SYNTHETIC DIAMOND AND ITS IDENTIFICATION

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Dr Simon Lawson, De Beers Technologies UK
RESEARCH RESOURCES

• 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
• 65th De Beers Diamond Conference was held at Warwick University 7-10 July 2014
DE BEERS CONSUMER CONFIDENCE PROGRAMME

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

consumer confidence

Detection

Differentiation

Disclosure
TRADE DEFINITION OF DIAMOND & TERMINOLOGY

“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

- 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

- Temperature = 1300 – 1500°C
- Pressure = 50 - 60 kbar
DIFFERENT GROWTH CONDITIONS LEAD TO DIFFERENCES IN SHAPE

**Natural diamonds**
- Octahedral/dodecahedral

**HPHT Synthetics**
- Mainly cubo-octahedral
- Minor facets present
IDENTIFICATION FEATURES - INCLUSIONS

• Metallic inclusions
  – From solvent/catalyst
    • Rod-shaped
    • May be attracted to a magnet
HPHT SYNTHESIS V. NATURAL FORMATION

• 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
POLISHED HPHT-GROWN SYNTHETIC UV ILLUMINATION:
FLUORESCENCE
DIAMONDVIEW IMAGES

Colourless Diamond

Colourless HPHT Synthetic
DIAMONDVIEW IMAGES - FLUORESCENCE

Colourless Diamond

Colourless HPHT Synthetic
DIAGRAM OF MICROWAVE CVD REACTOR

Microwaves

Vacuum window

Gases in

Observation window

To pump

Substrates

Pressure ~ 0.1-0.2 bar
Gases: typically methane and hydrogen
CVD SYNTHEtICS IN A CVD REACTOR DURING GROWTH

Image from Scio Diamond – Built in America:
http://www.youtube.com/watch?v=iQXVya-HAOk
EXAMPLES OF PART-PROCESSED CVD SYNTHEtics

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

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

Examples of dark and light inclusions observed in heat treated CVD synthetics.
• Near-colourless CVD synthetics are nominally type II (DiamondSure & IR)
• 1332 and 1344 cm$^{-1}$ ($N_S$)
• 3123 cm$^{-1}$ Not present after annealing
PHOTOLUMINESCENCE SPECTROSCOPY

- **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

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°C).
• There is a very noticeable colour improvement from P-T colour to I colour.
DIAMONDVIEW FLUORESCENCE IMAGES OF CVD SYNTHETICS

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
Colourless material
- Moderate size
- Orange fluorescence & striations
- Relatively high purity
- Not heat treated
Birefringence indicates relatively low strain

Bluish green fluorescence and blue phosphorescence

Fine-to-medium striations, and sometimes zoning in DiamondView images
DIAMONDVIEW (DV3F)

- 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

Natural

CVD Synthetic
DIAMONDVIEW – IMPROVED DETECTION WITH FILTERS

Natural

CVD Synthetic
DIAMONDVIEW – IMPROVED DETECTION WITH FILTERS

- No filter
- OG550
- GG475
- OG550
COMPARISON OF STANDARD MAG VS HIGHER MAG SYSTEM

- 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)
DIAMONDVIEW – SAMPLE HOLDER DEVELOPMENT
Instrument Screening Flow Chart

**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)
DIAMONDSURE™

- 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

Measures the way diamonds absorb light
DIAMONDVIEW™

- 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)

- 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

- **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%
SUMMARY

• 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
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