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HPHT Grown Near-Octahedral Shape Synthetic Small Diamonds using Even Temperature Method

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Today it is possible to grow octahedral shaped synthetic diamond by HPHT process (Fig 1). Maintaining certain growth conditions it is feasible to grow near-colourless lab diamonds having not only morphological features like perfect octahedron but also other "natural" shapes. The fact that rough lab grown diamonds with morphology previously unique to natural diamonds are now going to be seen in the market, it is important to have knowledge about these synthetically grown octahedral shaped HPHT diamonds as it would be difficult for less experienced rough traders to make a visual on the spot identification of lab grown rough diamonds.

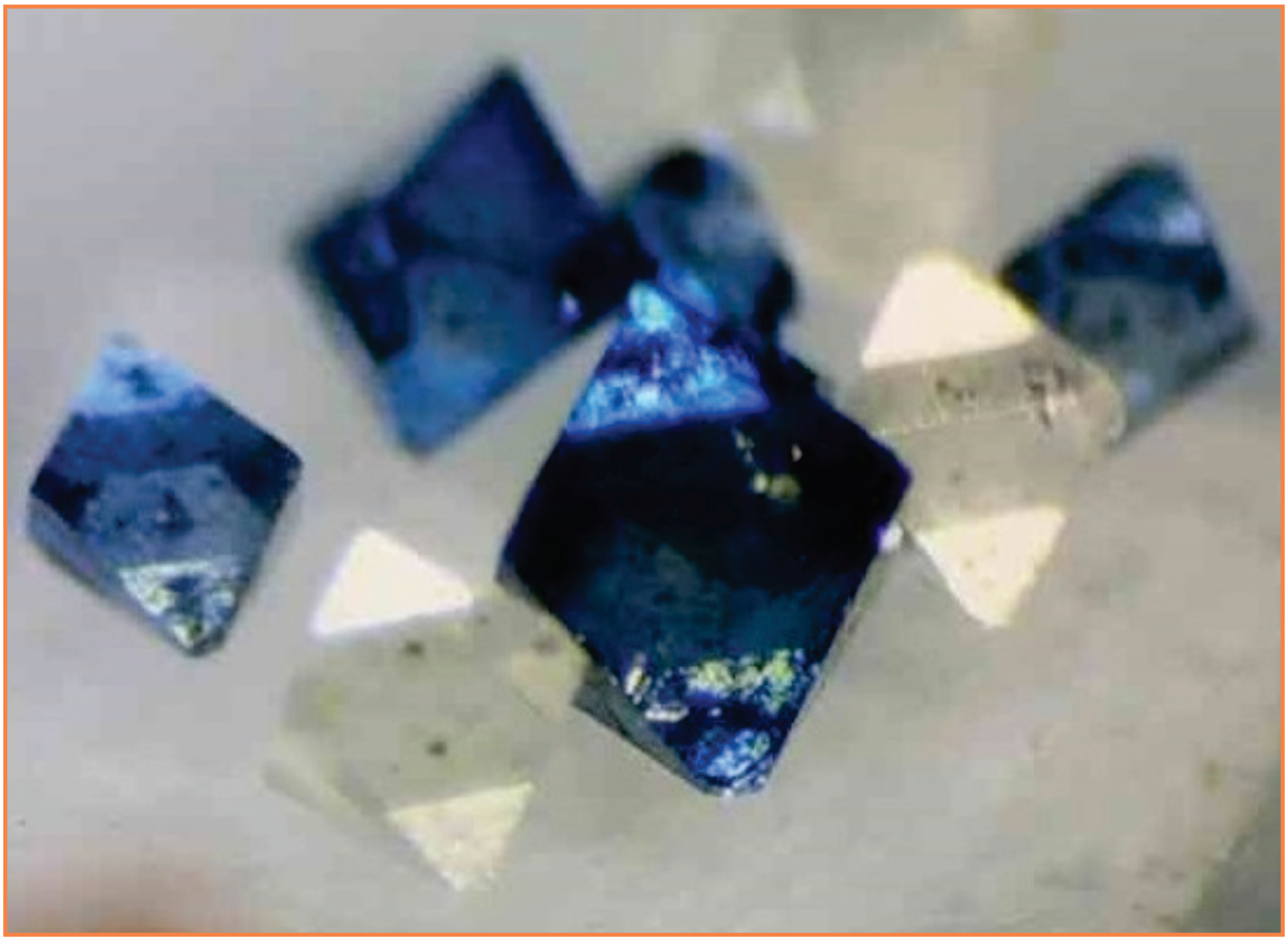

Fig 1: Perfect octahedral shaped synthetic diamond by the HPHT process

HPHT PROCESS :

High pressure high temperature (HPHT) diamonds are grown in two ways: one method in which the seed is maintained under gradient temperature and in the other method is where there is steady even temperature. Even temperature means the temperature in every single cell in the entire growth period as well as the whole cell the temperature is stable. It is maintained steadily even, between 1300degree Celsius and 1400 degree Celsius. The range of pressure is about 35000 to 40000 atmospheres. The gradient method requires a seed for growth where as the latter method can grow without seed but seed would expedite the crystallization. Earlier differentiating natural rough diamonds from the lab grown rough diamonds was done on the basis of their typical cubo-octahedral shape seen with diamonds produced by HPHT (High Pressure High Temperature) and the characteristic plate structure of diamonds produced by CVD (Chemical Vapour Deposition) which significantly differs from the morphology of natural diamonds. Now that perfect octahedral crystals can be created by HPHT it is important to know how they are formed.

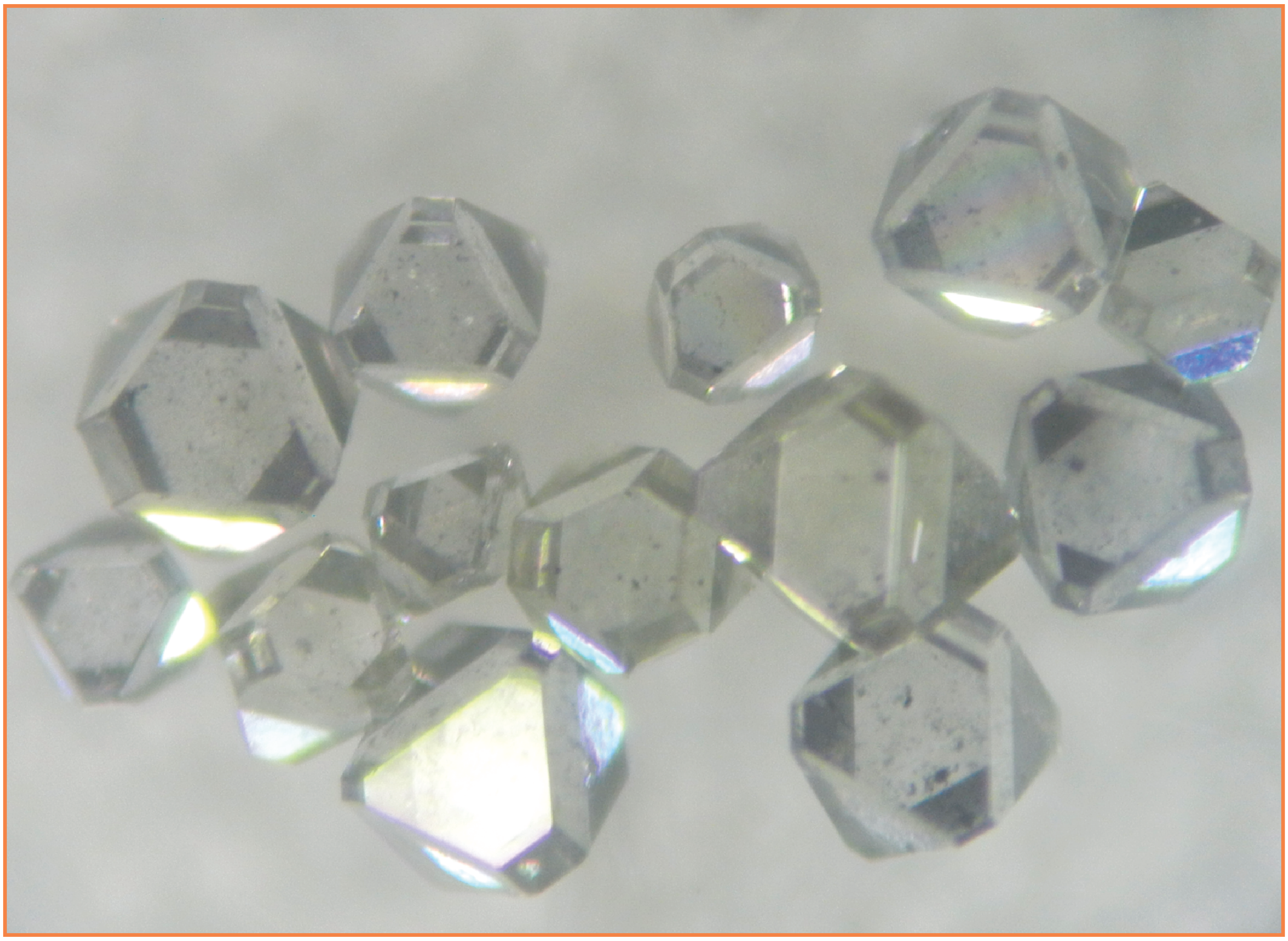

Fig 2: Cube-octahedral crystals of synthetic diamonds

Growth Process :

This even-temperature method is similar to the kind of traditional HPHT process which is used to grow the synthetic yellow diamond sand popularly known as industrial diamond grit. The energy required for growth is obtained by adding of pressure during the whole period of growth. The temperature is all steady or even during the whole period of growth. The cube/octahedral shaped small diamonds (Fig 2) can nucleate automatically. If higher temperature like 1400degree Celsius is maintained in the cell one can get more octahedral shaped crystals. If low temperature around 1300degree Celsius is maintained in the cell one can gets more cube shaped synthetic diamond crystals. Taidiam Technology has managed to grow different shapes from cube to octahedral. The Cubic Press gives pressure from 6 directions it has been developed in China. In China there are more than 7000 cubic presses, originally used for industrial diamonds.

As there is a limitation for the total pressure that can be added, so there is a limitation for size of diamonds that are grown. Normally, the size of the biggest synthetic octahedral yellow color diamond is 4mm and for the colorless is 1.5mm. This method is much more difficult than seed- gradient temperature method used to grow diamonds larger, whiter and cleaner. We at Taidiam Technology are the only research lab and factory in the world to do research and grow synthetic colourless diamond by this method. Time required is about 2 days to grow 4mm yellow color seed-gradient temperature method. It takes about 24 hours to grow 1-2 mm seed-gradient temperature colorless and around 12 hours to grow 1-1.5mm colourless octahedral diamonds by using even temperature method.

This even temperature method does not require a seed crystal to grow, all crystals are spontaneous nucleation. However a seed crystal could speed-up the process also this method can produce better shape and more carat weight from same size cell.

Clarity:

At Taidiam Technology the synthetic diamonds have clarity range SI to Pique. Mostly the inclusions are black carbide, black catalytic metal and cloud. The metals are iron and nickel and therefore these synthetic diamonds can be attracted by a magnet. When a strong magnet is moved on the rough or polished stones, these diamonds will be attracted towards it.

Seed-gradient temperature method grown small rough diamonds :

As of date HPHT seed-gradient temperature grown small rough diamonds are produced around 10,000 carats every day in China. The quantity will be increased very gradually. One can use the same strong magnet to attract and the diamonds can get attached to it, almost 85% will be attached. Mounted small HPHT diamonds are very difficult to detect. The cost of polishing is same as natural small diamonds, the cost for growth is quite high, so the polished small synthetic diamonds would be sold 20-30% lower than the similar quality small natural diamonds.

415nm N3 color center :

98% of natural diamonds are type Ia, they have 415nm N3 absorption peak under UV-Vis spectrum. All these synthetic diamonds grown in China are type IIa or Type Ib, they do not show the 415nm peak.

Comparison of synthetic and natural small diamonds :

The comparison of synthetic diamonds from different growth methods and natural diamonds is shown in the following table.

Comparison of Synthetic & Natural Colorless/Near Colorless Small Diamonds

Growing Method	HPHT Seed - Gradient Temp.	HPHT Even Temp.	CVD	Natural
Type	IIa/Ib	IIa/Ib	IIa/Ib	Ia & others
Crystal Shape	Tower Shape, Near Ball	Near Octahedral	Cut Corners from Cube Crystal	Octahedral, Dodecahedral
Color	Colorless/Near Colorless	Colorless/Near Colorless	Colorless/Near Colorless	Colorless/Near Colorless
Clarity	VS-P	SI-P	VVS-SI	VVS-P
Inclusion	Black catalytic metal, clouds	Black Carbide, Black catalytic metal, clouds	Polycrystalline Diamonds, Graphite, Carbon	Different Natural Inclusions
Strong Magnet Attachment	85%	20%	0%	0.01%
N_3 (415nm) Color Center	None	None	None	98% Yes

Conclusion:

The HPHT even temperature method for growing small colorless synthetic diamonds can have better shapes than those crystallized by other methods. The recovery after faceting is about 40% about 10% higher than that for diamonds by seed-gradient temperature method and will be suitable to the gem diamond market.

Treated amber beads

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An amber bracelet was brought into our gem lab for testing about four months ago. Our client wanted to find out whether the amber had been heat treated or not. Generally, nearly all amber from Baltic region is heat treated including from other countries like Dominique Republic. One bead was taken out from the bracelet for testing as our client claimed that the bracelet had cost him some US$13,000 and he requested us not to destroy the sample (Fig.1). General observation was conducted on the bracelet and one bead weighing 25.00 carats and measuring 20.99mm in diameter was removed from the bracelet for further analysis using infrared spectrometer.

Gemmological testing and infrared spectrometric analysis were performed and results are reported here. The colour of the amber ranged from pale yellow to light greenish-yellow with tint of brown. Under the microscope with 10x magnification, majority of the beads were quite clean except for some flow mark, no gas bubbles were observed but minute brownish debris was found (Fig.2). Spot reading of R.I. was determined as 1.53 and under polariscope the bead showed anomalous double reflection (ADR) effect and under ultraviolet light there was a strong chalky blue fluorescence under long wave and weak chalky blue under short wave. The ADR effect of the amber indicated that the amber was not composite material but one whole piece and the continuous flow mark throughout the bead confirmed it (Fig.3).

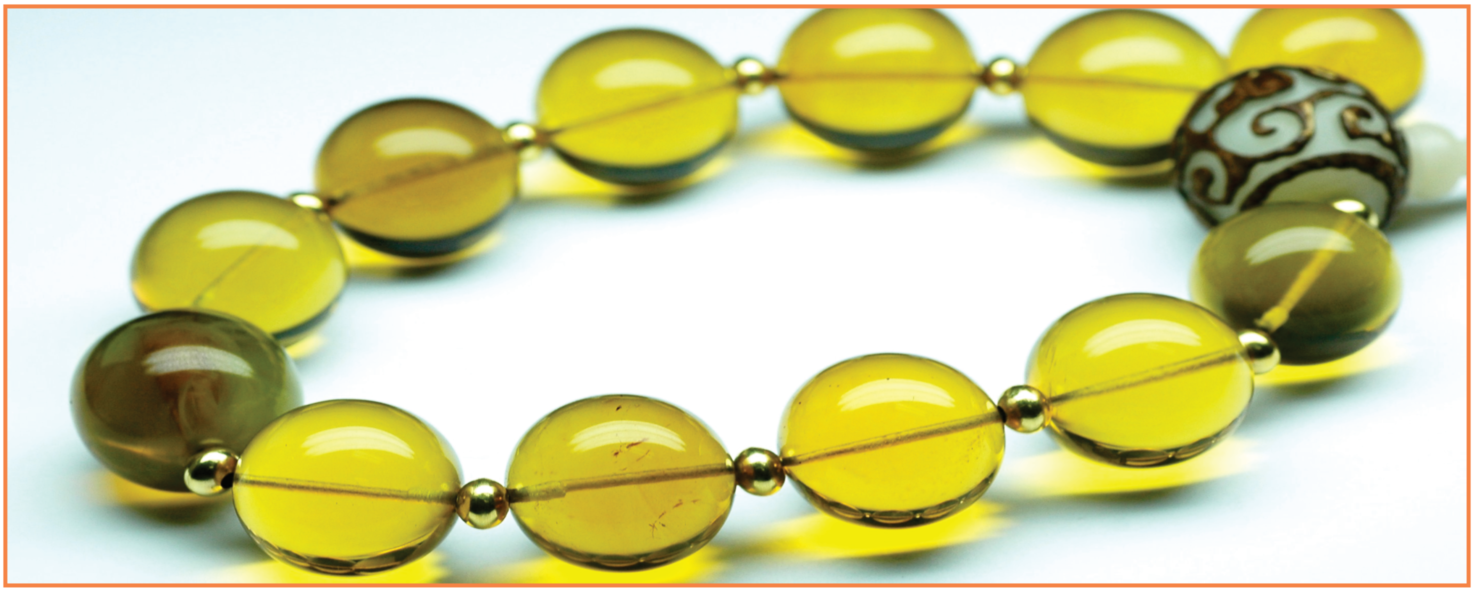

Fig.1 Amber prayer bead bracelet with an agate bead as a head, no inclusions observed in amber (Photo by Tay).

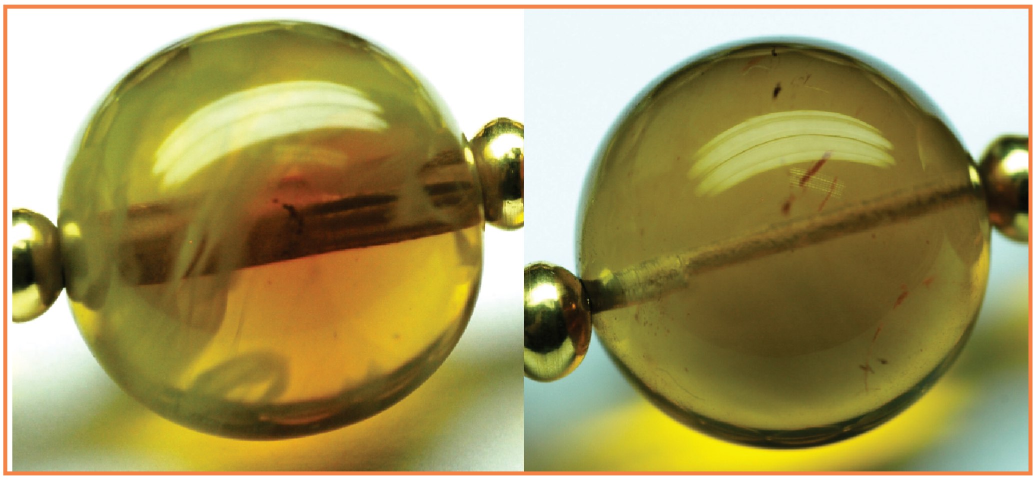

Fig.2. Bead on the left showing flow marks and bead on the right has dark brownish debris-like inclusions (Photo by Tay).

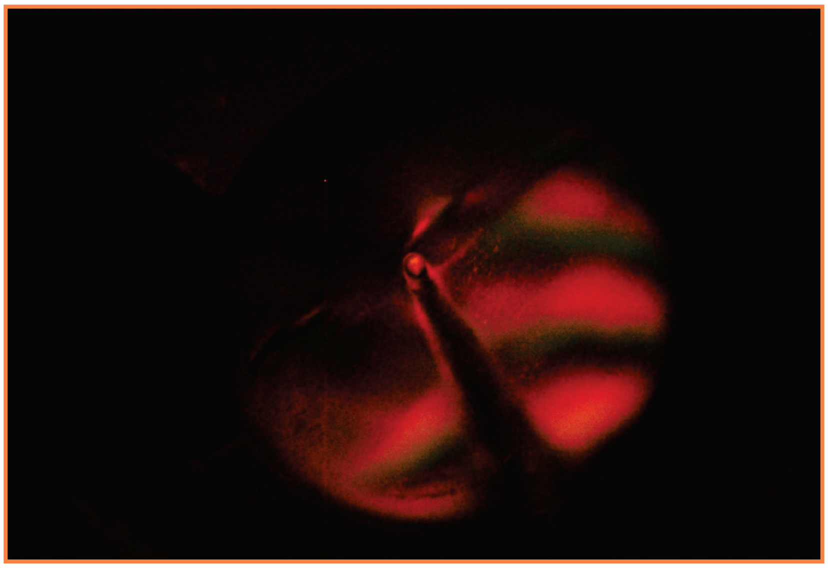

Fig.3. Under polariscope, the ADR effect of amber bead shows the flow mark throughout the bead indicating it to be a single piece of amber and not a composite (Photo by Tay).

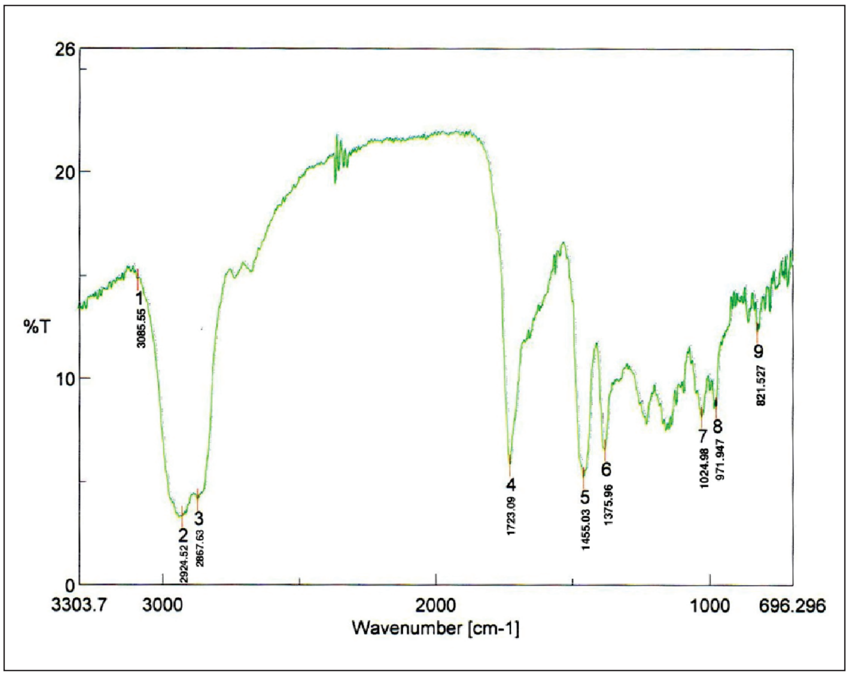

Fig.4. FTIR result indicates that this material is indeed amber and not copal resin and that the presence of 821 cm^{-1} indicates heat treatment (spectrum taken by Tay).

Heat treated amber may show sun spangle effect but in this sample, no sun spangles were found. Instead infrared spectra were used to assist in this analysis. Some amber powder from the drill holes was taken out for analysis. Initial suspect that the amber could be copal because of the size of the bead but the infrared spectrum show no peak at 3076, 1643, 1593 but and 887cm^{-1} to indicate copal. The peak 2924, 2867-2853cm^{-1}, and at 1455, 1384, and 1024-975cm^{-1} indicated that it was indeed amber (Fig.4). As for heat treatment, the peak 821cm^{-1} was found (Abduriyim et al, 2009).

In conclusion, based on one amber bead result, the bracelet is most likely made of amber beads and that it has undergone heat treatment and most probably using autoclave under controlled environment.

Reference:

Ahmadjan Abduriyim, Hideaki Kimura, Yukihiro Yokoyama, Hiroyuki Nakzono, Masao Wakatsuki, Tadashi Shimizu, Masataka Tansho, and Shinobu Ohki “Characterization of 'Green Amber' with infrared and nuclear magnetic resonance spectroscopy”, Gems & Gemology, Fall 2009, Vol.XLV, pg 158-177.

Amber Collection

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After having seen the spectacular collection of amber in Lithuania during the IGC 2015 our initial interest and fascination for amber has grown multifold. Added to that some of the inclusions observed in the Baltic Amber are so amazing that the fact that these inclusions makes the age of the amber around 50 to 60 million years makes the amber even more breathtakingly attractive.

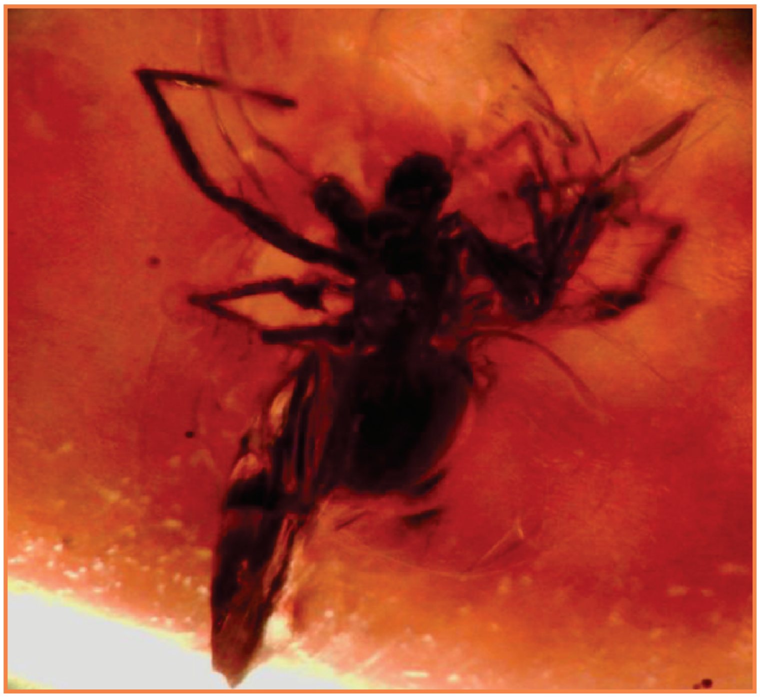

Fig 1: Death dance of the insect trapped in the Baltic amber some 50million years ago

We have now in our collection colorful varieties of amber, some with insect inclusions and some with gas bubbles and remnants of flora and fauna of ancient time. (Fig 2 to Fig 5).

Fig 2 Red Amber

Fig 3 Burmese Amber

Fig 4 Vietnamese Green Amber

Fig 5 Sumatra Blue Amber

The Baltic amber pieces have some mind blowing inclusions like the one shown in Fig 1. One can actually see the death dance of an insect as it got itself trapped in the flowing resin which later fossilized. Another inclusion in the Baltic amber has perfectly preserved wings of an insect. The fine venation of the wings can be seen clearly along with other appendages.(Fig 6). The fur like growth on the body is also visible of an insect trapped some 50million years ago in the amber (Fig.7, 8 and 9).

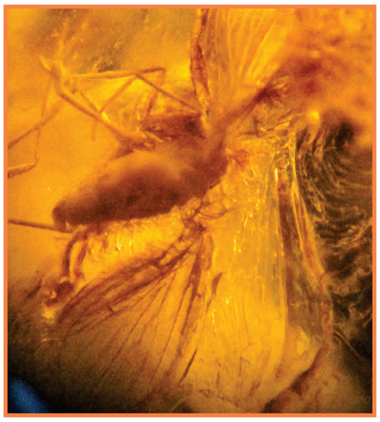

Fig 6: Well preserved winged insect trapped in Baltic amber

Fig 7 Baltic Amber

Fig8 Baltic Amber

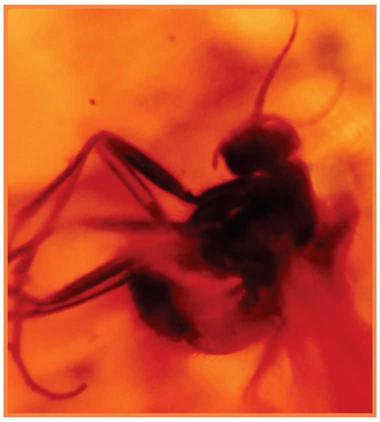

Fig 9: Some insect similar to Fungus Gnat trapped in Baltic amber

AG&J Trade Alert: Proposed Solution to Detecting Fraudulent Replicas of Certified Diamonds

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Recent advances in growing and treating diamonds has garnered much public attention, yet relatively little has been published on the subject. AG&J has witnessed large and small companies pursing investments in HPHT / APHT / LPHT treatments, irradiation and especially technology for growing HPHT and CVD synthetic diamonds. Many companies are already in place and ready to capitalize on these new techniques, and it is only the complexity and the sophistication of the technology that slows new investment. This text will deal with the problem of identification and certification of diamonds when synthetic diamonds, larger then 2ct, become common enough to sufficiently meet the needs of the market. We will address the potential risks certifying laboratories run in loosing consumer confidence, as well as the ultimate question; how can consumers and jewelers trust that mounted diamonds have been identified and certified? Based on the data contained within almost all diamond certificates, it is relatively easy - especially with round diamonds - to manufacture identical replicas of certified diamonds. These replica diamonds can be made from either HPHT treated or synthetic diamonds. The fraudulent replica will then replace the original diamond and will be sold with its authentic certificate. This process is done primarily with type IIa and IIb diamonds, with a high color- H + and primarily with high clarity - VVS +. This problem was predicted and spotted many years ago. Replicas were made using type IIa HPHT treated diamonds, but the process has evolved to include irradiation and heating as a “post treatments”. These new processes increase the likelihood that laboratories will be forced to bear the responsibility for this fraud. For example, in the case that a replica diamond with a certificate is sent to a laboratory and is issued a new certification (describing the diamond as treated or undetermined - GIA comments:*whether the color of this diamond is of natural origin or result of an artificial process cannot currently be determined) the fault will inevitably fall on the shoulders of the original certifying lab. This is one way in which these replicas threaten the integrity of our industry.

The exact number of cases in which this has occurred is difficult to know, but is most likely limited because replicas are made from natural type IIa diamonds. Until today these cases were only rumored about in closed professional circles, and estimates of the number of replicas vary greatly. Surely, though, the answer to this question resides within the Gem Labs databases. With an ever-increasing supply of synthetic diamonds hitting the market, it is not difficult to conclude that synthetic diamonds will be used as fraudulent replicas for certified diamonds. The problem is particularly serious in mounted diamonds and jewelry. AG&J has offered technical solutions for the screening and identification of mounted diamonds to many large jewelry manufacturers, but within the market, this very real threat to diamond confidence is not yet recognized as a problem worth addressing. Unfortunately, the most effective solution to this problem is both complicated and expensive. We call it “Diamond Fingerprinting.” It includes detailed spectroscopy/imaging in all areas. Unfortunately, this process is prohibitively expensive for most labs and requires a large investment of both time and money. AG&J proposes the following solution, which applies to both mounted and un-mounted diamonds: All diamond certificates should be expanded to include two graphs - IR absorption and 365 or 405 PL induced bulk photoluminescence, with the added obligation of storing these graphs in a database. FTIR instruments are already well known to the industry and owned by most laboratories. 405 Luminescence is a simple, inexpensive system and is described as an "express-spectrometer" in our literature (pg.6 of Simic / Zaitsev, "Characterization of diamonds color-enhanced by Suncrest Diamonds USA"). These two procedures could then be standardized into one simple system. Since mounted diamonds require different adjustments and customized instruments, new tools to accomplish this procedure would be the only new investment required. In our opinion, the essential principle of using at least two methods of identification would then be satisfied. Also the implementation of these methods would be relatively easy and inexpensive for small and large laboratories alike. Both methods have no limitations in size, color or shape of diamond. Based our experience, the combination of these two methods could easily determine the authenticity of more than 95% of all diamonds inspected.

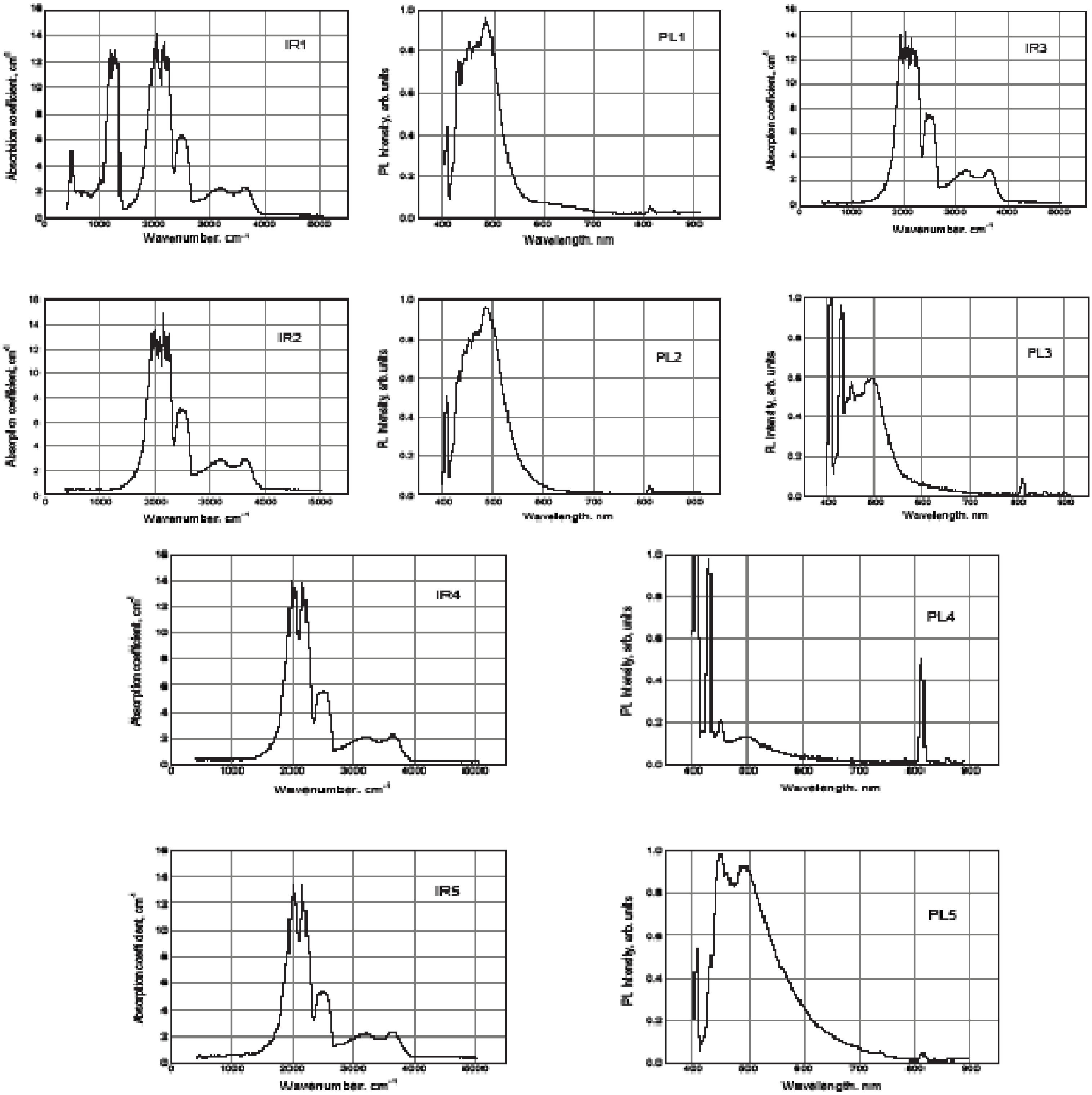

Figure 1.
IR1/PL1 - Natural Diamond/Natural Color – Type Ia
IR2/PL2 - Natural Diamond/Natural Color – Type IIa
IR3/PL3 - Natural Diamond/HPHT treatment – Type IIa
IR4/PL4 - Synthetic Diamond as grown/ HPHT process – Type IIa
IR5/PL5 - Synthetic Diamond HPHT treated/ CVD process – Type IIa