take a long time.
- Press breakage still a major fact of life.



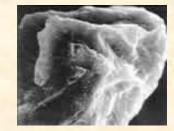
# DuPont Diamonds Synthesis by explosives (or implosives) - a shock-compaction technique

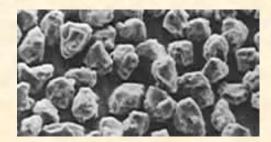






- Synthesis by explosives (or implosives) a shock-compaction technique
- Long, thick walled, steel pipes

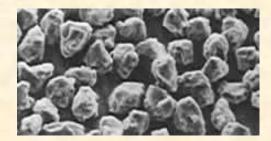






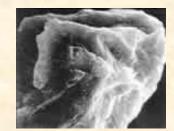
- Synthesis by explosives (or implosives) a shock-compaction technique
- Long, thick walled, steel pipes
- Filled with Cu and C powder

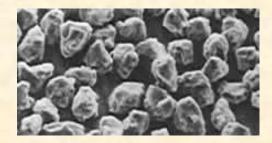






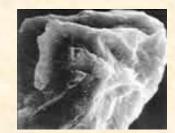
- Synthesis by explosives (or implosives) a shock-compaction technique
- Long, thick walled, steel pipes
- Filled with Cu and C powder
- Surrounded by a thick blanket of explosives

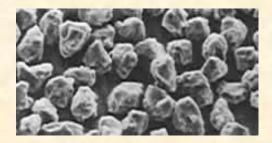






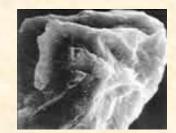
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- Initiated at one end

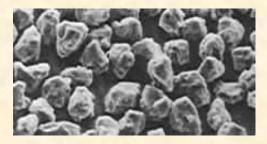






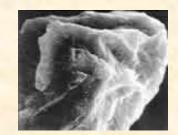
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- Long, thick walled, steel pipes
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- Surrounded by a thick blanket of explosives
- Initiated at one end
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- Pricier than HPHT but better selfsharpening





Small diamonds in a few minutes





- Small diamonds in a few minutes
- Big diamonds take a long time





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- Most are yellow nitrogen contamination



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- Most are yellow nitrogen contamination
- Colorless diamonds hard to make
- Can be told by inclusions, fluorescence
- Being made by several CIS, GE, Sumitomo and DeBeers!
- Used to Cost more than naturals but now....



# **Recent Developments - Gemesis** Recent article (Wired) about Gemesis in Sarasota, FL



**Recent Developments - Gemesis** Recent article (Wired) about Gemesis in Sarasota, FL –HPHT Russian designed apparatus



Recent Developments - Gemesis Recent article (Wired) about Gemesis in Sarasota, FL –HPHT Russian designed apparatus –3 days - 3 carat yellow crystals



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Main source is the Accendo Collection

**Apollo in Boston, MA** 



#### Apollo in Boston, MA - CVD



Apollo in Boston, MA – CVD – 0.5 mm per day times ?? Area



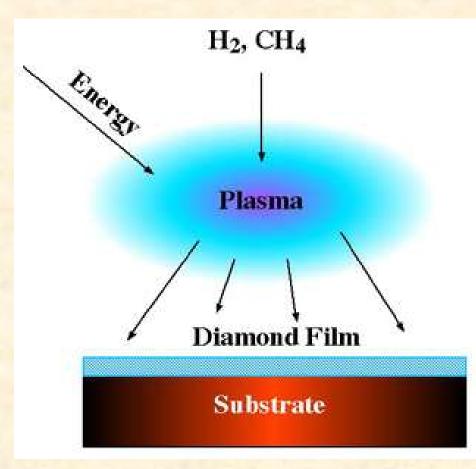
Apollo in Boston, MA – CVD – 0.5 mm per day times ?? Area – Going for white



Apollo in Boston, MA – CVD – 0.5 mm per day times ?? Area – Going for white – Wall Street



# **CVD** Process









- Many substitutes tried
  - TiO<sub>2</sub>
  - YAG
  - GGG
  - Spinel
  - Sapphire
  - SrTiO<sub>3</sub>



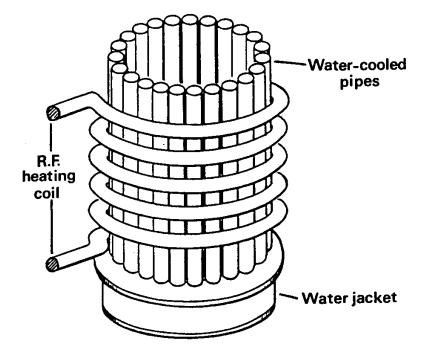
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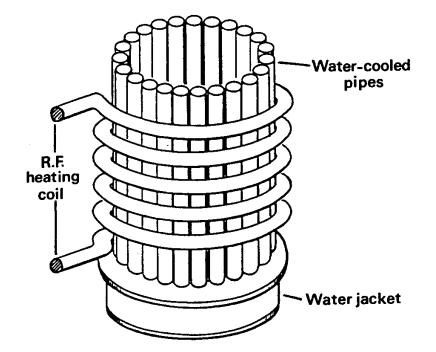
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  - SrTiO<sub>3</sub>
- CZ has best combination of properties
- Requires extremely high temps crucible?



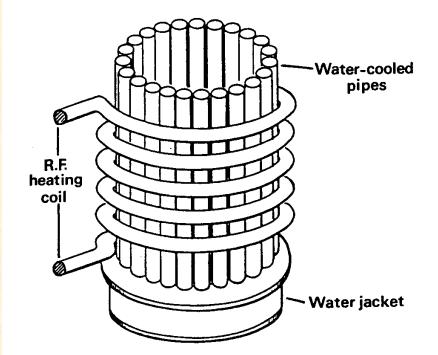
 Uses the starting powder as crucible - ZrO<sub>2</sub> + CaO or Y<sub>2</sub>O<sub>3</sub>



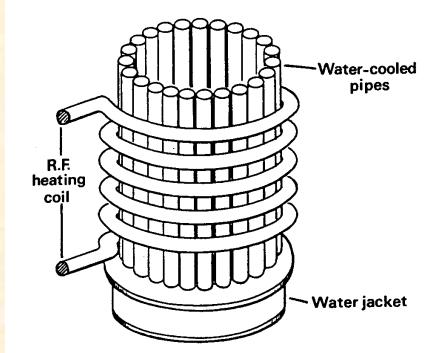
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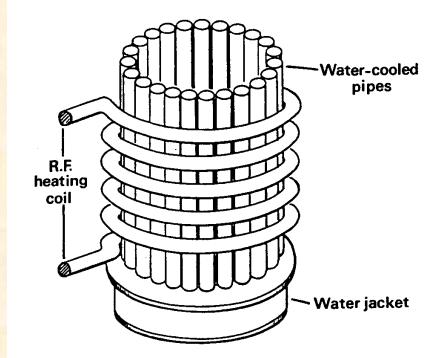
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- Melt keeps it going
- Water cooled Cu tubes keep the outer portions from melting
- Cool ZrO<sub>2</sub> powder acts as crucible



# **CZ** Notes

#### • CZ amazingly cheap considering

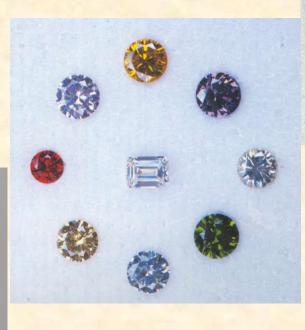






# **CZ** Notes

# CZ amazingly cheap considering Exotic material







CZ amazingly cheap considering

 Exotic material
 Exotic technique





- CZ amazingly cheap considering

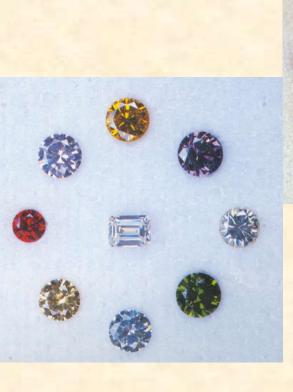
   Exotic material
   Exotic technique
- Own market now





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- (Invented Gem)

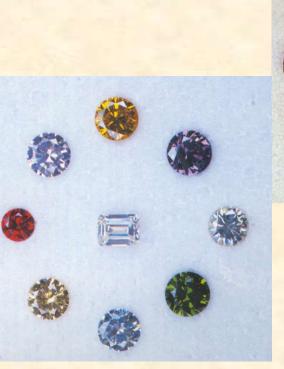






- CZ amazingly cheap considering
  - Exotic material
  - Exotic technique
- Own market now
- (Invented Gem)
- Lots of colors







➢ Mineral name for natural SiC



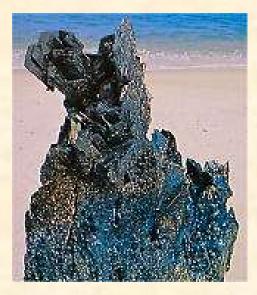


Mineral name for natural SiCUsually opaque, green to black





Mineral name for natural SiC
Usually opaque, green to black
3C Company - clear, gemmy





Mineral name for natural SiC
Usually opaque, green to black
3C Company - clear, gemmy
Almost pure white, also green





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Higher Luster





Mineral name for natural SiC
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Old thermal testers fail







>Mineral name for natural SiC >Usually opaque, green to black >3C Company - clear, gemmy >Almost pure white, also green >Higher Luster >Higher dispersion >Old thermal testers fail  $\succ$ (Doubly refracting)







• Process proprietary



- Process proprietary
- Indication of a thermal diffusion technique



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- Indication of a thermal diffusion technique
- SiC diffused through SiC tubes



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- Indication of a thermal diffusion technique
- SiC diffused through SiC tubes
- Very high temperature



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- Indication of a thermal diffusion technique
- SiC diffused through SiC tubes
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- "Hot new material"



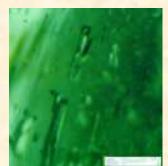
- Process proprietary
- Indication of a thermal diffusion technique
- SiC diffused through SiC tubes
- Very high temperature
- "Hot new material"
- (Synthetic Diamonds?)



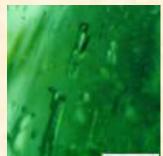
Telling synthetic gems from naturals is;
 Variable in difficulty



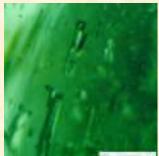
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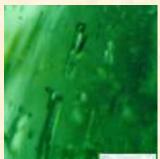
- Telling synthetic gems from naturals is;
  - Variable in difficulty
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- Synthetic gems sales do not usually "hurt" naturals



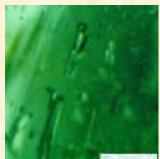
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  - Variable in difficulty
  - Usually based on inclusions
- Synthetic gems sales do not usually "hurt" naturals
  - They increase demand for naturals
  - Create own niches
- Gems are usually not good investments



## **That's All Folks!**

#### You may now applaud!



I'll be happy to answer questions now.

