NEWSLETTER
Summer 2014
ISSUE No.3 / 2014
Chief Editor Jayshree Panjikar Editor Tay Thye Sun www.icglabs.org

Identification of CVD Synthetic Diamonds

By Kook-WheeKwak, Wooshin Gemological Institute of Korea (WGK)

Colorless or near colorless CVD synthetic diamonds are posing a serious commercial challenge to natural diamonds,as gem quality synthetic diamonds are produced and distributed routinely nowadays. Separating synthetics from natural diamonds, however, is very difficult using conventional gemological tests, since high quality synthetics hardly have a gemological feature that distinguishes them from natural diamonds. Thus advanced spectroscopic tests including FT-IR, PL (Photoluminescence) spectrometer, and fluorescence imaging tools are needed for the identification of synthetic diamonds.

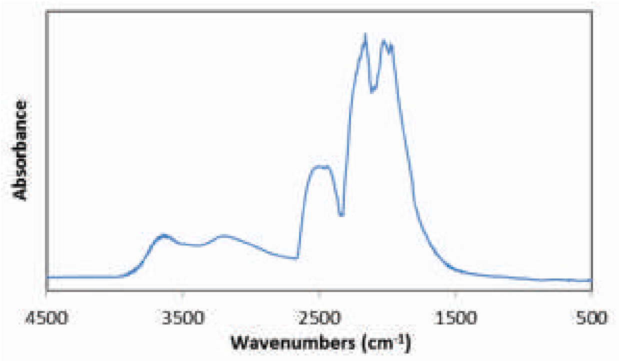


Fig.1.FT-IR spectrum of a type IIa diamond

The first step of the spectroscopic tests is to determine the type of the diamond using FT-IR. It is well known that colorless synthetic diamonds are of type IIa, the diamonds identified by FT-IR as type I can be considered as natural, while those identified as type IIa (Fig. 1) need further investigations.

The next step is PL measurement (Fig.2). When a diamond is exposed to a laser beam, defects in the diamond emit light with wavelengths specific to each defect; this emitted light is called PL. So, PL spectrum can reveal the defects contained in the diamond.

During CVD synthesis process, silicon is often introduced into the synthesized diamonds from the Si-containing components in the CVD reaction chamber.737/738nm double peak in PL spectrum (Fig. 3), due to a Si-related defect, is a very strong indicator of CVD synthetic diamonds. However, it should be noted that the Si-related defect is recently reported to exist in natural diamonds, even though the occasion is very rare (Wang et al., 2012). Thus, another test to distinguish synthetics from natural is desirable.

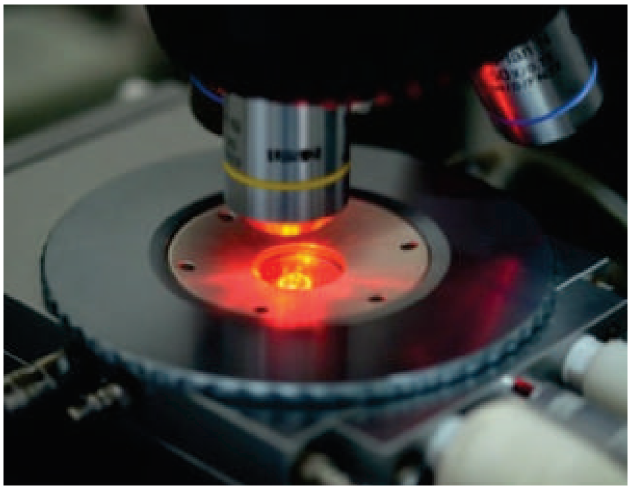


Fig.2.PL measurement of a diamond with 633nm red laser excitation

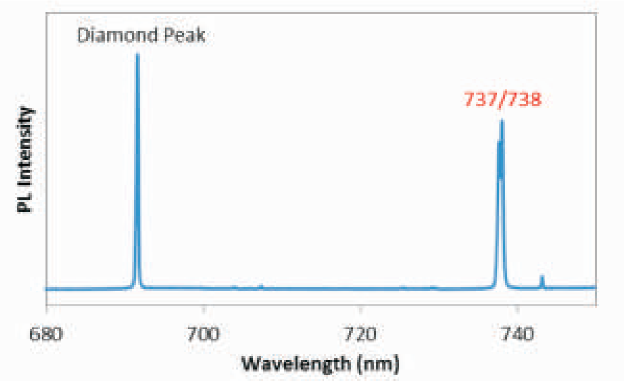
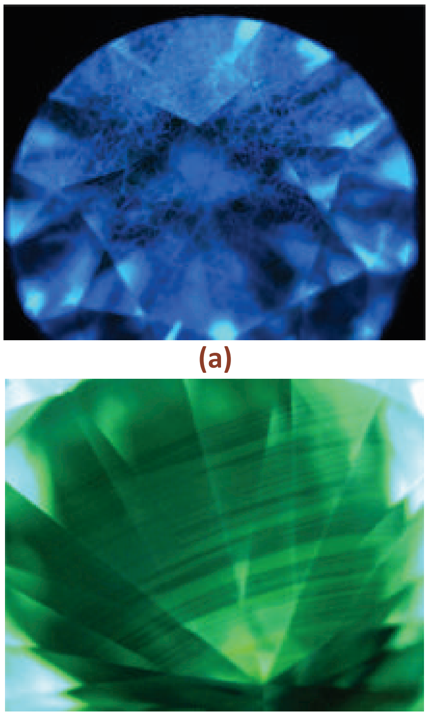


Fig.3.PL spectrum of a CVD synthetic diamond (633nm excitation)

Synthetics produce fluorescence images distinct from those of natural diamonds when exposed to a strong short-wavelength ultraviolet (UV) light. They also exhibit different fluorescence patterns depending on growth conditions, such as nitrogen contents. Fig. 4 shows UV fluorescence images of type IIa natural and CVD colorless diamonds recently identified by WGK. identified by WGK. Fluorescence images were observed using DTC's DiamondView™.

Natural diamonds can produce a variety of fluorescence images. Fig. 4(a) shows one of the typical images; blue fluorescence with a so called "mosaic" network pattern. The mosaic patterns, originated from dislocations in the crystal structure, have been observed only in natural type IIa diamonds, a conclusive feature distinguishing natural diamonds from synthetic ones.Fig. 4(b) shows a typical fluorescence image of CVD diamond green growth lines with blue/green fluorescence. The growth line pattern is a unique and the most important visual identification feature of CVD synthetic diamonds(Wang et al., 2012).



(a)

(b)

Fig.4.Fluorescence images of (a) a natural diamondand (b) a CVD synthetic diamond

In summary, owing to the advances of synthesis techniques, the quality of CVD synthetic diamonds have improved so much that distinction of synthetics from natural diamonds is very difficult using conventional gemological tests. However, the synthetic diamonds can be identified by the combined use of PL and fluorescence image tests.

Reference:

W. Wang, et al. (2012), CVD Synthetic Diamonds from Gemesis Corp., Gems & Gemology, Vol. 48 No. 2, pp. 80-97

Recent CVD diamonds tested
at
Pangem Testing Laboratory

Jayshree Panjikar and Aatish Panjikar, Pangem Testing Laboratory, Pune, India
jayshreepanjikar@gmail.com

Eight faceted CVD synthetic diamonds ranging from 1.08 ct to 1.17ct (Fig. 1) had come for certification to our laboratory. Standard gemmological tests like thermal conductivity, specific gravity, fluorescence under ultra-violet light were carried out on the stones.

All stones conducted heat, had SG=3.51to 3.53 and showed no fluorescence under UV light. This was rather strange as generally in a packet of diamonds at least one would show some fluorescence.

Fig 1: CVD diamonds ranging from 1.08 to 1.17carats

The synthetic diamonds had a distinct brownish tint was very clearly observed along the girdle when compared with a J colour Diamond Master (Fig 2)

Fig 2: Distinct brownish tint was observed for all the synthetic CVD diamonds

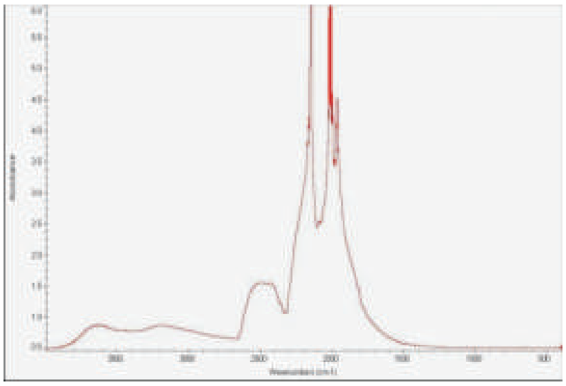


Fig 3 All diamonds were Type Iia

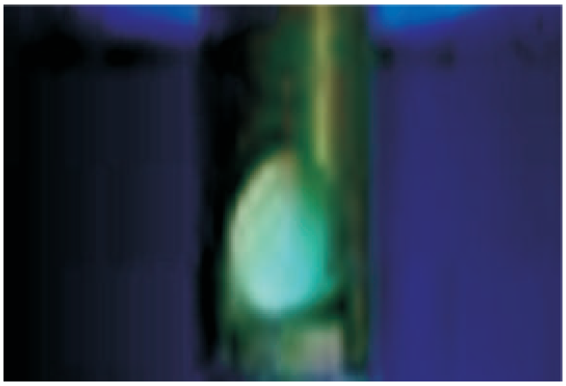


Fig 4 Type IIa determined by Diamond spotter

All these diamonds were of Type IIa was determined by Diamond Spotter and confirmed by FTIR. Under Crossed Polar Filters attached to the microscope interference colours were observed characteristic to CVD diamond. (Fig 3 & 4)

Although synthetic CVD diamonds do not show very conspicuous inclusions, these diamonds did have some characteristic features. Microscopic inclusions were observed in the form of pinpoints (Fig 5). Some of the pinpoints were scattered and some formed fibrous structures of brownish colour (Fig 6 and Fig 7) giving rise to dense fibrous type of inclusions.

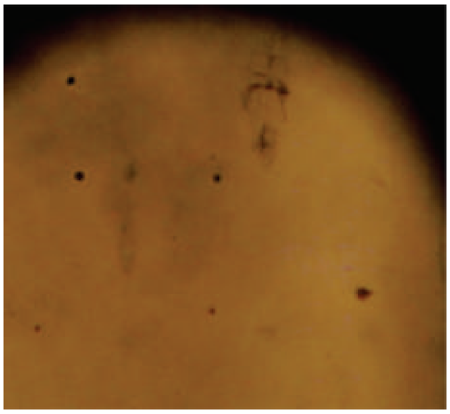


Fig 5 Fine fibrous inclusion

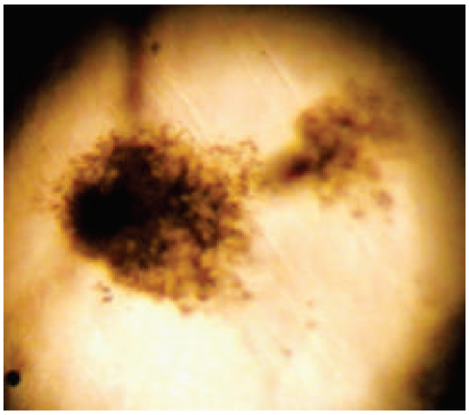


Fig 6. Dense Fibrous inclusions

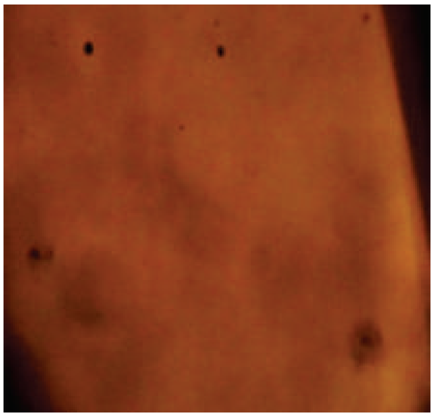


Fig 7 Fine dots and fibers

Under the microscope one could observe a clear frosted type of inclusion (Fig 8). When a crossed polar filter CPF is attached to the microscope (Fig 9) one can see the inclusions prominently. Besides one could see the characteristic interference figures (Fig 10) under the CPF attached to the microscope.

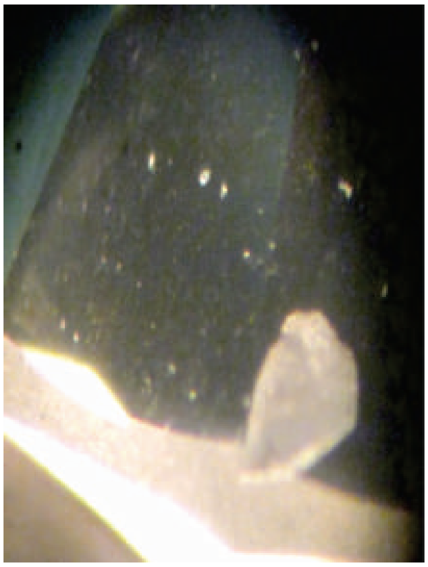


Fig8: Frosted appearance 60x

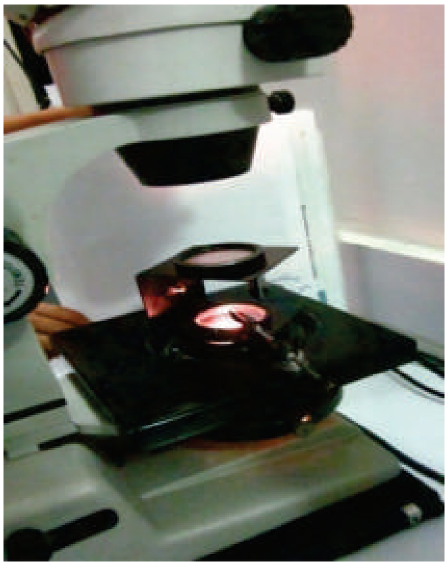


Fig 9: Microscope with CPF

Fig 10 Interference figures

Although instruments like DiamondPlus, DiamondCheck, Automatic Melee Sorting (AMS) instruments are available for verification, all these instruments can be used when the diamonds are loose, the problem arises when the diamonds are set in jewellery. It is the need of the hour to collect as much data as possible. CVD synthetic diamonds are available in all sizes and therefore very ideal for jewellery. In such cases when the CVD synthetic diamonds are set in jewellery then inclusions and the possible polarisation images may give some clue to their synthetic origin.

References:

C.M. Breeding and J.E. Shigley, The "Type" classification of diamonds and its importance in gemology, Gems & Gemology Vol.45, No.2, 96-111 (2009).

P.M. Martineau, S.C. Lawson, A.J. Taylor. S.J. Quinn, D.J.F. Evans and M.J. Crowder, Identification of synthetic diamond using chemical vapor deposition (CVD) . Gems & Gemology Vol.40, No.1, 2-25 (2004).

W. Wang, T. Moses, R. Linares, J.E. Shigley, M. Hall and J.E. Butler, Gem-quality synthetic diamonds grown by chemical vapour deposition (CVD method. Gems & Gemology, Vol.39, No. 4, 268-283 (2003)

D. Simic and Dejlanin B,.2010 " Identifying Diamond types and synthetic diamonds with CPF (Crossed Polar Filters)". Booklet D& S Gem service

Treated Black Diamond Earring

By Tay Thye Sun & Loke Hui Ying

Far East Gemological Laboratory, Singapore

A pair of black diamond earring was submitted to our lab for testing and the customer wanted to know whether the diamond in the earring was natural or not. The diamond earring weighed 13.21 grams and the measurement was approximately 2 x 1.6 x 0.4cm. The surface of the diamond was studded with many small rounded gold pin drilled into the black diamonds.

Fig. 1. A pair of black diamond earring weighs 13.21 grams with gold pin studs (Photo by Tay).

Our first test was using the Presidium Diamond Test pen, and the result indicated diamond but it gave a beep sound. As it is rather unusual for test pen to give the beep sound on touching a diamond sample unless the probe touches gold or alloy metal. Tests were conducted on the front and hind sides of the diamonds, all gave beep sound except in the cavities. Initially, we suspected the diamond could be a composite as mentioned before (Fritsch, 2000).

As the diamonds were opaque and the only way to observe the diamond was through microscope using reflected light. Our observation showed that the surface of the diamonds had many irregular cavities (Fig.2). Also the jewellery manufacturer had drilled holes into the diamond and studded them with small round gold pins giving a rather attractive appearance. Actually the gold pins distracted the observation of the cavities. The quality of polish is good, the facets are in shallow angles, and some of the polish overlapped onto the gold pins (Fig.2). Natural black diamond is made up of naats and polishing it is very difficult. In this case, this pair of earring does not show polishing marks.

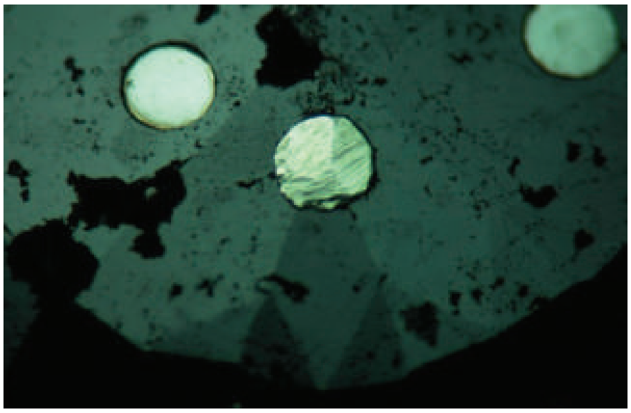


Fig.2. Many cavities and good quality of polishing with some polishing of diamond overlap onto the gold pin (15x).

Under higher magnification at 20x, the fissures on the diamonds showed black carbon like filling probably graphite. At the back of the earring, there were parallel groove like marks running across the surface and the grooves seemed to appear to fuse together along the centre creating a small ridge like appearance (Fig.3). No explanation could be given as this was our first time to observe such unusual markings. Even the side of the earring are drilled holes at interval (Fig.4).

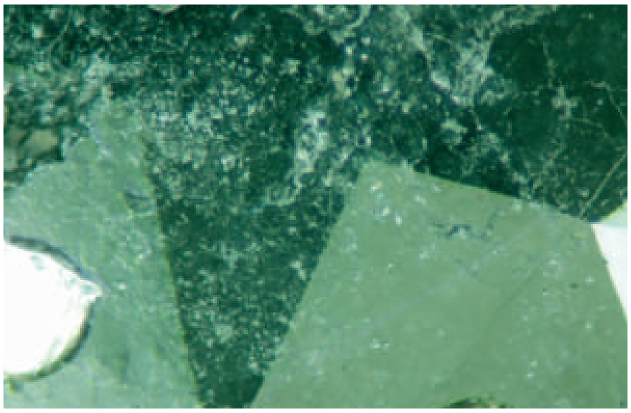


Fig.3. Fissures show black carbon like staining probably graphite (20x under reflected lighting).

On the rear side of the earring, there were parallel groove marks running across the surface and the grooves seemed to fuse together along the centre creating a small ridge like appearance (Fig.3). No explanation could be given as this is our first time to observe such unusual markings.

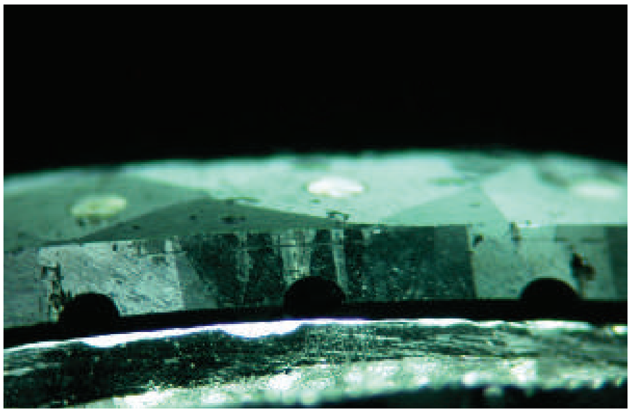


Fig.4. Drilled from the sides of the diamond earring (10x).

The diamond earring was sent to the Singapore Government Material Science Laboratory for further testing. EDAX was performed on one of the samples the result was pure carbon 97.37 wt %, O 2.41 wt %, and Si 0.22 wt%. Further test using Raman spectroscopy was performed at the Gems & Jewelry Institute of Thailand (GIT) and found that there was graphite in the cavities.

Discussion:

Through discussions with various senior gemologists around the world, it was concluded that this pair of black diamond had been heat treated from poor quality rough diamond material or boart. Heating under vacuum or under low pressure turns the diamond black along the fissures and cavities. As a result, the graphitisation of this treated black diamond sometimes gives metallic beep on diamond tester. This form of treatment became popular since 2008 and carried out in Surat (per comm. Dr Panjikar, 2014).

Acknowledgements:

For many discussions through emails and for their being kind to share references, my sincere thanks to Dr Jayshree Panjikar, Prof. Henry Hanni, Ms Wilawan Atichat and Thanong Leelawatanasuk of GIT, Prof. Emmanuel Fritsch, Dr Hiroshi Kitawaki, Jean-Pierre Chalain of SSEF, Brendan Laurs and Masaki Furuya.

References:

Fritsch Emmanuel: 2000 “Black diamond imitation” Gems & Gemology, Summer pg171-173

Roskin Gary: 2008 “Treatments and Lab-Grown Diamonds” JCK, Nov. issue

Hall Matt & Moses Thomas: 2001 Gem Trade Lab Notes “Heat-Treated Black diamond: before and after” Gems & Gemology, Fall pg 215

Schweizer Lucas: “Herstellung und Handel in Surat” A German & French Diamond Magazine

10 carat Asteriated Diamond

By George Spyromilios CDG

"The flower of life" is a 10.00 ct blocked octahedral shape natural diamond displaying a geometrical figure composed of six petal like parts (fig. 1.). Each of the six petals originates from the centre of the crystal and it is oriented in the direction of the six corners of the crystal. This kind of diamond is called in published literature "Asteriated diamond" and pattern is due to concurrent cuboid and octahedral growth (see reference). Six darker slices coming from the centre are six cuboid sectors when viewed perpendicular to the slice. Visible spectra of this diamond displayed 415 and 478 nm absorption lines, typical for "Cape" type la natural diamond, what is confirmed with typical pattern under Cross Polarized Filters (CPF). HPHT grown diamonds also have cubo-octahedral growth, but different morphology, spectra and absence of pattern under CPF.

Fig 1 Ten carat Asteriated Diamond

Each petal is composed of numerous blackish tiny pinpoints. These inclusions are typical for hydrogen rich natural diamonds.

The color of the diamond is light yellow - green and it shows no reaction under short wave and long wave ultraviolet radiation.

Lab Alert #1

Octahedral crystals with adamantine lustre and triangular markings

By Jayshree Panjikar

Fig 1 Octahedral shaped crystals of topaz coated with nano diamond coating

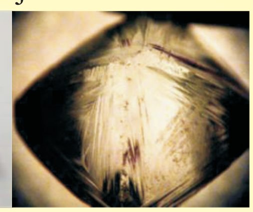


Fig 2 Triangular Surface Markings

Transparent, sparkling white, crystal clear, octahedral crystals above 2carats each with perfect triangular surface marking (as seen on some Russian roughs) were submitted for testing...crystals had SG=3.52, with normal Presidium diamond tester indicated diamond, Hardness 9+(gets scratched with diamond)! Two had crystalline inclusions! Looked perfect diamonds!

When FTIR did not give usual diamond peaks then they were sent for Raman Spectra which indicated Topaz coated with diamond slurry! On immersion in methylene iodide one of them showed 2phase inclusions.

These fraudulent crystals were bought as natural diamonds from Angola. A syndicate is working behind this fraud of cutting topaz crystals into grooved octahedral shaped crystals and coating them with nano-diamond slurry

– Pangem Testing Laboratory, Pune India

Reference :

Three historical 'asteriated' hydrogen-rich diamonds : growth history and sector - dependent impurity incorporation (2004) : Benjamin Rondeau, Emmanuel Fritsch, Michel Guirauda, Jean-Pierre Chalain and Franck Notari, Diamond and Related Materials, 1658 - 1673.

-Independent Gemological Laboratory, Greece

Introducing Founding Member 005:

Masaki Furuya

Year of Establishment: 1998 Full Name of the Owner of the Laboratory: Masaki Furuya

Gemmological Qualifications of the Owner / Founder: European Gemmologist, FEED (D.Gem.G), GIA.G.G. C.G.J.

Address: 824-5 Furukamijo Kofu Yamanashi 400-0051 Country: Japan
Telephone +81-55-243-2690 /Fax +81-55-243-8377
Email : jggl@sapphire.co.jp
Website : http://www.sapphire.co.jp

Owner's Experience in the Trade: Coloured gemstone and diamond identification, Diamond grading, Pearl identification

What Standard Gem Testing Equipments do You have: All standard equipment with some modification like LED or HID light source

What Advanced Instruments do you have: FT-IR, UV-Vis-NIR, Raman, Photoluminescence spectrometer, Cathode luminescence and EDXRF

Have you published or presented papers at conferences/magazines/seminars?
Our own journal of "Gem Information" in Japanese, and at IGC, GIT conferences

Are you a Member of a Gem Trade Organization?
Of the Association of Gemmological Laboratory of Japan (AGL)

Are you giving lectures and educational programs to trade?
Yes also on TV shows.

Why did you decide to found ICGL?
Japanese jewellery market is isolated especially for nomenclature of pearls. ICGL will help to provide international standard certification.