

# SYNTHETIC DIAMOND AND ITS IDENTIFICATION

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GemFest Basel  
22<sup>nd</sup> March 2015

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# RESEARCH RESOURCES

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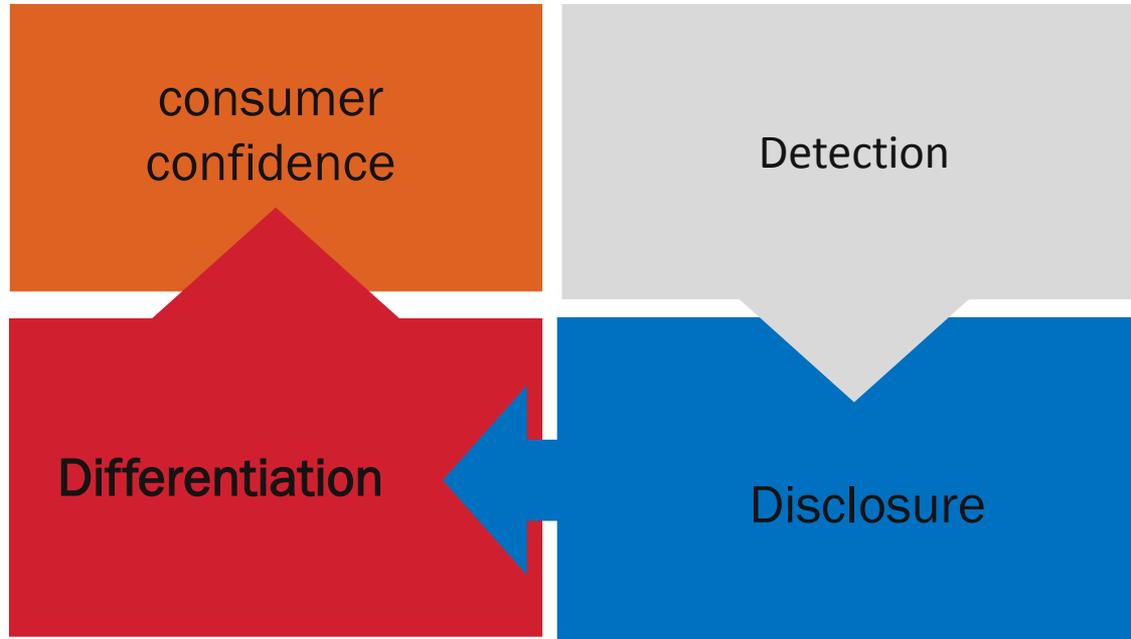
- De Beers Group
  - Over 65 years of fundamental research on diamond
- De Beers Technologies UK
  - 30 years of Consumer Confidence Technical Research (CCTR)
  - Contract synthesis and treatment research at specialist laboratories
- De Beers Technologies supports
  - Diamond research at UK universities
  - Centre for Doctoral Training in Diamond Science & Technology
  - Annual Diamond Conference
- 65<sup>th</sup> De Beers Diamond Conference was held at Warwick University 7-10 July 2014



# DE BEERS CONSUMER CONFIDENCE PROGRAMME

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maintaining & enhancing consumer confidence in the mystique & integrity of diamonds



**“A diamond is a natural mineral consisting essentially of pure carbon crystallised with a cubic structure.”**

– Source: FTC & CIBJO

## What is a synthetic diamond?

- A diamond that has been either partially or wholly crystallised or re-crystallised artificially in a laboratory

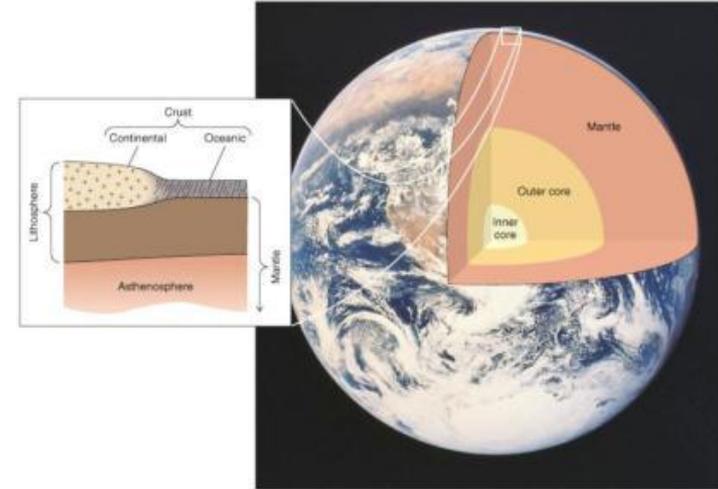
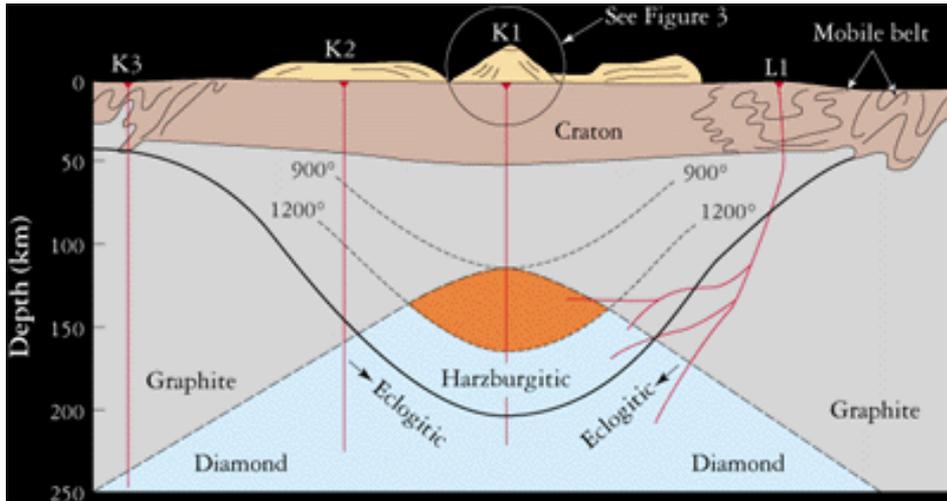
## What are its properties

- Synthetic diamonds have the same chemical and physical properties as natural diamonds, but have different growth structures and impurities which enable rapid identification using specialist equipment
- ‘Synthetic’ is from the Greek ‘Sunthetikos’ meaning ‘expert in putting together’ and is applied to ‘a substance or material made artificially by chemical reaction’
- This entirely appropriate term was applied by the scientists who first synthesised diamond in the 1950’s
  - General Electric in the US and ASEA in Sweden
- Using the term ‘grown diamonds’ is ambiguous and potentially deceptive since it does not clearly differentiate natural diamonds from synthetic
  - Natural diamonds are ‘grown’ by mother nature

# DIAMOND FORMATION

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- Formed in the earth's mantle at depths of 150 - 200 km and temperatures of 900 – 1200°C
- Formed 0.9 – 3.5 billion years ago (from isotopic analysis of inclusions)
- Stored under cratons at least 110 km below the earth's surface
- Until brought up in Kimberlite / lamproite eruptions

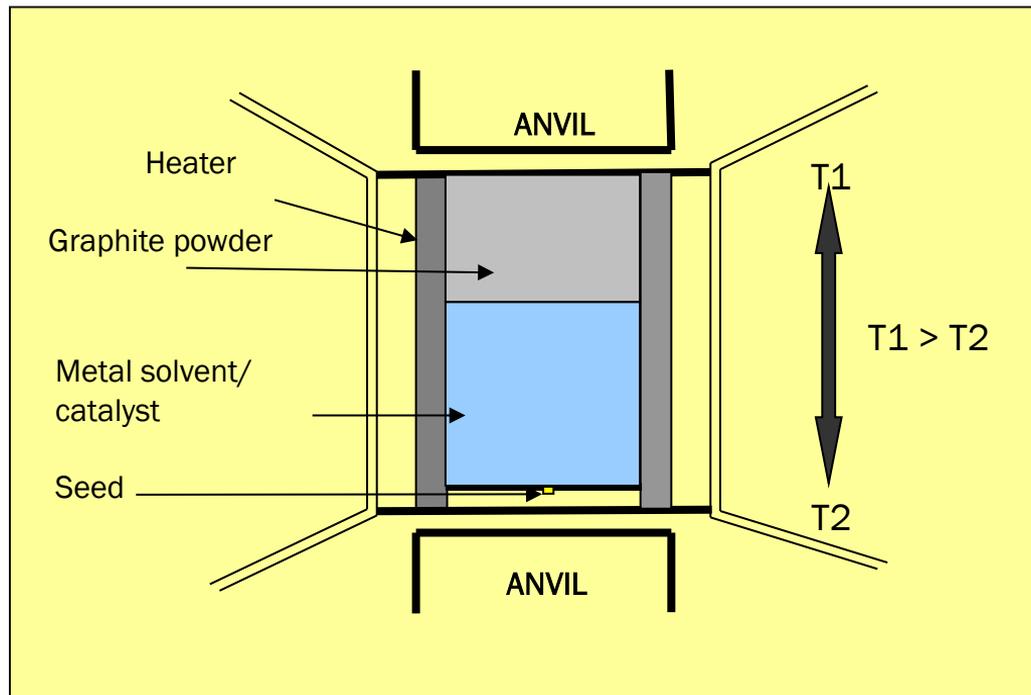


# SCHEMATIC DIAGRAM: HPHT SYNTHESIS

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Temperature-  
gradient method

Metal  
solvent/catalyst  
Nickel/Iron  
Cobalt/Iron



- Temperature = 1300 - 1500°C
- Pressure = 50 - 60 kbar

# DIFFERENT GROWTH CONDITIONS LEAD TO DIFFERENCES IN SHAPE

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## Natural diamonds

- Octahedral/dodecahedral



## HPHT Synthetics

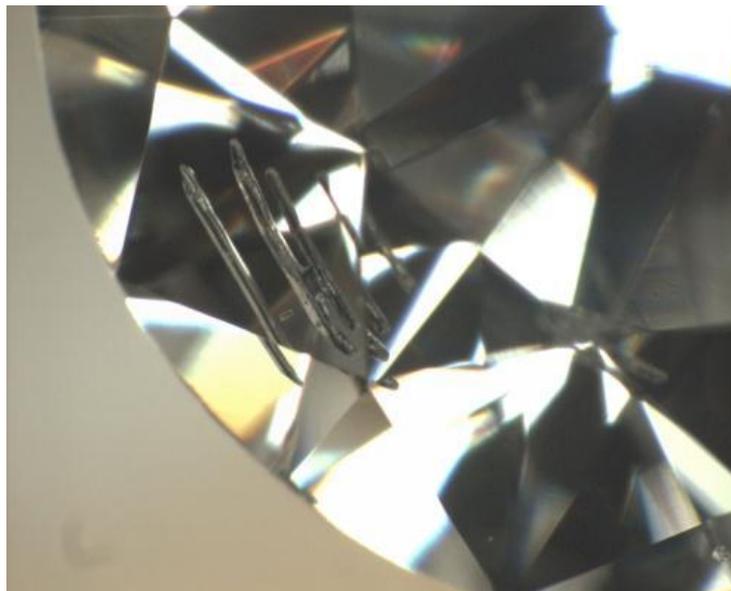
- Mainly cubo-octahedral
- Minor facets present



## IDENTIFICATION FEATURES - INCLUSIONS

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- Metallic inclusions
  - From solvent/catalyst
    - Rod-shaped
    - May be attracted to a magnet

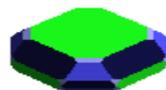


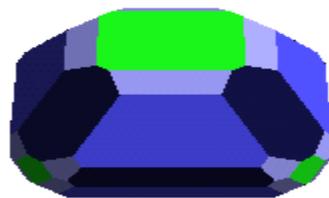
# HPHT SYNTHESIS V. NATURAL FORMATION

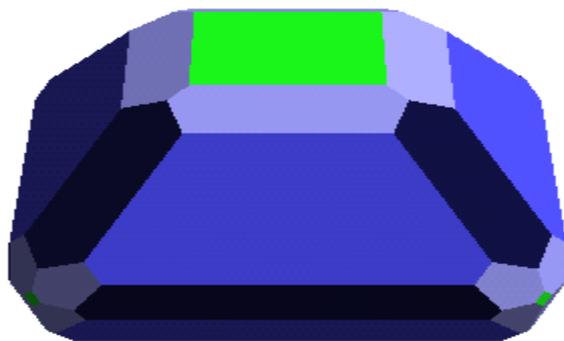
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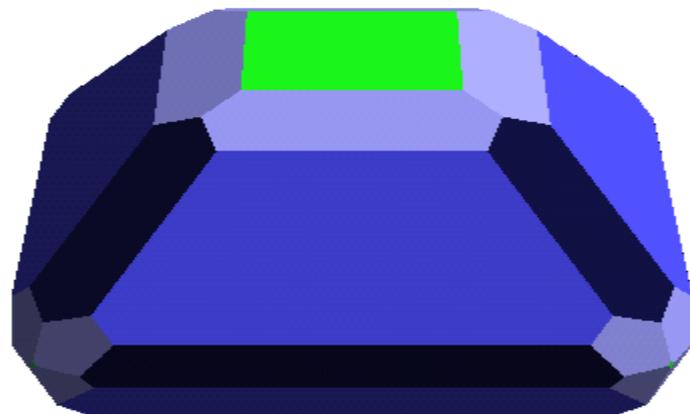
- HPHT synthesis relative to natural growth
  - Uses a different chemical environment
    - Metallic inclusions: visual inspection
  - Takes place at a higher temperature
    - Different crystal shape: DiamondView
  - Uses an engineered temperature gradient
    - Different crystal shape and development: DiamondView
- Synthetics have not been at high T for hundreds of millions of years
  - Less/different aggregation of nitrogen impurity
  - The DiamondSure screening instrument makes use of this difference

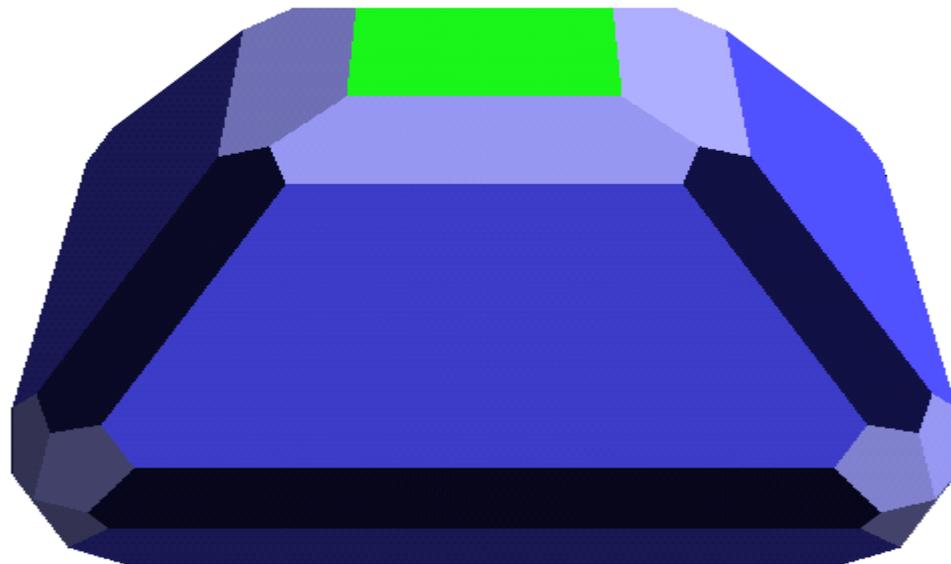


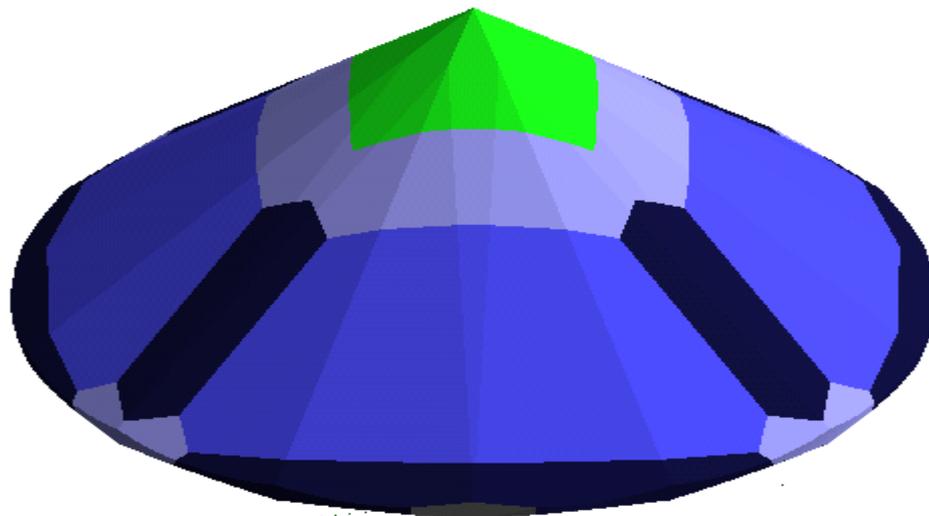


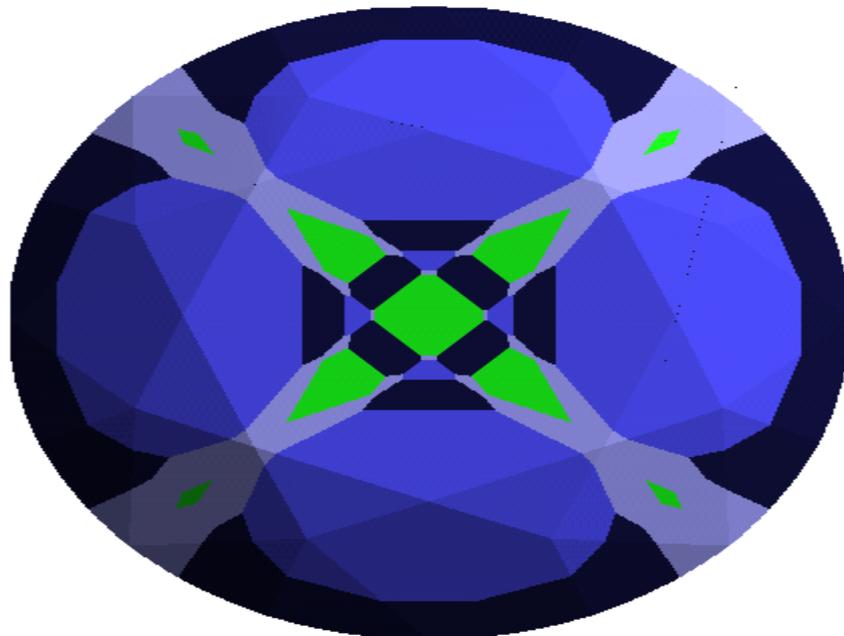






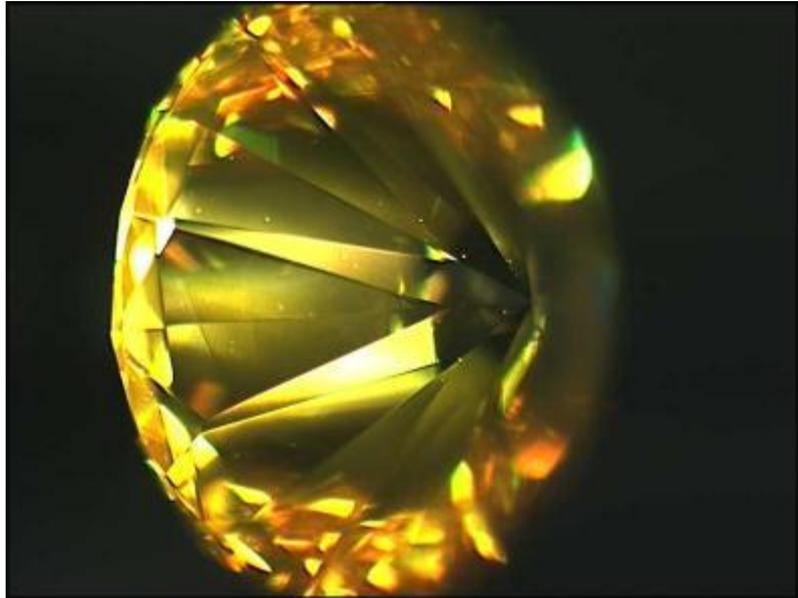
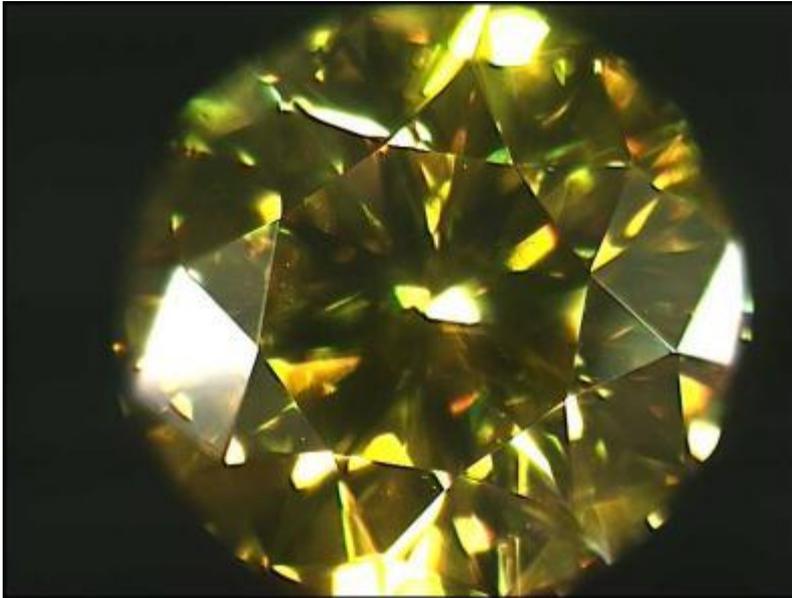






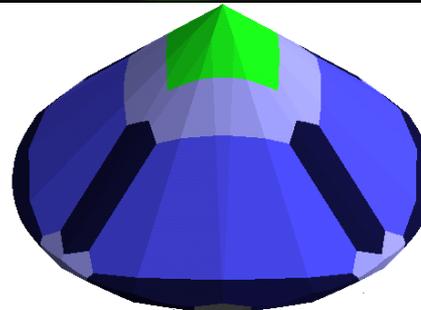
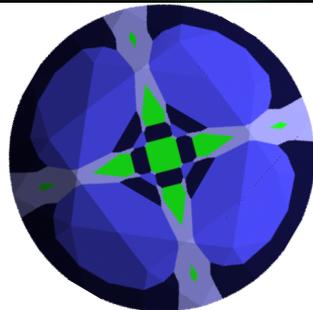
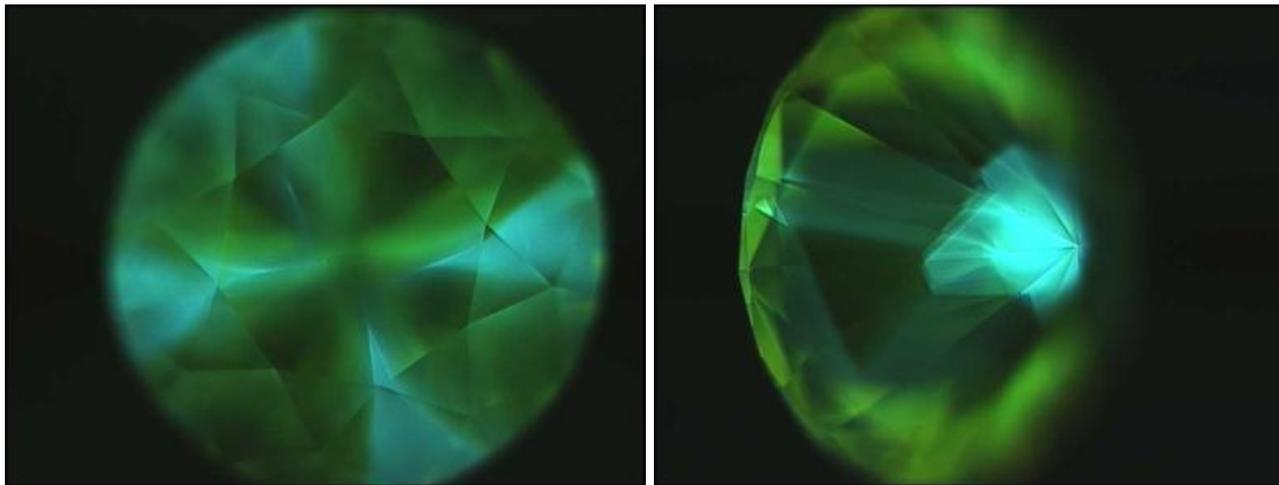
## POLISHED HPHT-GROWN SYNTHETIC DIAMONDVIEW WITH VISIBLE ILLUMINATION

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# POLISHED HPHT-GROWN SYNTHETIC UV ILLUMINATION: FLUORESCENCE

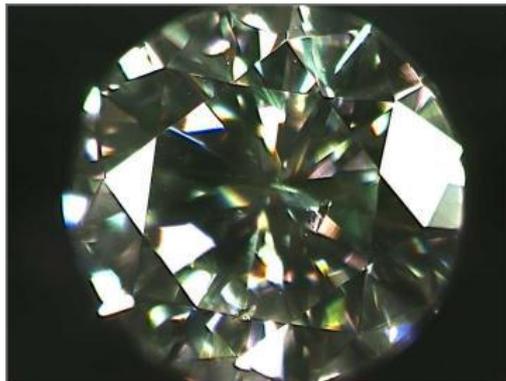
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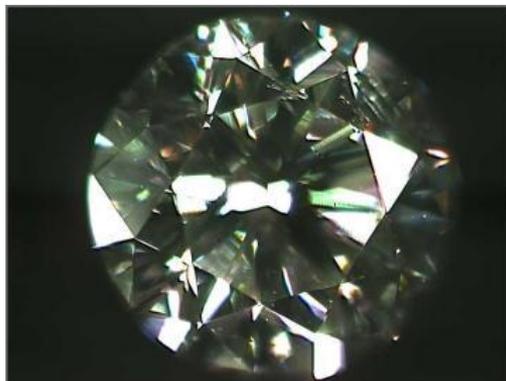
# DIAMONDVIEW IMAGES

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Colourless  
Diamond



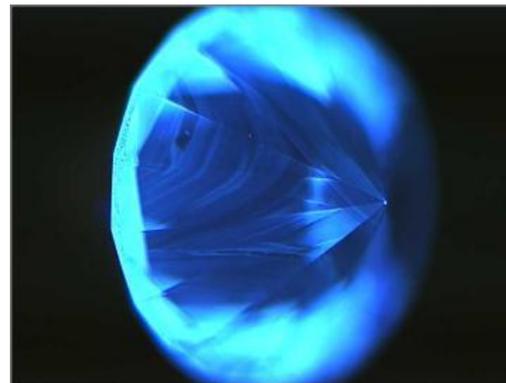
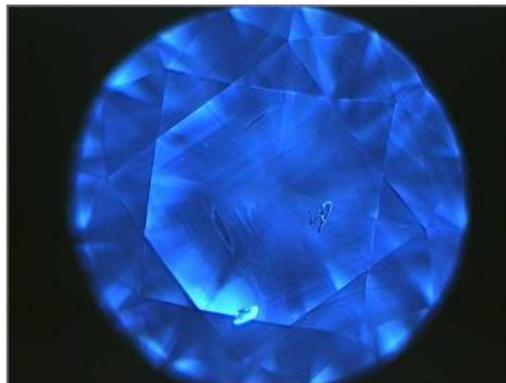
Colourless  
HPHT  
Synthetic



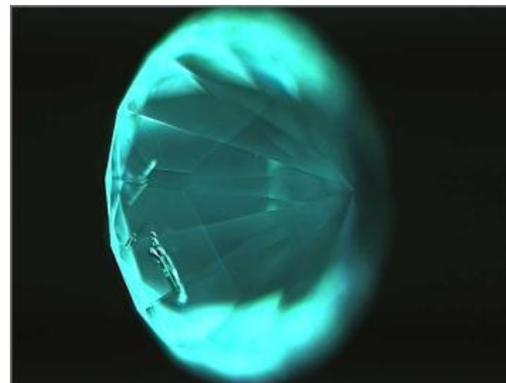
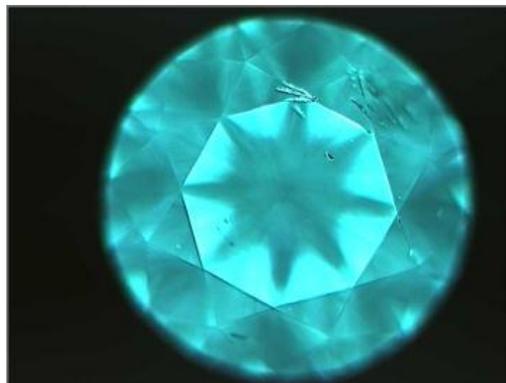
## DIAMONDVIEW IMAGES - FLUORESCENCE

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Colourless  
Diamond

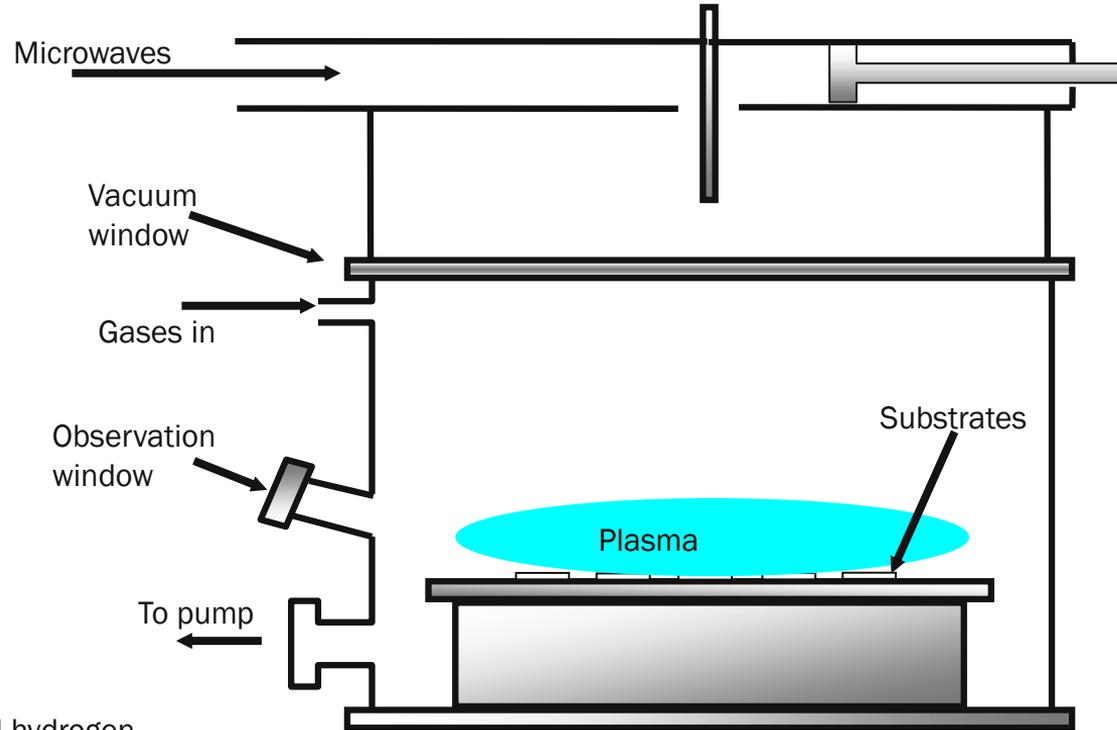


Colourless  
HPHT  
Synthetic



# DIAGRAM OF MICROWAVE CVD REACTOR

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Pressure ~ 0.1- 0.2 bar

Gases: typically methane and hydrogen

# CVD SYNTHETICS IN A CVD REACTOR DURING GROWTH

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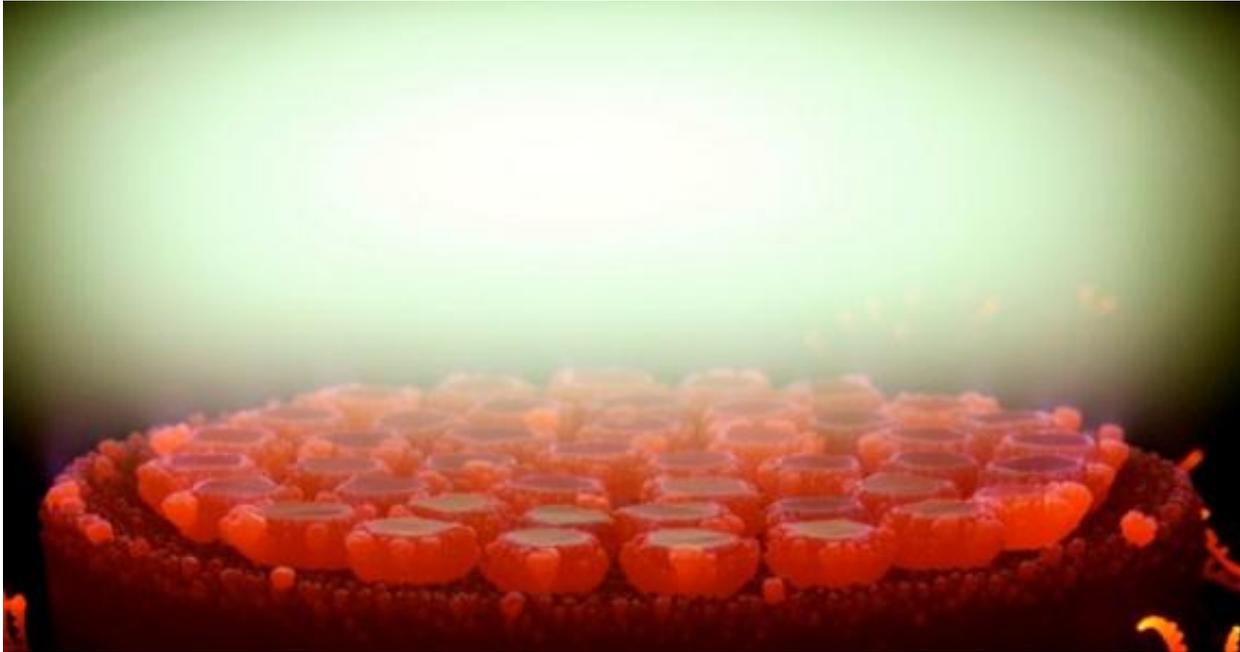


Image from Scio Diamond – Built in America:  
<http://www.youtube.com/watch?v=iQXVya-HAOk>

## EXAMPLES OF PART-PROCESSED CVD SYNTHETICS

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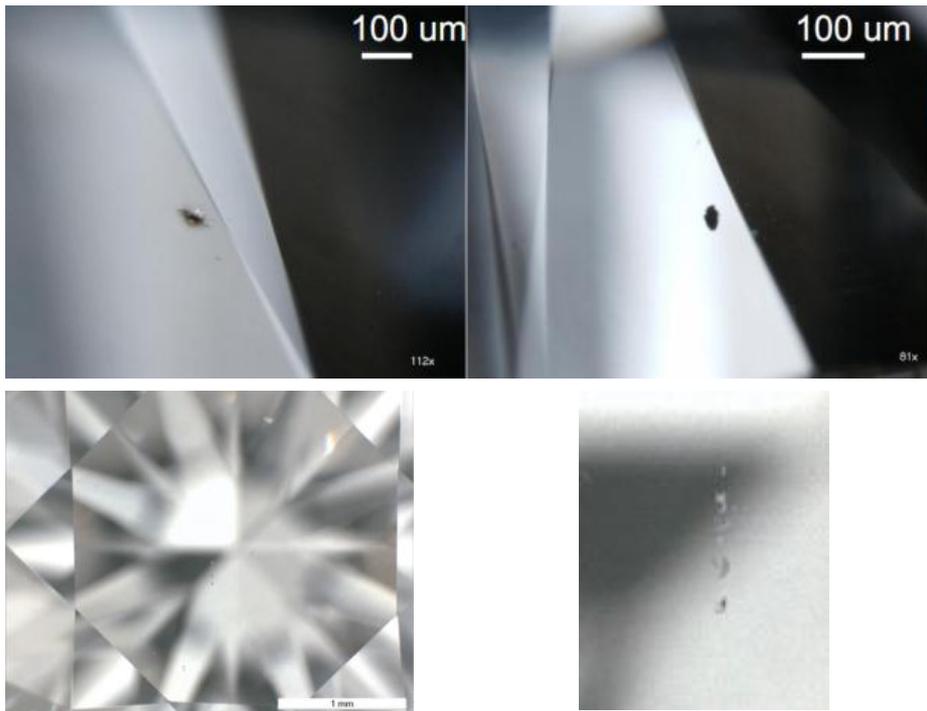
Approx. 5 mm x 5 mm x 3 mm  
(after removal of low quality  
edge material by laser sawing)



Ila Technologies CVD synthetics  
after laser sawing

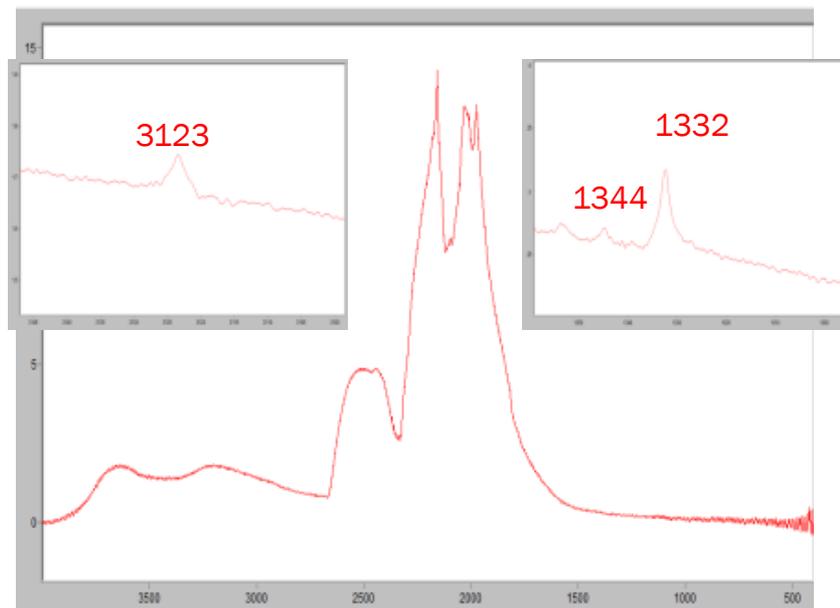
## KEY FEATURE: INCLUSIONS

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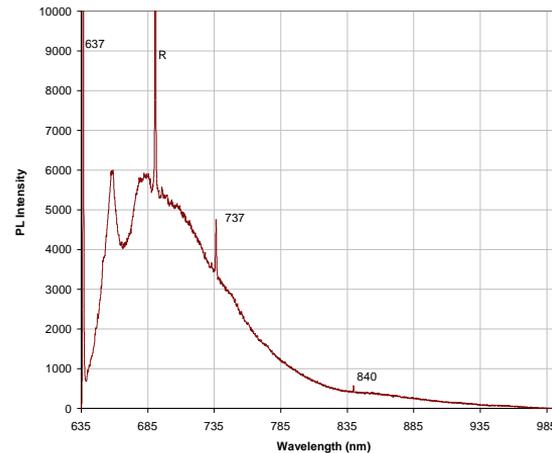
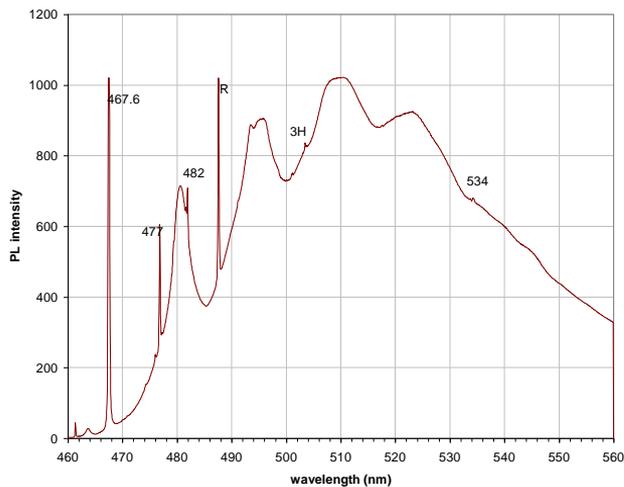


Examples of dark and light inclusions observed in heat treated CVD synthetics

Inclusions in CVD synthetics can be strikingly similar to natural inclusions.  
**Microscopy is insufficient for identification of CVD synthetics.**



- Near-colourless CVD synthetics are nominally type II (DiamondSure & IR)
- **1332 and 1344 cm<sup>-1</sup>** (N<sub>G</sub>)
- **3123 cm<sup>-1</sup>** Not present after annealing

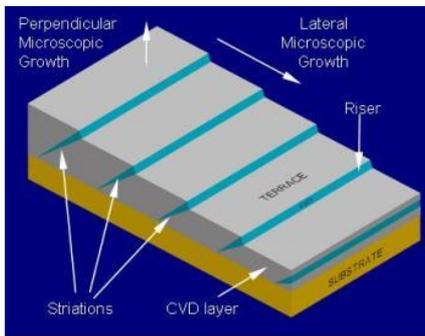


- **468 and 533 nm lines**
  - Commonly shown by as-grown CVD synthetics
- **737 nm line** is caused by impurity silicon
  - difficult to eliminate / remove

## KEY FEATURES: FLUORESCENCE / STRIATIONS

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Micrograph of the surface of an unprocessed CVD synthetic



DiamondView of a polished cross section of an as-grown CVD synthetic



In as-grown CVD synthetics, the *orange fluorescence* is caused by nitrogen-vacancy defects

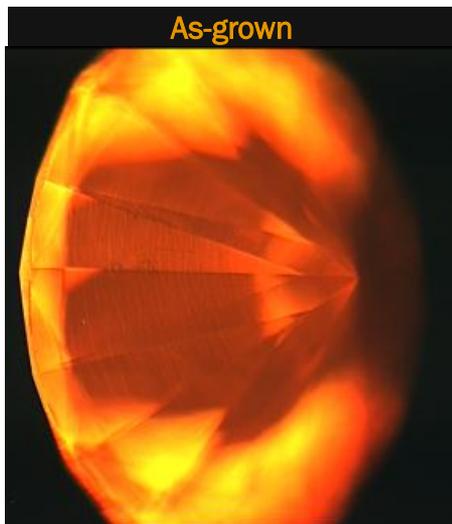
Striations result from the differential uptake of defects on different surfaces (risers versus terraces) during growth



- This CVD synthetic gemstone was high temperature treated in an HPHT press (temperatures  $>1600^{\circ}\text{C}$ ).
- There is a very noticeable colour improvement from P-T colour to I colour.

# DIAMONDDVIEW FLUORESCENCE IMAGES OF CVD SYNTHETICS

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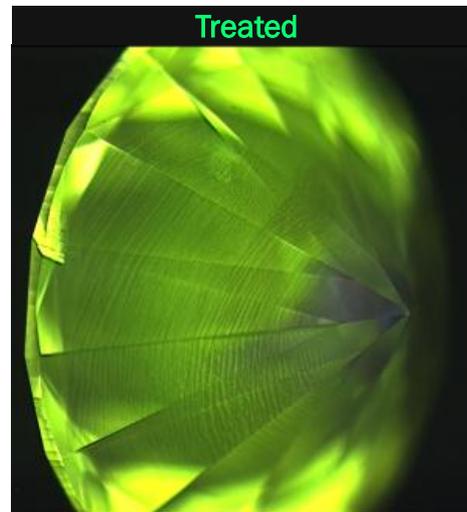


As-grown

Orange fluorescence

Striations normally present

Sometimes zoning observed (caused by growth events)



Treated

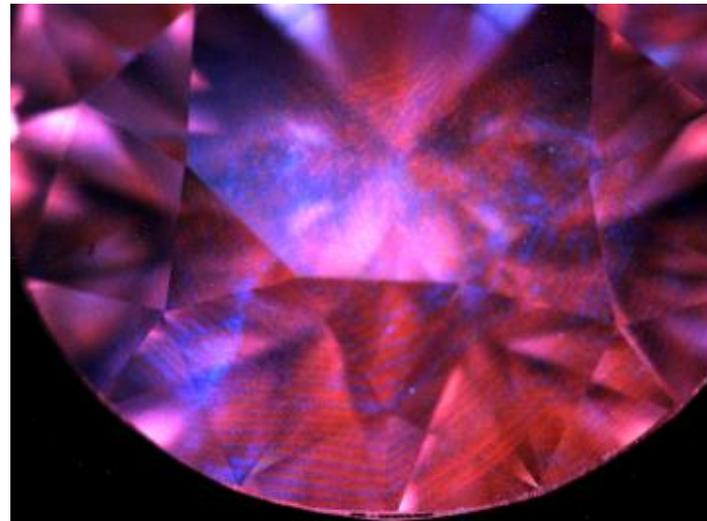
Orange luminescence replaced by green or greenish blue fluorescence

Phosphorescence generated

Striations / zoning remain

## APOLLO / SCIO CVD SYNTHETICS EXAMPLES SEEN 2007-2010

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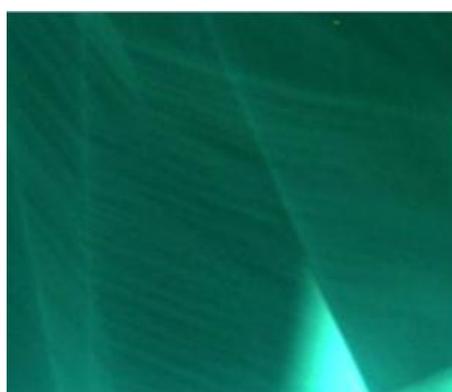
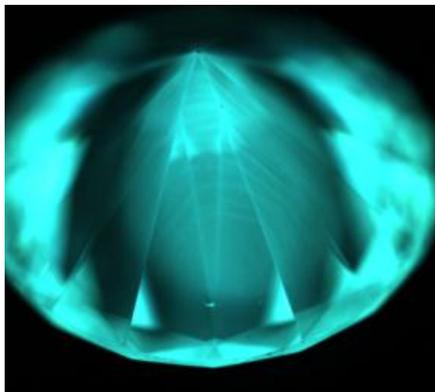


Colourless material

- Moderate size
- Orange fluorescence & striations
- Relatively high purity
- Not heat treated

# GEMESIS CVD SYNTHETICS

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Birefringence indicates relatively low strain

Bluish green fluorescence and blue phosphorescence

Fine-to-medium striations, and sometimes zoning in DiamondView images

## DIAMONDDVIEW (DV3F)

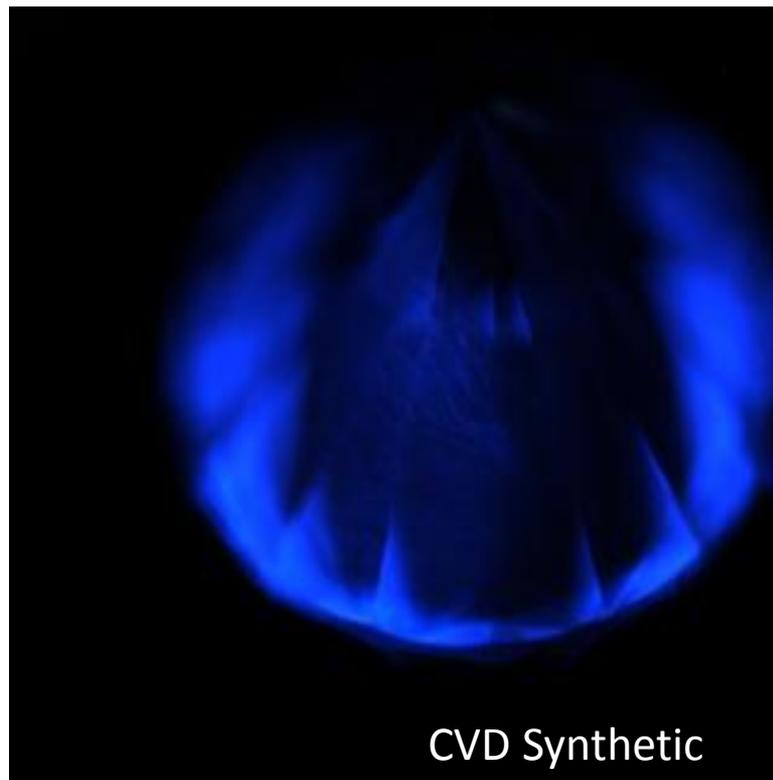
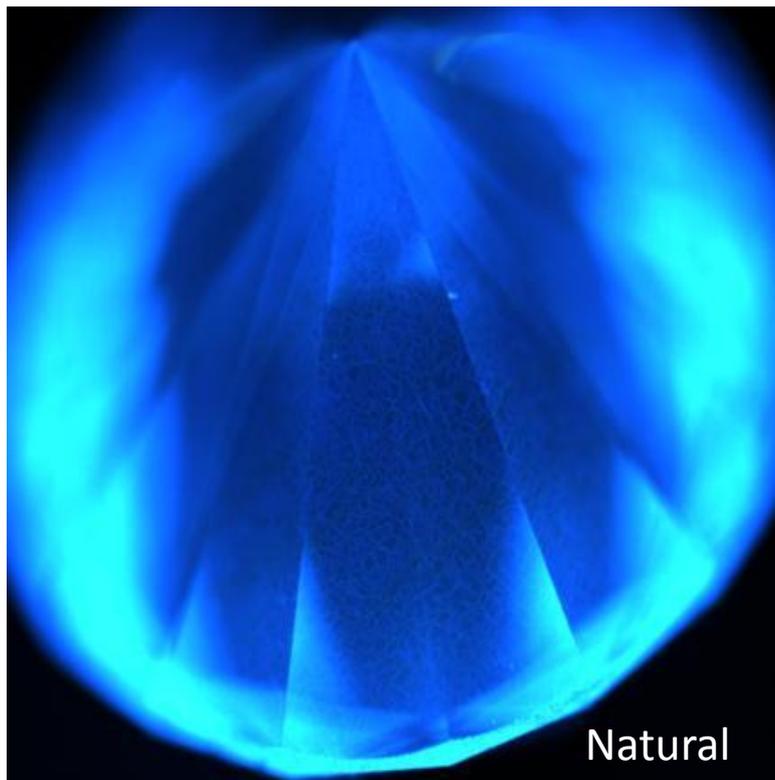
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- New improved DiamondView
- Interchangeable filters
- Improves detection of CVD synthetics
- Improved dislocations imaging in natural diamonds
- Plan to make available from beginning of H2, 2015

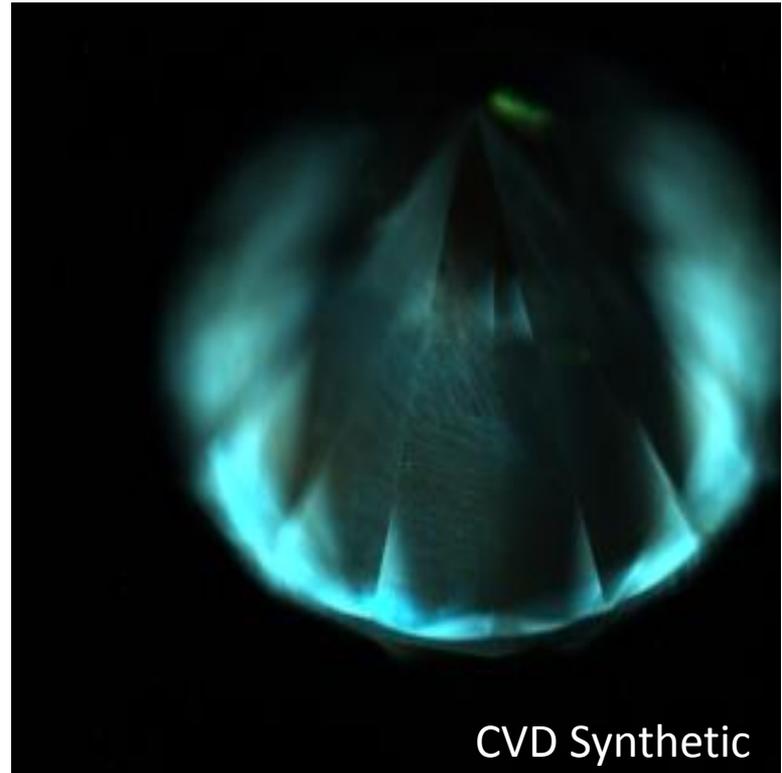
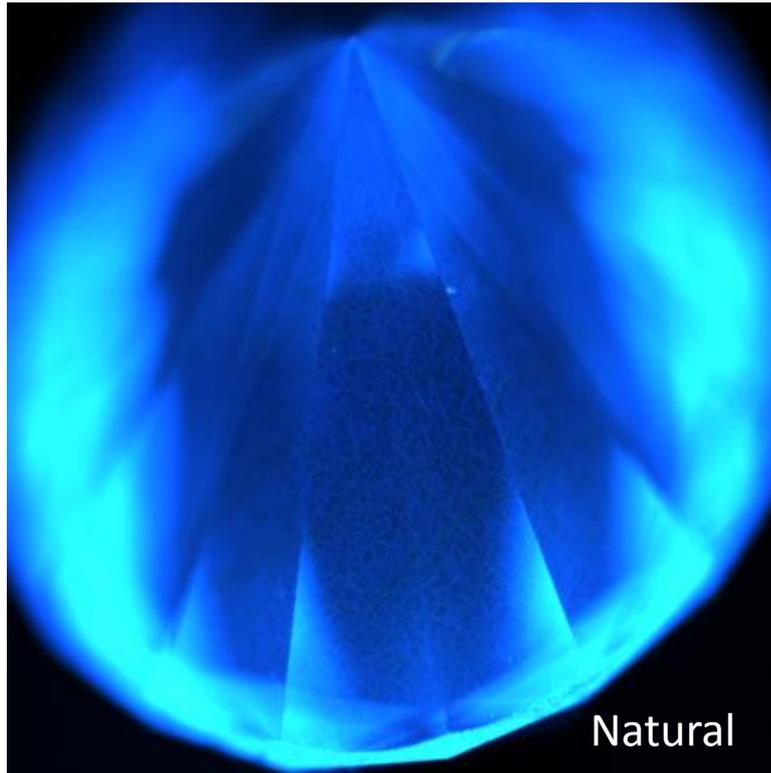
## DIAMONDVIEW – IMPROVED DETECTION WITH FILTERS

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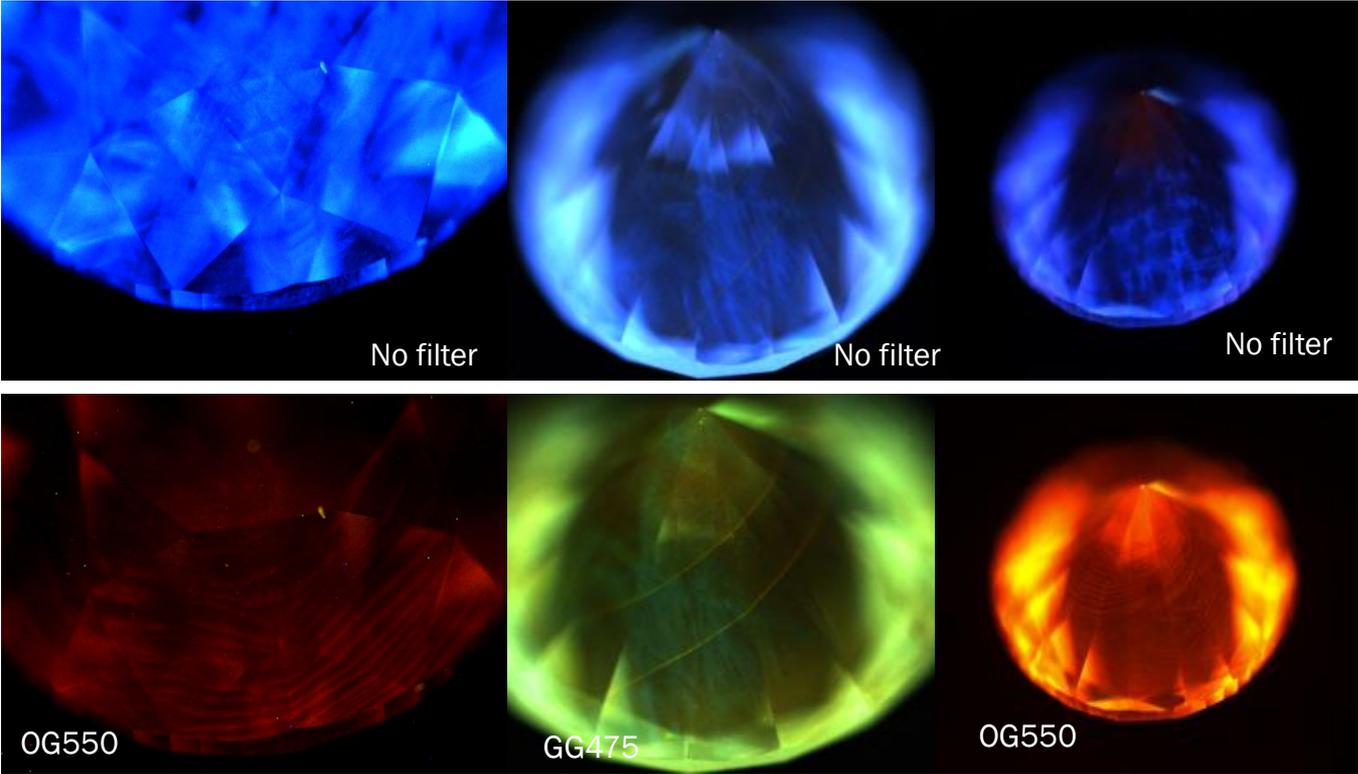
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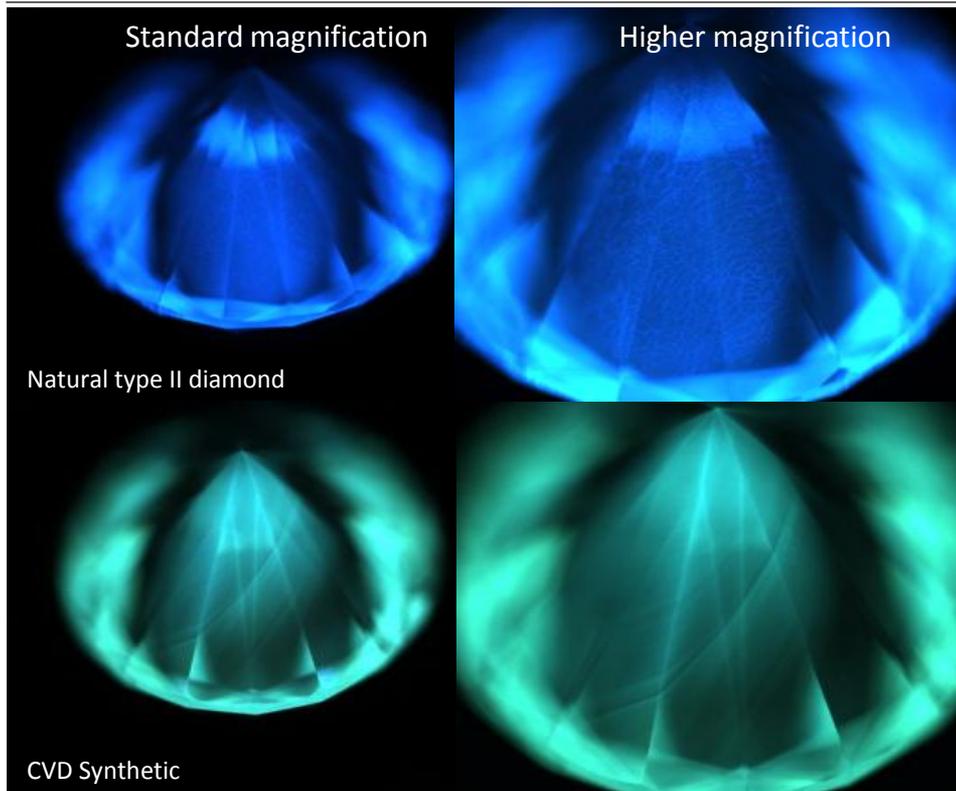
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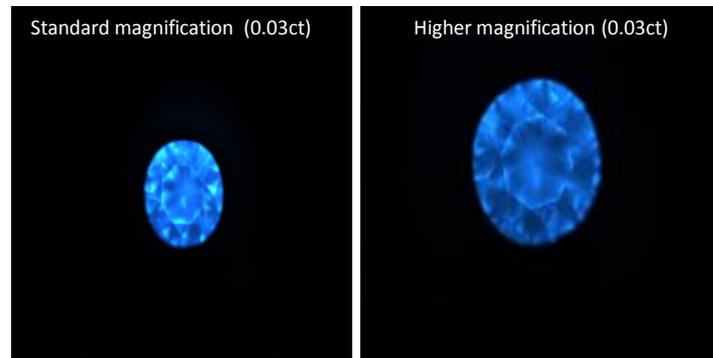


# COMPARISON OF STANDARD MAG VS HIGHER MAG SYSTEM

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- x1.6 higher optical magnification
- Further magnification possible digitally
- Useful for looking at fine detail
  - Natural type II diamonds
  - CVD-grown synthetics
- Higher mag system useful for AMS referrals (smaller sizes)



## DIAMONDDVIEW – SAMPLE HOLDER DEVELOPMENT

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# Instrument Screening Flow Chart

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## *DiamondSure*

All polished goods



~98% Pass  
(No further testing required\*)



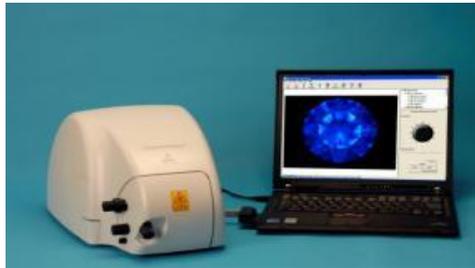
\*however, fancy colour type I Pass results will need additional testing for HPHT treatment

*DiamondView*



All synthetics/ simulants  
+ ~2% of natural diamonds

*DiamondPLUS*



Natural type II

(HPHT treatments test)





*Measures the way diamonds  
absorb light*

- Rapid screening instrument
- Very easy to operate
  - Measurement time: 4 seconds
- ALL synthetics & simulants are referred for further tests
  - Type II diamond
  - Synthetic Moissanite
- Mounted goods tested with extendable probe
  - Auto-detect capability for initiating measurement
- Removable dish for testing melee goods



- All DiamondSure referrals are tested on DiamondView
- Generates a surface fluorescence image from which synthetics may be identified
- Loose and mounted goods
- Needs user interpretation

## AUTOMATED MELEE SCREENING (AMS)

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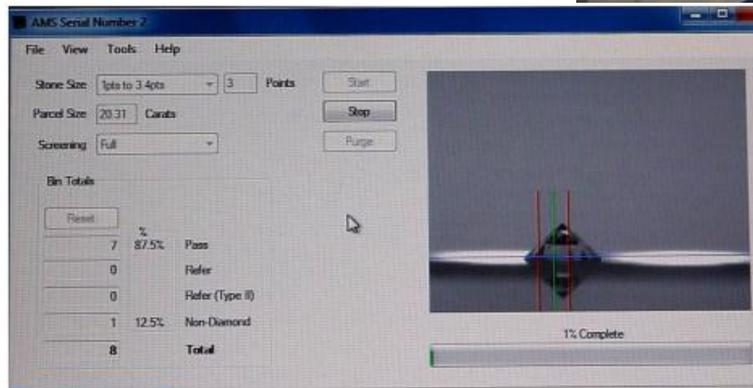
- Designed for testing
  - Colourless and near-colourless
  - 0.01 – 0.20 ct
  - Round brilliants
- Automatic feeding, testing and dispensing
  - Speed: 360 stones/hr
- Can be left unattended
  - One person can run many machines while carrying out other tasks
  - Overnight running is possible



# AMS OUTPUT CATEGORIES

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- Pass
  - not synthetic or simulant
- Refer
  - Rare result: further testing required
- Refer Type II
  - Low nitrogen. Could be synthetic. Further testing required.
- Non diamond
  - Simulant or synthetic
  
- Typical pass rates for natural diamond
  - 10 pts: 98%
  - 5 pts: 97%
  - 1 pt: 96%



- 
- Current synthetics can be detected using IIDGR diamond verification instruments used by key grading labs and regarded as an industry standard
    - Sold by IIDGR (<https://www.iidgr.com/>)
  - Automated melee screening equipment is available for sale to De Beers Sightholders and a screening service has been launched in China and will be launched in India from the beginning of H2 this year.
  - Our forward looking research continues to probe potential weaknesses of the identification methods so that improvements can be implemented if required
  - Practical detection technology will be part of the solution to the challenge that the trade currently faces in maintaining consumer confidence

## ACKNOWLEDGEMENTS

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I would like to thank the consumer confidence technical research team in Technologies UK for carrying out the research that has made this presentation possible.

In particular; Dr Brad Cann, Dr David Fisher, Dr Philip Martineau, Dr Nick Davies, Samantha Sibley, Dave Evans, Julia Samartseva, Christina Martins, John Freeth and Hugh Leach.