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Irradiation May Cause Synthetic Melee Diamonds to behave like natural diamonds

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INTRODUCTION

Melee and Star sized diamonds are used in large quantities in jewellery. When such small diamonds are studded in jewellery it is not economical to carry out the necessary tests to determine whether the diamonds are natural or synthetic. HPHT grown and CVD grown synthetic diamonds and irradiated HPHT grown and CVD grown synthetic diamonds have been investigated using Diamond Spotter, Gemologis®, DiamondSure, FTIR and UV-Vis spectroscopy, and standard gemmological microscopes. When observed under the Diamond Spotter which detects Type IIa1, diamonds gave varied results. Infrared spectroscopy indicated Type Ib for the irradiated synthetic diamonds. The synthetic diamond detector Gemologis® which detects small sized the Type IIa diamonds indicated the treated diamond as natural Earth mined diamond. It was noticed that the irradiated CVD grown synthetic diamonds give natural diamond reading in some cases. Majority of these diamonds show strong greenish yellow to chalky-yellow or yellowish green fluorescence under long-wave ultra-violet light.

INVESTIGATIONS

Twenty samples of faceted known synthetic diamonds and known synthetic irradiated diamonds ranging from 0.02 to 0.07carat were studied. Among these there were 5 samples of HPHT synthetic diamonds, 5 samples CVD Synthetic diamonds, 5 samples Irradiated HPHT synthetic diamonds and 5 Irradiated CVD diamonds. These diamonds were investigated using DiamondSure®, Diamond Spotter, Gemologis®, FTIR and UV-Vis spectroscopy, and standard gemmological microscopes.

RESULTS

It was found that under the DiamondSure all diamonds were under the category "Refer", but under the Diamond Spotter varied and confusing results were obtained some were Type II and some were not. No characteristic inclusions were observed under the microscope.

SPECTROSCOPY

UV-Vis-NIR spectra were recorded in the range 200nmto 900nm at room temperature for all the diamonds. Some showed the broad band at 270nm and further small peaks at 340nm, 370nm, 554nm, 612nm and 769nm absorption peaks in the form of a gradual rise towards the shorter wavelength across the NIR, Visible to the Ultraviolet region2, 3.

FTIR spectra confirmed that all the faceted as grown HPHT and CVD diamonds were Type IIa, but irradiated CVD diamonds were showing Type Ib peaks whereas the irradiated HPHT grown diamonds were showing the Type IIa peaks.

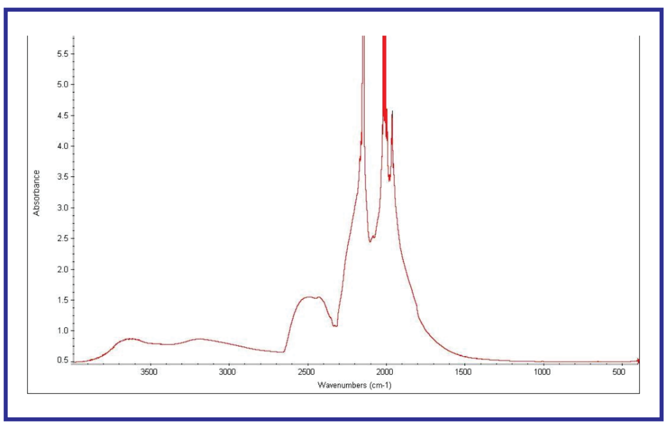

Fig 1 : FTIR spectrum of as Grown HPHT/CVD diamonds

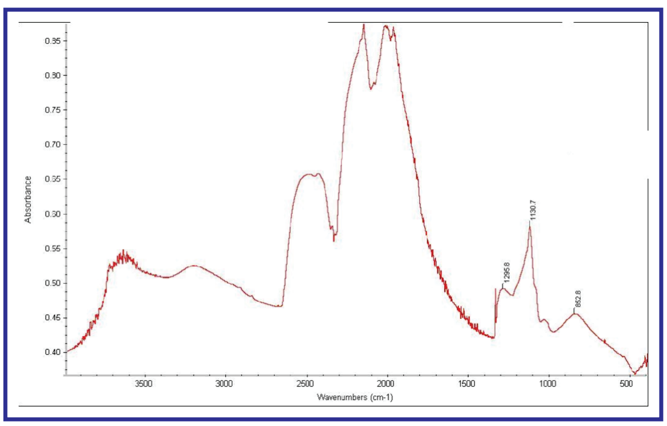

Fig 2 : FTIR spectrum of known irradiated CVD diamond

GEMOLOGIS®

The melee sized diamonds are easy to test with the instrument Gemologis® (Fig 3) as it has double sensors it can give a fair idea whether diamond is TypeIIa. Most of the diamonds if they are natural Type I, a green colour light glows indicating the diamond to be Earth mined. If the diamond being tested is synthetic then a yellow orange light glows indicating the diamond to be synthetic of HPHT/CVD origin. The diamond samples in this present study when tested with Gemologis®, it was found that the as grown HPHT and CVD diamond were clearly being determined as HPHT /CVD with the yellow orange glowing light (Fig 4). When the known irradiated synthetic CVD diamonds were tested the instrument gave a green colour light indicating "Earth Mined" the instrument determined irradiated synthetic diamonds as natural diamonds (Fig 5).

Fig 3 : Gemologis Instrument

Fig 4 : Yellow Orange glow seen for as grown HPHT and CVD

The irradiated HPHT grown synthetic diamonds clearly showed the yellow orange glow indicating their true nature as synthetic diamonds.

Fig 5 : Green glow seen for irradiated Synthetic CVD

Fig 6 : Yellow orange glow for irradiated Synthetic HPHT diamond

CONCLUSION

Changes in the FTIR spectra indicate that the irradiation process does have a substantial impact on the synthetic CVD diamonds changing them from Type IIa to a pseudo Type Ib, and thereby the instrument Gemologis® gives the result as "Earth mined" with a green glow. There is not much impact on the HPHT grown synthetic diamonds as they retain their Type IIa qualities and hence the Gemologis® indicates them as synthetic diamonds. Long exposure to SWUV light also has an effect on the body colour. Such type of erratic results would be major problem for diamonds that are set in jewellery. Further investigations on these diamonds are being carried out.

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LAB NOTE:

Fracture-filled diamonds an example from the daily work in gem-testing laboratory

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Fracture-filled diamonds are encountered on the market for more than 20 years.The name is used for describing diamonds with distinct fractures that get filled with a transparent material, usually glass with a high refractive index Fig. 1. The treatment will make the fractures disappear from view or will at least diminish their visibility but it will not make them disappear.

Fracture fillings can be recognized by pink, purple or purplish-blue flashed that are caused by the difference in dispersion between diamond and filling material. Fig. 2 shows this. Flashes can be seen most easily when the diamond is viewed under magnification from the pavilion side, they may be more difficult to distinguish with stones set in jewellery. An experienced viewer may trace them even with the naked eye. The treatment has to be declared and clarity grading has to take the actual number and size of fractures into consideration.

Fig.1: When viewing from above,fractures are noticed as thin lines on the lower rim only and pink flashes are hardly noticeable. Gemolite, 10x. Photograph: E.Strack

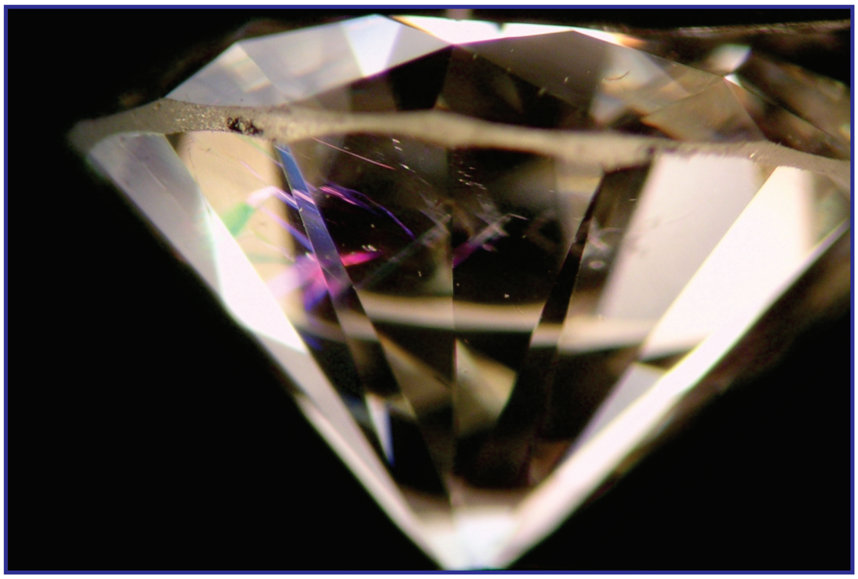

Fig. 2: When viewing the same diamond from the pavilion, several distinct Fractures that cross each other can be seen and pink and purplish-blue flashes are easily noticeable.. Gemolite, 10x. Photograph: E. Strack

Nano-coated Pink Diamond

A Study on Surface Analysis Technology and Coating Materials

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Coating is one of the oldest methods to enhance the color of a diamond, but combined with the latest technologies it has advanced to the technological level being completely different from the previous levels. The issue is that the

advanced technology may weaken the boundary between

Figure 1. Ice Pink Diamond (0.68 ct) of Lotus Colors Inc.

Two pieces of Ice Pink Diamonds (0.68 ct and 0.18 ct each)

Figure 1.Ice Pink Diamond (0.68 ct) of Lotus Colors Inc.

were purchased from Lotus Colors Inc., and used in this study. Natural pink diamonds are one of the rarest and most expensive of all gemstones. Thus, there have been many attempts to artificially produce pink color diamond, including irradiation, HPHT synthesis or treatment, and coating. Ice Pink Diamond realized a clear light pink color by coating a thin film on the pavilion surface [1].

To identify the types of the coated diamond samples, FTIR (Bruker, LUMOS) measurements were natural and treated diamonds, lowering the consumer trust in the natural diamond market. From this point of view,

analysis methods and information on the coating material of a coated pink diamond product are addressed. carried out at room temperature. Both samples showed the type IaAB spectra typically observed in the FTIR measurements. The peak related to hydrogen was also detected at 3107 cm-1.

Figure 2 shows the fluorescence images (De Beers, DiamondViewTM) of the 0.68 ct sample, which display a typical growth pattern of a natural diamond. The measurement results of FTIR and DiamondViewTM show that the diamond used for coating is a type IaAB natural diamond.

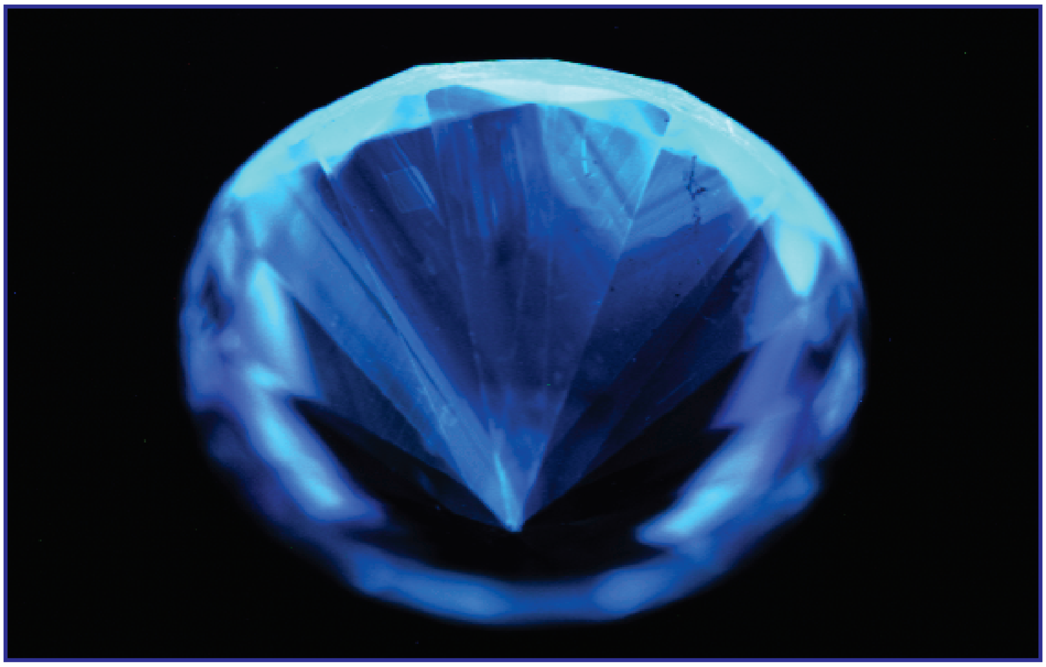

Figure 2. The fluorescent images of an Ice Pink Diamond (0.68 ct)

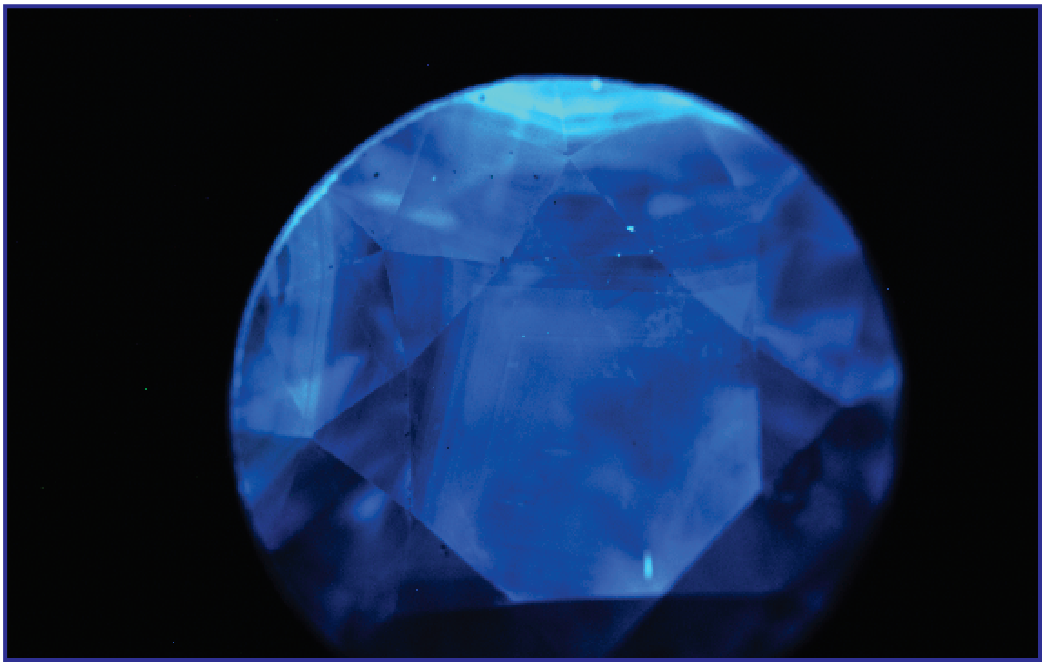

Figure 2. The fluorescent images of an Ice Pink Diamond (0.68 ct)

Spectroscopic characteristics of natural pink diamonds are a broad absorption band centered at around 550 nm, which is occasionally accompanied by a broad absorption band at 390nm [2]. The broad absorption band at 550 nm is caused by plastic deformation and is also called "Pink Band" [3]. Coated pink diamond is reported to have a broad band centered at around 525nm [2]. For the comparison of the spectroscopic characteristics among natural pink, coated pink, and Ice Pink (light pink color) diamonds, the UV-Vis (JASCO V-670) spectra of the Ice Pink Diamond samples were measured. Figure 3 shows the absorption spectra of the Ice Pink samples in the visible range. The absorption spectra indicate two defect centers, N3/N2. A broad absorption band was observed at around 533 nm. The absorption peak of the light pink-coated Ice Pink Diamond appears in the middle of the absorption peaks of natural pink diamond and coated pink diamond. This absorption shift could be affecting the color so that Ice Pink Diamond has a clear light pink color.

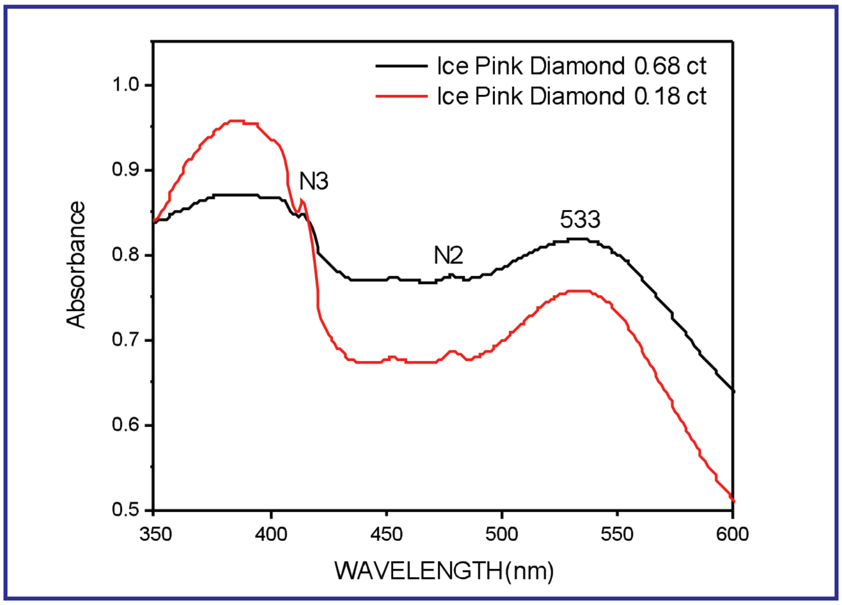

Figure 3. The absorption spectra of Ice Pink Diamond samples in the visible range

EDXRF(EDX-8000, Shimadzu), LIBS(Applied Spectra, J200), and Secondary Ion Mass Spectrometry(TOF.SIMS 5, Münster, Germany) were used for the chemical analysis of the coating layer of Ice Pink Diamond samples. For the semi-quantitative chemical analysis using EDXRF, the table and pavilion surfaces of the samples were measured. No element was detected from the table surface. However, Si(93.5%) and Au(6.5%) were detected from the pavilion surface of the 0.68 ct sample. The result similar to that of the 0.68 ct sample was also obtained from the 0.18 ct Ice Pink Diamond. The result of EDXRF is consistent with the information from the manufacturer (Lotus Colors Inc.) that the coating of Ice Pink Diamond is applied only on the pavilion surface. Si, which constitutes most of the coating layer, improves the durability of the coating. The doped gold in coating layer absorbs the light from the specific area in the visible range in order to create the pink color [4].

The LIBS measurement with laser ablation revealed the presence of Ti, Na, and K in addition to Si and Au from the pavilion surface. LIBS showed much better detection limit compared with EDXRF. Depth profiling was attempted to acquire the concentration of the elements in the coating layer from the surface to the depth direction. However, the depth profiling was unsuccessful, since the coating layer was thinner than the sputtering depth of one shot of the laser. From the LIBS measurement, overall chemical composition of the coating layer was gained, but not depth profile.

Figure 4 shows the depth profiles of 0.68 ct IcePink Diamond sample measured by SIMS. Two different positions on the pavilion surface were selected based on the EDXRF results. The SIMS depth profile was measured on negative and positive ion data collection modes. The element concentrations (counts) are displayed along the depth direction (nm). The result of SIMS shows that the coating layer of the Ice Pink Diamond sample is ~85nm thick in total and has a multi-layered structure. Si and O with high concentrations indicate that the major ingredient of the coating layer is SiO2. Moreover, other metals or metallic oxides constitute other coating layers. Figures 4 (b) and 5 (b) reveal that 5~10 nm ultra-thin layer exists on the diamond surface. Titanium, which constitutes the layer, is used for increasing the adhesion between SiO2 and the diamond [5]. The gold layer shown in Figures 4 (a) and 5 (a) is 30~40 nm thick and has a clear boundary. The structure of the coated film shows four layers consisting of [SiO2]-[Au]-[Si or SiO2]-[Ti]-Diamond. Gold is located in the middle of the layered structure and plays the role of the color center realizing pink color.

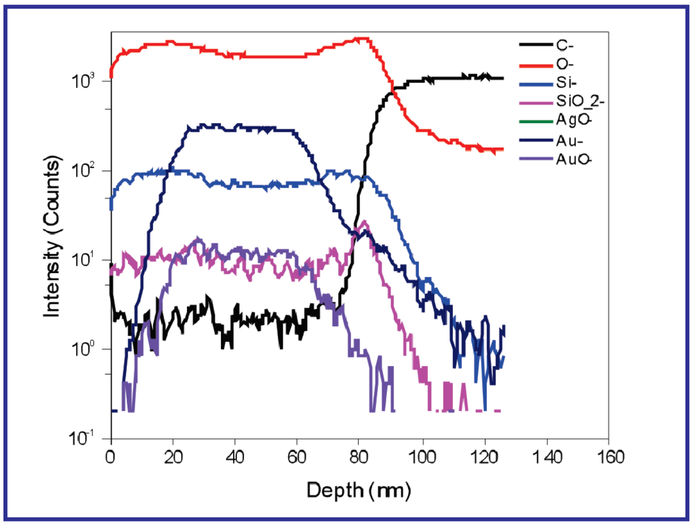

Figure 4 (a). The SIMS depth profile of the 0.68 ct Ice Pink Diamond. Negative ion data collection mode.

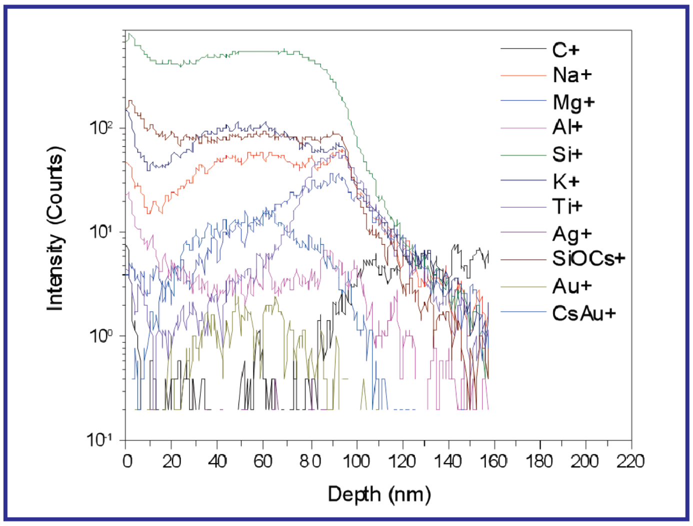

Figure 4 (b). The SIMS depth profile of the 0.68 ct Ice Pink Diamond. Positive ion data collection mode.

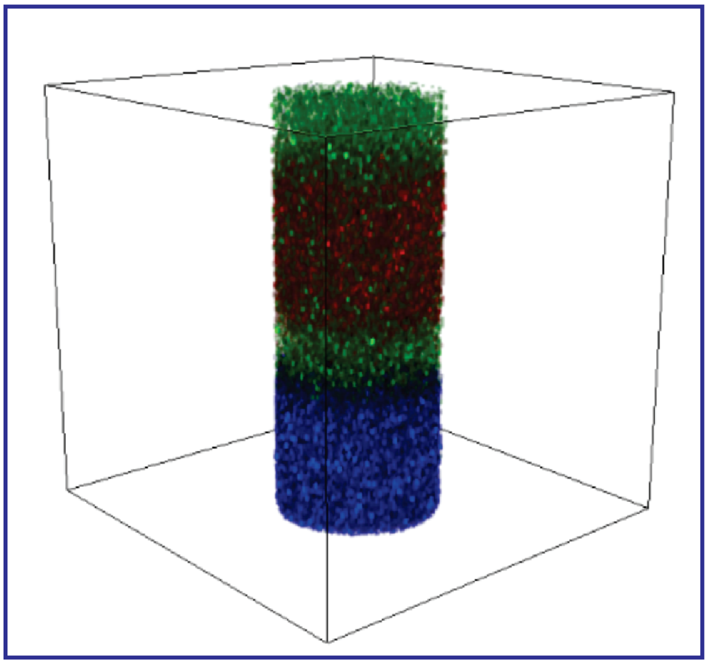

Figure 5 (a). 3D Render overlay of Au^-(Red), Si^-(Green), and C^-(Blue)

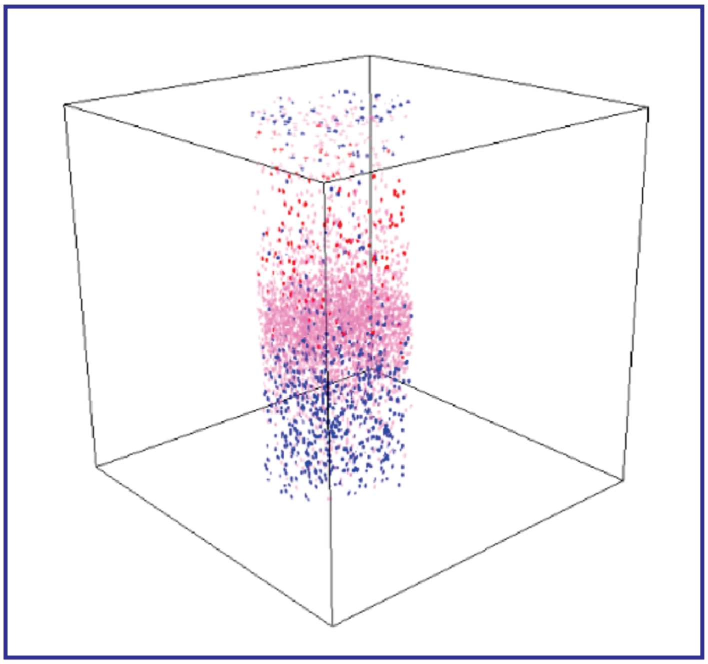

Figure 5 (b). 3D Render overlay of Au^+(Red), C^+(Blue), and Ti^+(Pink)

In summary, the spectroscopic study and chemical analysis of a coated pink diamond product were performed. The thin film coated on the sample is no thicker than 100 nm and consists of a multi-layered structure. In the coating film, the titanium on the diamond surface increases the adhesion between the diamond and SiO2, which constitute most of the coating layer and gold is placed within the SiO2 layer. The multi-layers are stabilized by Ti and Si while Au is functioning as a color center.

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COATED DIAMOND TO IMITATE PINK DIAMOND

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A customer brought a 1.6 carat 'pink diamond' ring which he suspected that the price of US$13,500 was too good to be true and asked our laboratory to examine it. He requested that the diamond is not to be removed from the setting during our observation.

The round brilliant cut 'fancy greyish yellowish-pink diamond' measures approximately 7.56 x 7.56 x 4.8mm, the clarity grade was I1 and the cut grade was good in polish and symmetry (Fig.1). Using Presidium Gem Tester, it indicates diamond.

Fig 1: The 18K rose-gold ring with questionable central stone of "pink-diamond" essence and shank with small round brilliant cut diamonds

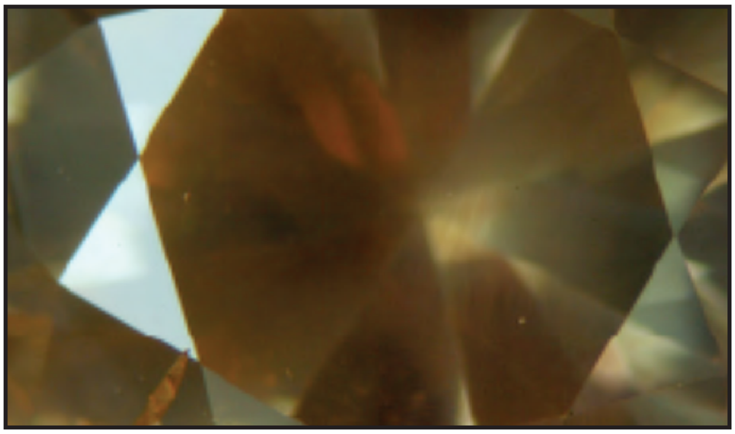

Fig2: Clarity grade of the pink diamond was found to be I1 with hazy inclusions and fractures on bezel facet

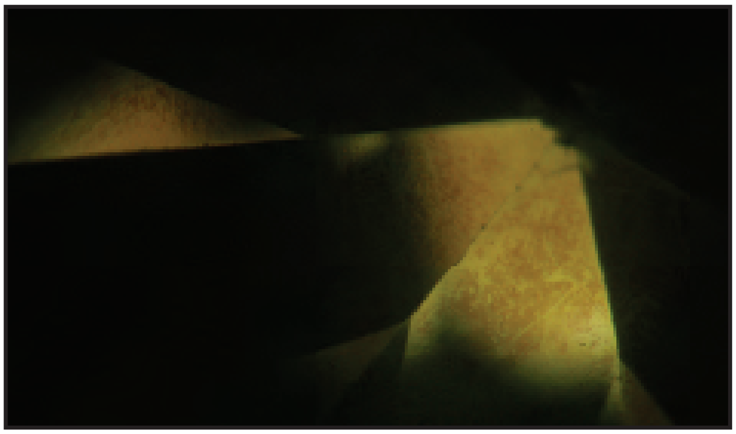

Fig. 3: Using transmitted light on focusing into the diamond from the crown region, the pavilion facets showed pink colour with no colour on the facet edges (25X magnification)

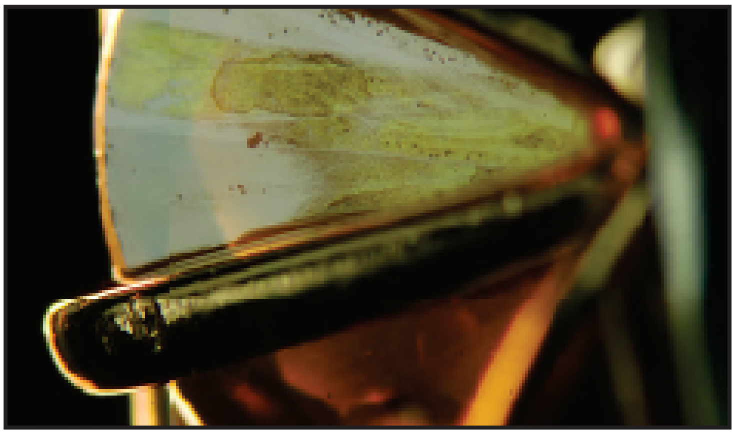

Fig. 4: Using reflected and transmitted light the pavilion region surface showed yellowish colouration due to wear and tear

Fig.1. The 18K rose gold ring with questionable 'pink diamond' essence with small round brilliant cut diamond on the shank. Fig.2. The clarity grade is I1 with rather hazy inclusions and fractures on the bezel facets (15x magnification). Fig.3. Focus into the diamond from the crown under transmitted lighting, the pavilion show patches of pink colour with clean edges (25x magnification). Fig.4. At the pavilion area under reflected and transmitted lighting, the surface staining show yellowish in colour and some were worn off due to wear and tear (15x magnification).(Photo by Tay)

Under magnification, from the crown area, the diamond appears rather hazy. This is due to very minute cloudy inclusions and some minor fractures on the side of the diamond (Fig.2). On focusing into the diamond from the crown area under transmitted lighting, the pavilion area shows patches of pink colour with some coating being scratched off and the facet edges appearing lighter in colour (Fig. 3). Turning to the pavilion area, under reflected lighting, the surface coating appears more prominent with yellowish staining which is partially worn off due to wear and tear (Fig.4). Using desk top prism spectroscopy, no 415nm absorption was observed. The diamond fluorescence faint yellow in the long wave UV and none in the short wave UV.

Conclusion: Coated diamond to imitate pink diamond has been reported in the literature before, but this is the first time that our laboratory encountered this treated material. The coating of this diamond is found on the pavilion area, and is quite easily identified using magnification. Although no advance gemological method could be employed, classical gemological method such as magnification is still very much important in this work.