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# Data collection on chemical vapor deposition synthetic diamonds confirming the achievability of high quality as-grown CVD synthetics.

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### ABSTRACT:

Three, almost colorless, CVD synthetic diamonds were extensively studied with standard gemology techniques and advanced laboratory equipments as those furnished by M&A Gemological Instruments. The results obtained indicated, as expected, that these stones were CVD synthetics. They are type IIa as-grown CVD synthetic diamonds with spectroscopic and imaging properties matching with a Washington Diamond Corporation reference CVD gem diamond. Through DTC DiamondView™ imaging, the trace of growth planes was observed in two stones. An unusual absorption feature centered at 753 nm was shown in the Vis-NIR spectra of one sample and was not active in the 532 nm laser excitation PL spectroscopy. The F color sample reminds us that good quality CVD synthetic diamonds can be achieved without the need of further HPHT annealing treatment.

**INTRODUCTION:**

In the process of collecting and studying synthetic and/or treated colorless diamond references, the author had the opportunity to acquire IGI-certified CVD synthetic diamonds (Fig. 1) from an undisclosed brand/origin. These stones were analyzed in order to collect data, learn as much as possible from them and, if possible, compare results with well documented reference CVD.

On the other hand this study made it possible to evaluate specific spectrometers and different ways of capturing signal in Vis-NIR spectrometry (% transmission vs absorption).

**MATERIAL AND METHODS:**

Three samples were bought through internet (JMA LinkedIn network). They are numbered as follows: see Table I.

The stones were laser inscribed “LABORATORY GROWN IN USA IGI LG...” (Fig. 2).

This kind of inscription, associated with IGI report, is indicative of the Washington Diamond Corporation (WDC) CVD synthetic diamond production.

Shortwave ultraviolet (SWUV) transparency was evaluated with a homemade setting allowing visualization of transmitted SWUV on a fluorescent F254 silicagel surface.



Fig. 1: The three 0.30 to 0.35 ct CVD synthetic diamond samples studied here and described in table I Note: Top oval 0.35ct K, bottom oval 0.34ct F. Field of view ~30 mm. Picture Mikko Aström.

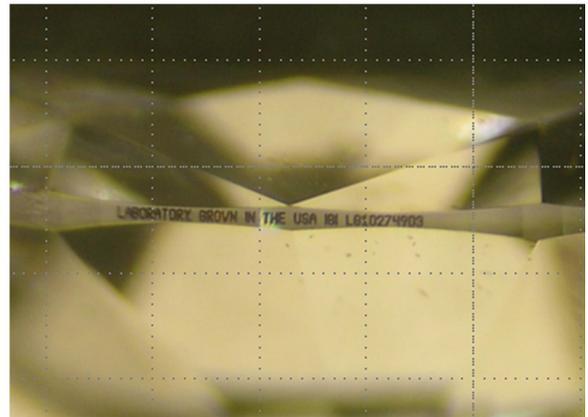


Fig. 2: All the stones were laser inscribed by IGI with the mention “LABORATORY GROWN IN THE USA IGI” followed with the reference number LG10274903 in this case. Magnification ~30X. Picture J.-M. Arlabosse

Table I: Description table of the three studied samples from internet plus one MAGI (MAGI: M&A Gemological Instruments) reference sample from WDC (WDC: Washington Diamond Corporation) production (Fig. 1).

ID	Weight (ct)	IGI-Color scale	IGI-Purity scale	Shape	Source
LG10274903	0.34	F	VS1	Oval	Internet
LG10260207	0.30	I	I1	Pear	Internet
LG10265711	0.35	K (faint color)	SI1	Oval	Internet
LG10275617	0.30	G	I1	Round	WDC : MAGI Ref.

Visible-NIR spectroscopy was conducted with two different instruments.

First, an Ocean Optic USB 4000 spectrometer equipped with a home-made setting allowing to capture signal from an integration sphere while the stone is immersed in liquid nitrogen (in order to reach best resolution and signal/noise ratio). Spectral domain was 300 to 1000 nm with a resolution of 1.0 nm. The resulting curves were set in % Transmission (default software setting for integration sphere measurement).

Second, a MAGI GemmoSphere™ spectrometer set with an integration sphere. Spectral domain was 365 to 1000 nm with 1.0 nm resolution. In this case, the software setting, for resulting curves, was set in Absorbance.



*Fig. 3: MAGI GemmoFtir™ spectrometer.  
Picture Mikko Aström.*

Fourier transform infrared (FTIR) spectrometry was done with a MAGI GemmoFtir™ spectrometer (Fig. 3) using a low noise DLaTGS detector and equipped with a diffuse reflectance type (DRIFT) signal capture device and was run at 4 cm<sup>-1</sup> and 1 cm<sup>-1</sup> resolutions. Spectral range was 400 to 7000 cm<sup>-1</sup>.

FTIR spectrometry was equally done with a BRUKER Alpha spectrometer using a low noise DLaTGS detector and equipped with a diffuse reflectance type (DRIFT) signal capture module and was run at 4 cm<sup>-1</sup> and 0.8 cm<sup>-1</sup> resolutions. Spectral range was 400 to 8000 cm<sup>-1</sup>



*Fig. 4: MAGI GemmoRaman-532SG™ spectrometer and the cryogenic cell used for measurements at the liquid nitrogen temperature. Picture Mikko Aström.*

Photoluminescence (PL) spectroscopy essays were conducted using a MAGI GemmoRaman-532SG™ (Fig. 4) device equipped with a compact 200 mW, 532 nm DPSS Laser (Diode Pumped Solid-State Laser allowing compactness and efficiency) covering 536 to 760 nm spectral range at 0.2-0.3 nm resolution and a MAGI prototype Raman/PL spectrometer equipped with a 200 mW, 405 nm DPSS Laser covering a spectral range from 412 to 900 nm at 0.7 nm resolution.

Very short UV imaging was done using a - DTC : Diamond Trading Company - DiamondView™ (Welbourn et al., 1996).

## RESULTS:

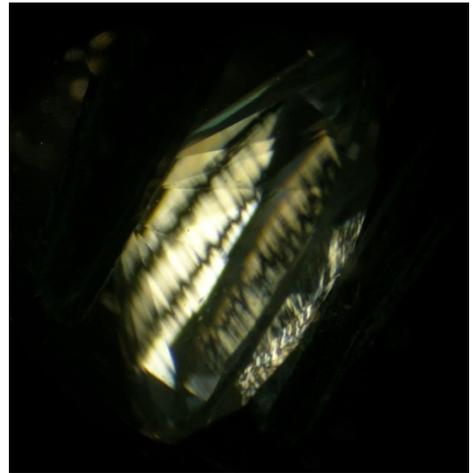
Sharpness of the facets edges and brilliancy suggested diamond for the three studied samples.

Transparency to short wave UV (~254 nm) radiations indicated these stones could be IIa or IaB type diamond. As the majority of colorless CVD synthetics diamonds are of type IIa (Martineau et al. 2004) and this diamond type is a rare naturally occurring one, spotting IIa type by using transparency to SWUV radiation is a useful test to perform.

Observation between crossed polarizing filters show “brush like” stress pattern (Fig. 5a).

This observation can be made almost whatever the rotation of the stone was around an axis perpendicular to the table facet (Fig. 5b).

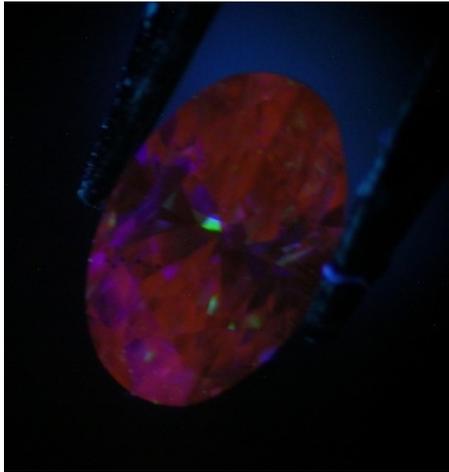
These observations, between crossed polarizing filters, of some distinct “brush-like” irregular stress patterns nearly perpendicular to the stone’s table facet and following the stone rotation is to be considered as strongly indicative of CVD-grown synthetic diamonds. This was the case for all diamond studied here (Fig. 5b).



*Fig. 5a: “Brush like” stress pattern is easily seen below the girdle when viewed between crossed polarizing filters and immersed in methylene iodide as in the LG10265711 sample depicted here. Field of view ~7 mm. Picture J.-M. Arlabosse*

	Initial position	Rotate 1	Rotate 2	Rotate 3
G 10274903  F				
LG 10260207  I				
LG 10265711  K				

*Fig. 5b: Different polariscope views, when immersed in methylene iodide and rotated around an axis perpendicular to the table facet. Field of view ~10 mm. Pictures J.-M. Arlabosse*



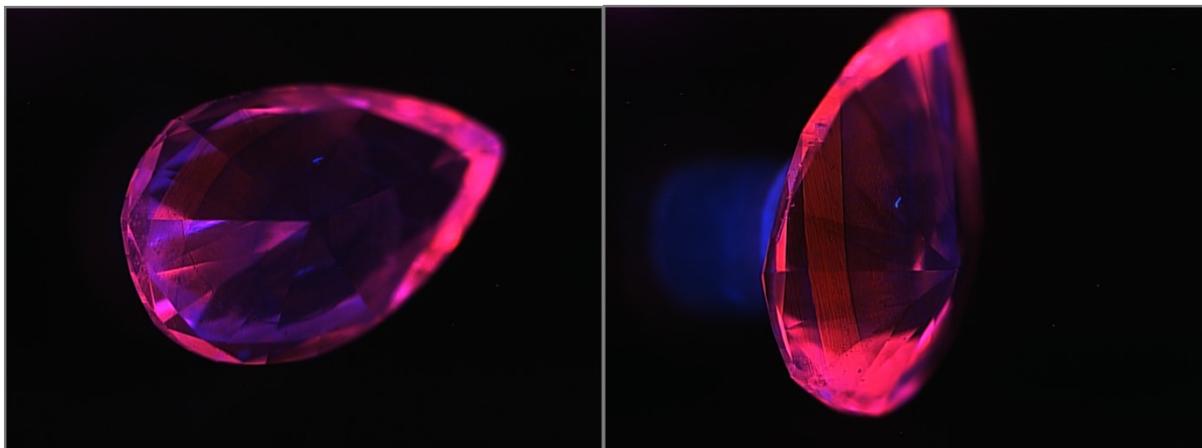
*Fig. 6: The most colored sample LG10265711, a 0.35 ct K-faint color/SI1 oval brilliant, showed orange fluorescence under 254 nm SWUV. Field of view ~7 mm. Picture J.-M. Arlabosse*

All samples were inert under long wave (366 nm) UV radiation.

Under short wave (~254 nm) UV radiation, the most colored stone (K color LG10265711) showed orange fluorescence (Fig. 6) with short time red phosphorescence. The stone LG10260207 with I color showed a faint pinkish fluorescence without appreciable phosphorescence whereas the F color sample (LG10274903) was inert.

Having a diamond with fluorescence intensity greater under SWUV than under LWUV is often considered as a warning regarding possible synthetic origin (Shigley, 2005; Delaunay, 2016).

Using very short wave photoluminescence imaging (DiamondView™), every sample showed short-lasting phosphorescence. The pear shaped LG10260207 stone showed purple, blue and pink fluorescence colors with striations and very distinct red band (Fig. 7a).



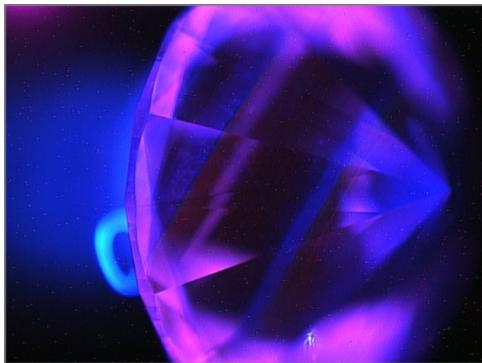
*Fig. 7a: DiamondView™ photoluminescence imaging showed purple, blue and pink fluorescence colors in the pear shaped LG10260207 stone with striations and red band due to start-stop growth. Magnification ~15X. Photo Mikko Aström.*

Similar observations were made with the LG10265711 sample (the most colored one) (Fig. 7b). This observation was equally and previously seen in the MAGI's reference (Fig. 7c).

However, the most colorless stone LG10274903 showed some striations (not easily visible on Fig. 7d) and blue, pink colors component of fluorescence without obvious color band (Fig. 7d). The sharp color zoning observable in very-short wave UV imaging is the result of multiple start-stop growth (Dieck et al., 2015).



*Fig. 7b: DiamondView™ photoluminescence imaging showed strong pinkish-orange fluorescence colors in the most colored stone LG10265711. Striation and a red red band due to start-stop growth can be seen. Magnification ~15X. Photo Mikko Aström.*

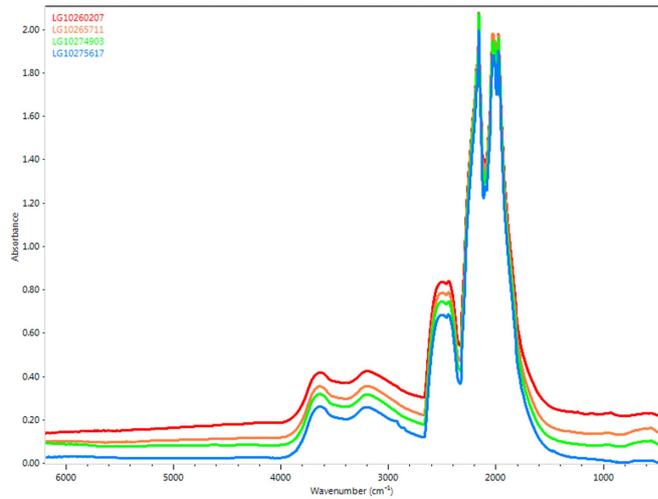


*Fig. 7c: Bands with different fluorescence color in DiamondView were previously observed in the WDC diamond reference stone. Magnification ~20X. Photo MAGI reference library.*



*Fig. 7d: In the less colored stone LG10274903, DiamondView™ imaging showed pink and blue emission colors. Although there were not easily visible on the photo, some striations were actually visible. Magnification ~15X. Photo Mikko Aström.*

FTIR spectroscopy indicated that, whatever the resolution was, all the samples were of type IIa.



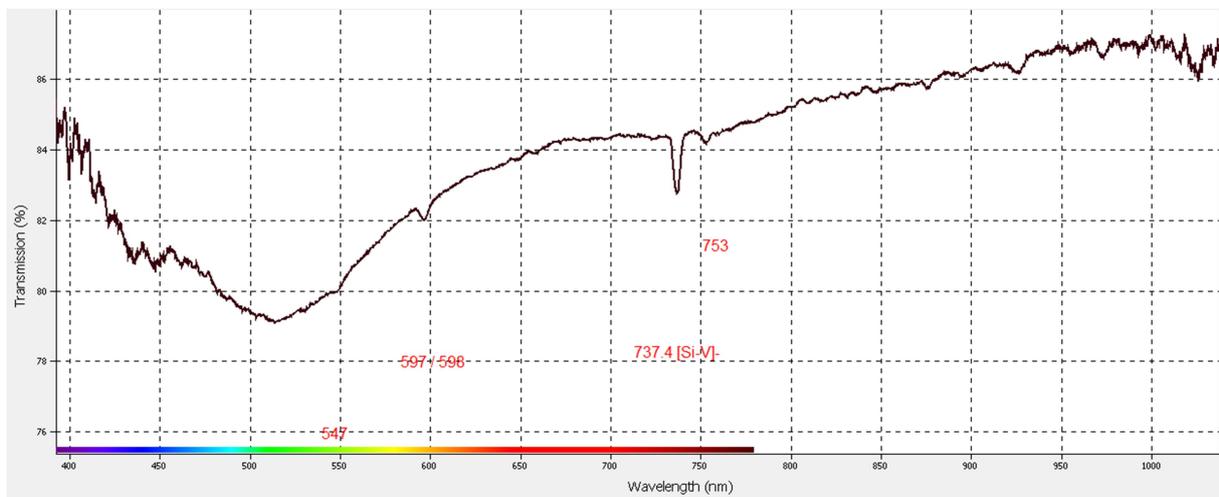
*Fig. 8: All samples have the same Type IIa FTIR spectrum. No appreciable quantity of nitrogen or boron nor H-related defects were detected. Picture Mikko Aström.*

No trace of isolated nitrogen (Ib type, C centers) or hydrogen related defects were seen (Fig. 8), nor any signs of boron.

Note that the absence of specific H-related defects in CVD synthetics such as absorptions around 3123  $\text{cm}^{-1}$  and/or 3323, 6524, 6856, 7354, 8753  $\text{cm}^{-1}$  (Martineau et al. , 2004) does not mean the stone has received post grown HPHT annealing.

Actually some high purity as-grown CVD synthetic could have FTIR spectrum that show intrinsic absorption only (again see Martineau et al. 2004).

Visible absorption spectra are collected to check for potential weak absorptions, as the stones are near-colorless.



*Fig. 9a: Visible spectrum, using the Ocean Optic spectrometer, of the most colored sample LG10265711 (the 0.35 ct. K-faint color/SI1 oval brilliant) recorded when the stone was immersed in liquid nitrogen indicated 547 nm, 597-598 nm peak (possibly unresolved doublet), strong SiV center at 737 nm, and almost never seen 753 nm center. This spectrum was set in %Transmission (peaks oriented down) because of typical software setting used for this reflection-like signal capture. Picture J.-M. Arlabosse*

In the K - faint color stone (LG10265711) visible spectrum, measured at  $\sim 77\text{K}$  (liquid nitrogen temperature) with the Ocean Optic home-made setting, revealed distinct and reproducible absorption features as a shoulder near 547 nm, a peak (possibly an unresolved doublet) at 597-598 nm, distinct peak at 737 nm and a peak at 753 nm (Fig. 9a). All these features, except the 547 nm



one, were seen also, though weaker, at room temperature.

In the two other stones only a very weak peak at 737 nm could be guessed (Fig. 9b).

In these very cases, because the signal/noise ratio was around 2 this spectral feature should not be considered as representative (signal/noise ratio must be of 3 or greater to be considered as representative, Fritsch pers. Com. 2017). However, although a too weak signal at 737 nm is not a proof, it should lead to more investigation regarding possible CVD synthetic diamond.

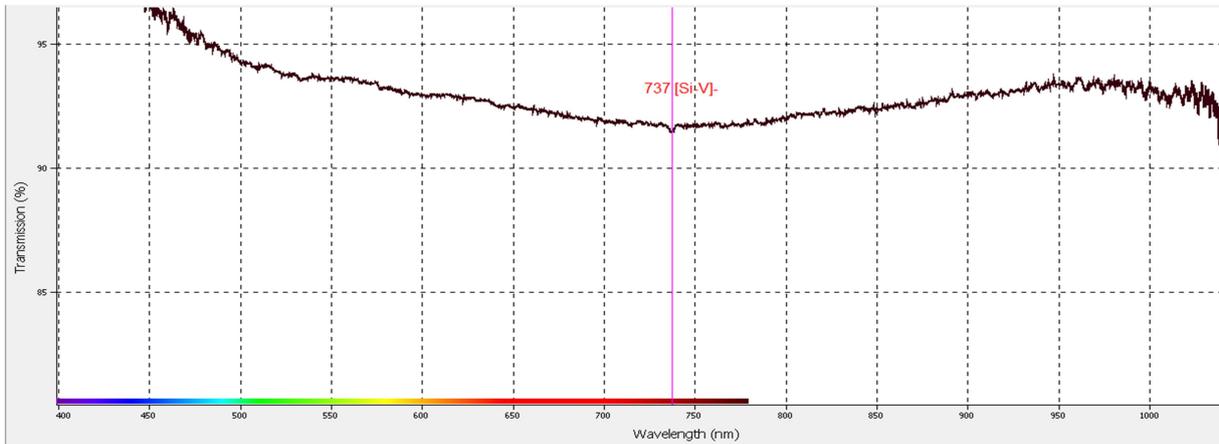


Fig. 9b: Visible spectrum, using the Ocean Optic spectrometer, of the less colored sample LG10265711 (LG10274903 a 0.34 ct. F/VS1 oval brilliant) recorded when the stone was immersed in liquid nitrogen indicated only a possible very weak SiV peak at 737 nm. Though this peak was too weak to be considered as representative, it should however order more investigations. This spectrum was set in %Transmission (peaks oriented down) because of typical software setting used for this reflection-like signal capture. Picture J.-M. Arlabosse

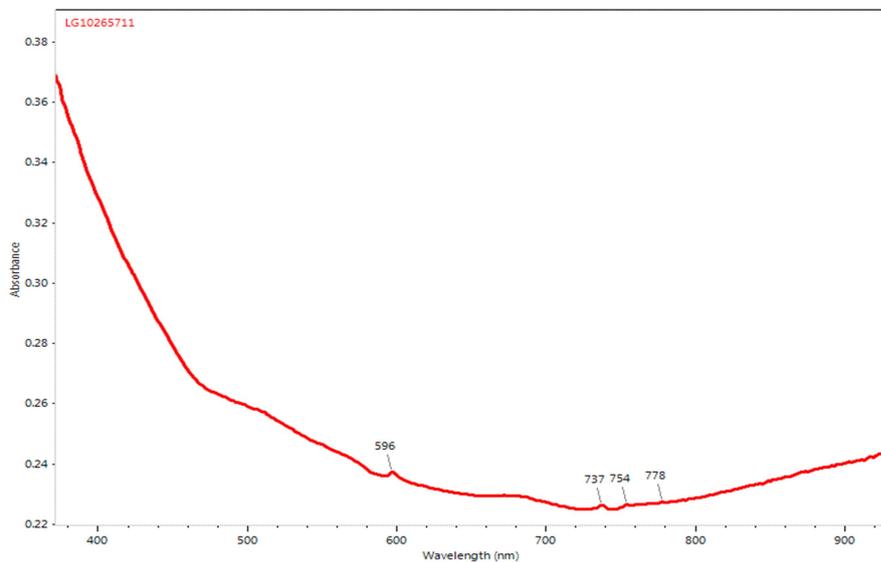


Fig. 10: Visible spectrum, using MAGI spectrometer, of the most colored sample LG10265711 (the 0.35 ct. K-faint color/SI1 oval brilliant) recorded at room temperature.

596-598 nm peak, 737 nm SiV, and 753-754 nm absorption features can be seen. This spectrum was set in Absorbance (peaks oriented up) because of typical software setting used by MAGI for its spectrometer setting. Picture Mikko Aström.

Visible-near infrared (Vis-NIR) spectroscopy of the sample showing the most intense absorption features (i.e. LG10265711, the 0.35 ct K-faint color/SI1 oval brilliant), when tested at room temperature with the MAGI Vis-NIR spectrometer, indicated the presence of the 753-754 nm feature as well as all others features that were previously identified (Fig. 10).

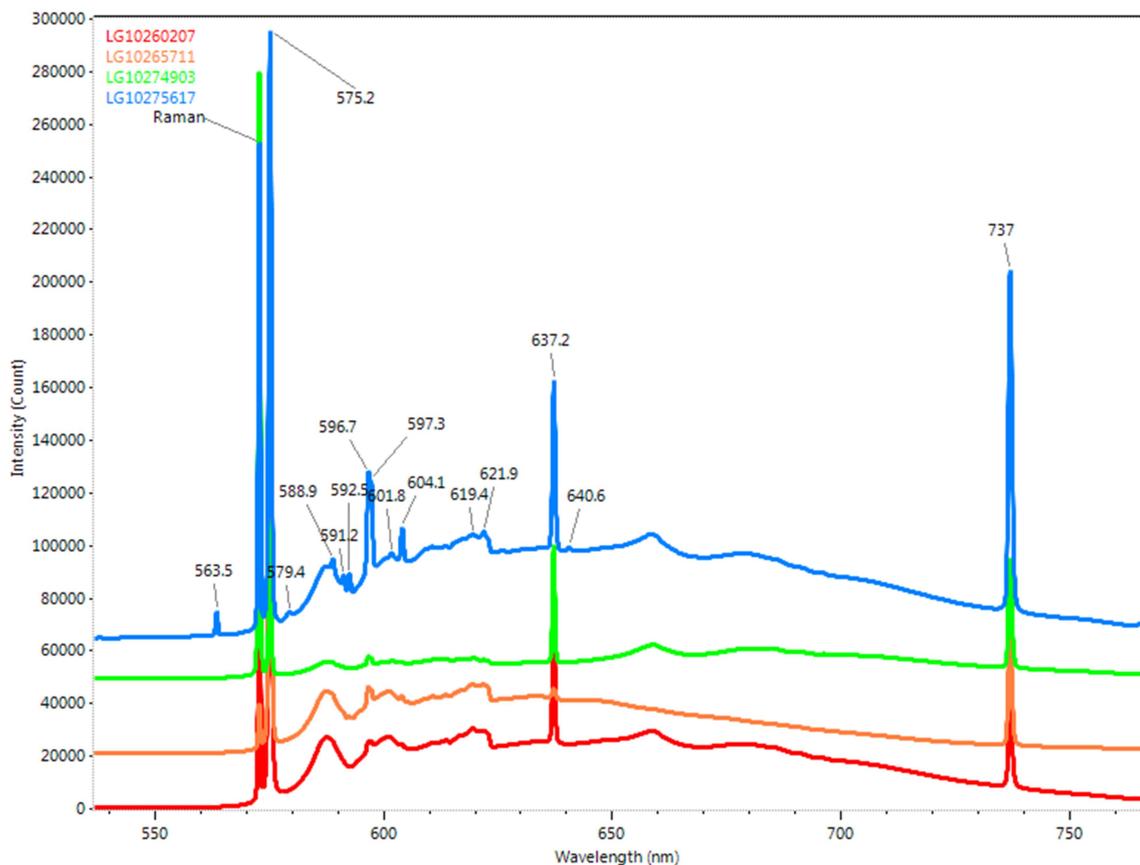
Visible spectroscopy at liquid nitrogen temperature, in some case, can show the 737 nm SiV center. Although this defect was very intense in the most colored stone (i.e. LG10265711 K-faint color oval ), it was at the spectrometer limit of detection in the two other samples (LG10274903 F oval & LG10260207 I pear). The feature at 596-598 nm (believed to be an unresolved doublet), that is strongly indicative of as-grown CVD synthetics (Martineau et al., 2004), was equally seen in the most colored sample.

In the most colored stone again, an uncommon peak at 753-754 nm has been found.

Because of the “isolated position” of this 753-754 nm peak in the spectrum and its small width, we assumed that it couldn’t came from a vibronic structure.

The SiV feature at 737 nm is well known in photoluminescence (PL) and can sometimes be seen in absorption. This is present in most of CVD synthetics (Martineau et al, 2004). The 596-598 nm feature has previously been described in Vis-NIR or PL spectroscopy in some as-grown CVD synthetic (Dieck et al., 2015; Martineau et al., 2004). Regarding the 753 nm peak, we found (to date) just one record of it in a LPHT-Annealed pink CVD synthetic diamonds (Soe Moe et al. 2015).

The PL spectroscopy with 532 nm excitation laser (Fig. 11) didn’t show the 753 nm center. However, all the samples showed emissions at about 575 nm (NV<sup>0</sup>), 637 nm (NV<sup>-</sup>), 737 nm (SiV), partially resolved doublet in 596-598 nm region and 604 nm PL features.



*Fig. 11: 532 nm laser excitation PL spectroscopy at the liquid nitrogen temperature gave similar results for all four samples: 596-597 nm doublet ( partially resolved), SiV 737 nm and a combination of NV<sup>0</sup> 575 nm and NV<sup>-</sup> 637 nm indicate all the stones are indeed CVD synthetic diamonds and that they look very similar to the WDC reference (blue trace).*

*The presence of the 596-597 nm center indicated these sample weren’t HPHT post treated (Martineau et al, 2004). Picture Mikko Aström.*

In 405 nm laser excitation PL spectroscopy (Fig. 12), strong emission features were seen at 467 and 481-482 nm.

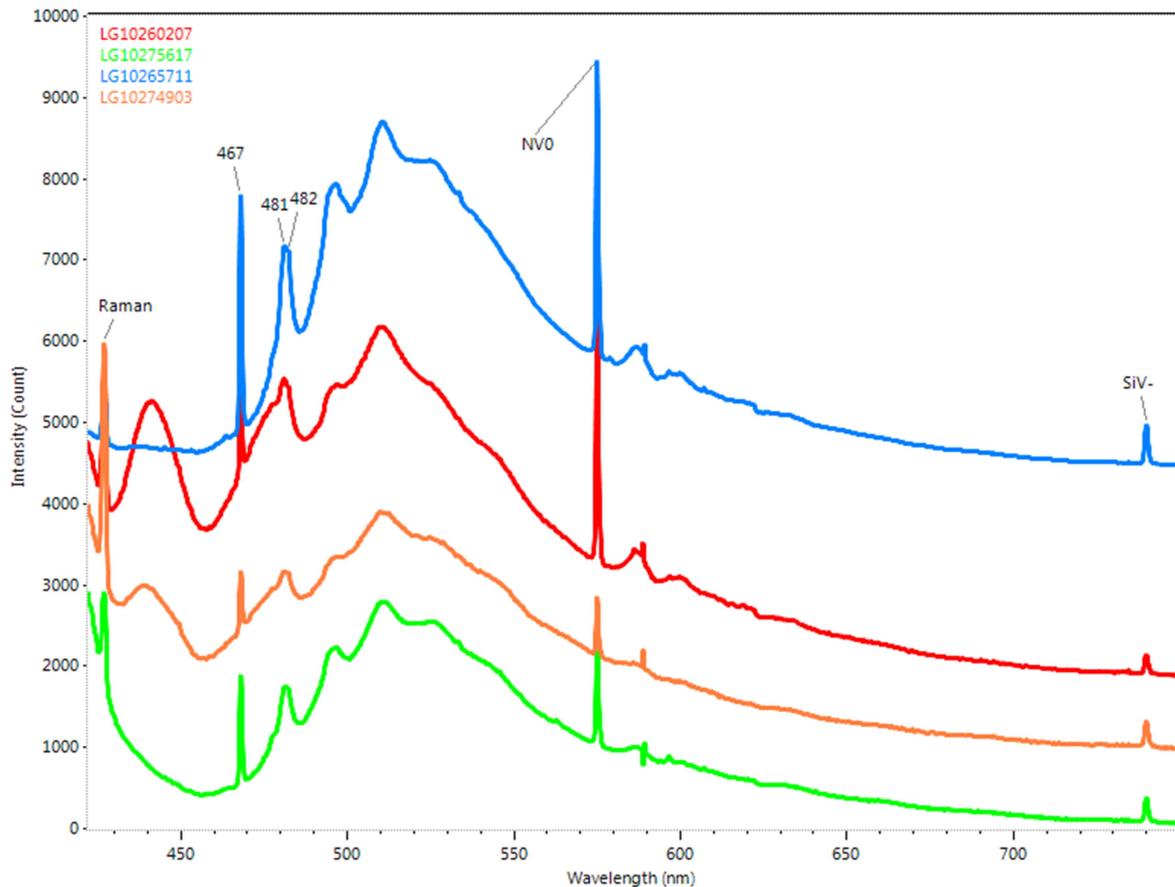


Fig. 12: 405 nm laser excitation PL spectroscopy at the liquid nitrogen temperature indicated same profile for each sample. 467 nm, 481-482 doublet, 575  $NV^0$  (Note the NV 637 nm center was not seen under this excitation), SiV 737 nm indicate all the stones are indeed CVD synthetic diamond and that they look very similar with WDC reference (blue trace). The presence of the 467 nm center indicated these samples weren't HPHT post treated (Martineau et al, 2004). Picture Mikko Aström.

Though the 753-754 nm absorption feature observed in the LG10265711 sample was reproducible with two different spectrometers, even at room temperature, with Vis-NIR spectroscopy, PL spectrum obtained with a 532 nm laser excitation indicated this defect is not a PL-active one at this excitation wavelength.

The presence of the 596-598 nm doublet and/or the 467 nm centers observed in every PL spectrum indicated these stones would not have been subject to post grow HPHT annealing (Martineau et al, 2004). The three samples studied here and the WDC reference should thus be considered as "as grown CVD synthetic".

Because almost all observations (and girdle inscriptions) on the three samples studied are very similar to data obtained for WDC reference sample, it is proposed that these stones are synthetic CVD diamonds that come from same process as the one used for producing Washington Diamond Corporation synthetic diamonds.

## CONCLUSIONS:

These data indicate that high purity and very good color (e.g. VS1 / F color sample LG10274903) synthetic CVD diamonds can be reached as grown, without the need of post-grown HPHT treatment. The will to achieve ultrapure material without time and/or cost consuming post-growth techniques, is logical when considering the potential of the colorless synthetic diamond market.

Specific gemology-designed and portable equipment used here (e.g. portable FTIR spectrometers, Raman-532SG™ and Vis-NIR spectrometers) prove to be efficient and well suited for diamond testing, even in most challenging cases as for high purity CVD synthetic.

Even if girdle inscription is absent (or removed), we saw too that some conjugation of observations (e.g. DiamondView) and spectroscopic profiles (e.g. PL spectroscopy) can be representative of a specific brand of CVD synthetic diamond like in this very case with these WDC synthetic diamonds.

In addition to these observations, a distinct absorption feature has been seen at 753-754 nm in one of these as-grown CVD synthetic diamonds. To the best of our actual knowledge, we only saw one record of a tiny 753 nm peak in a spectrum image of an LPHT-Annealed Pink CVD Synthetic Diamonds (Soe Moe et al. 2015). It could be interesting now to know (among other things) whether this defect will anneal out in HPHT treatment or not.

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