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Bulk Luminescence Spectroscopy - Fast Way to Screen and Separate Natural Diamonds from Treated and Synthetic Diamonds

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1. INTRODUCTION

This paper deals with the problem of identification and certification of diamonds when synthetic diamonds, larger then 2ct, are becoming common enough to meet the needs of the diamond gem market.

Recent advances in growth and treatment of diamonds have attracted much public attention. Yet relatively little has been published on the subject.AG&J has witnessed large and small companies investing in production of HPHT-grown and CVD-grown synthetic diamonds as well as in diverse treatment technologies including radiation, HPHT, APHT andLPHT techniques. 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 current level of the technologies that slows new investments.

We are addressing the potential risk the certifying laboratories may run in when being unable to identify the certified diamonds. That is, when the certifying laboratoriesare unable to answer the ultimate question whether a diamond accompanied by a certificate is indeed the diamond this certificate has been issued on.

Based on the data contained in most diamond certificates, it is relatively easy - especially with round diamonds - to manufacture closeor identical replicas of the certified diamonds. These fraudulent twin diamonds, made of much cheaper material (either treated, or synthetic diamonds), replace the original stones and are sold with the authentic certificates for premium price. This dirty business is primarily done with typeIIa and IIb diamondsof high color grades (grade H and better) and high clarity grades (VVS and better).

The solution of this problem, which we propose in this communication, is the inclusion in diamond certificates standardized information on infrared absorption and visible fluorescence of the certified diamonds. This information, presented either digitally, or in form of spectra, can be reproducibly obtained by inexpensive FTIR and fluorescence spectrometers and thus can be easily checked in any gem laboratory, or even by individual jewelers. FTIR instruments designed for diamond characterization are well known to the industry and owned by most laboratories. The suitable fluorescence spectrometers are not commercially available yet. However, the design of such a spectrometer and its integration in one system with standard FTIR spectrometer does not pose any problem and can be accomplished with little investment.

The information presented in most certificates relates to diamond shape/cut, measurements, carat weight, clarity grade, color grade and fluorescence. It is not so difficult to find or make replicas that match the parameters of shape/cut, weight, clarity and color. To reproduce identical measurements is also possible, but it is time consuming and expensive. Thus the measurements, especially the comprehensive ones,can cause considerable a problem in terms of making twin diamond. The "reproduction" of fluorescence could be the most severe tasksince every natural diamond has its individual fluorescence. Yet, since the fluorescence grading is too simple and vague and, consequently, insufficient. Indeed, a standard fluorescence grading qualitatively differentiate diamonds only in terms of their fluorescence intensity: strong, medium, weak and none, where the "intensity" is based merely on the perception of human eye. Usually, no information on thecolor of fluorescence is provided. However, it is well known that every diamond (natural and synthetic) produces its unique fluorescence, which is always "visible" if measured by an appropriate spectrometer. This peculiarity of fluorescence is the reason why fluorescence/photoluminescence spectroscopy is used as the most reliable method of separation diamonds of different origin and different treatment history.

Working on the problem of recognition of seemingly identical diamonds we have come to conclusion that the most vulnerable parameters of gem diamond in terms of their fraudulent "reproduction" are visible fluorescence excited in the near UV range, e.g. at a wavelength of 405 nm. Besides the fluorescence, IR absorption in the spectral range 300 to 4000 cm^{-1}could also considerably contribute to reliable identification of diamonds.

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Below, we show a few examples of spectroscopic differentiation between the diamonds of same color, but different origin and different treatment history.

2. MATERIALS, METHODS, INSTRUMENTS

In the present communication we present exemplary data obtained on 18 diamonds in four colors – colorless, yellow, pink and blue. Color grade of all diamonds per group were almost identical (or with one grade difference).

Colorless— natural/natural; natural/HPHT treated; synthetic/HPHT grown; synthetic/CVD grown.

Yellow— natural/natural; natural/treated; natural/HPHT treated; synthetic/HPHT grown; synthetic/ CVD grown – treated.

Pink— natural/natural; natural/multi step treated; synthetic/HPHT grown – treated; synthetic/CVD grown – treated.

Blue— natural/natural; natural/treated; natural/HPHT treated; synthetic/HPHT grown; synthetic/CVD grown – treated.

IR absorption of the diamonds was measured with Bruker Alpha FTIR Spectrometer. This spectrometer could provide reliable spectroscopic data for each diamond even working in a fast regime of low resolution (4 cm^{-1}) and 4-scan accumulation.

For fluorescence characterization we used custom-built "Express-Spectrometer" based on two Ocean Optics spectrometers with spectral resolution 2 nm and 10 nm respectively. A 300 mW laser diode working at a wavelength of 405 nm was used to the excitation. All measurements were performed at room temperature.

The whole process of spectroscopic characterization was very fast, taking not more than 5 seconds for IR absorption 5 seconds for fluorescence.

3. RESULTS AND DISCUSION

3.1 IR Spectroscopy

IR absorption spectra of the studied diamonds are shown in Fig. 1 below.

Colorless diamonds— Majority of natural colorless diamonds are of type Ia, whereas all colorless treated and synthetic diamonds at the moment areof type IIa only. However, IR absorption fails to differentiate between colorless natural type IIa and HPHT-treated type IIa diamonds. Fortunately, these type IIa diamonds are rare.

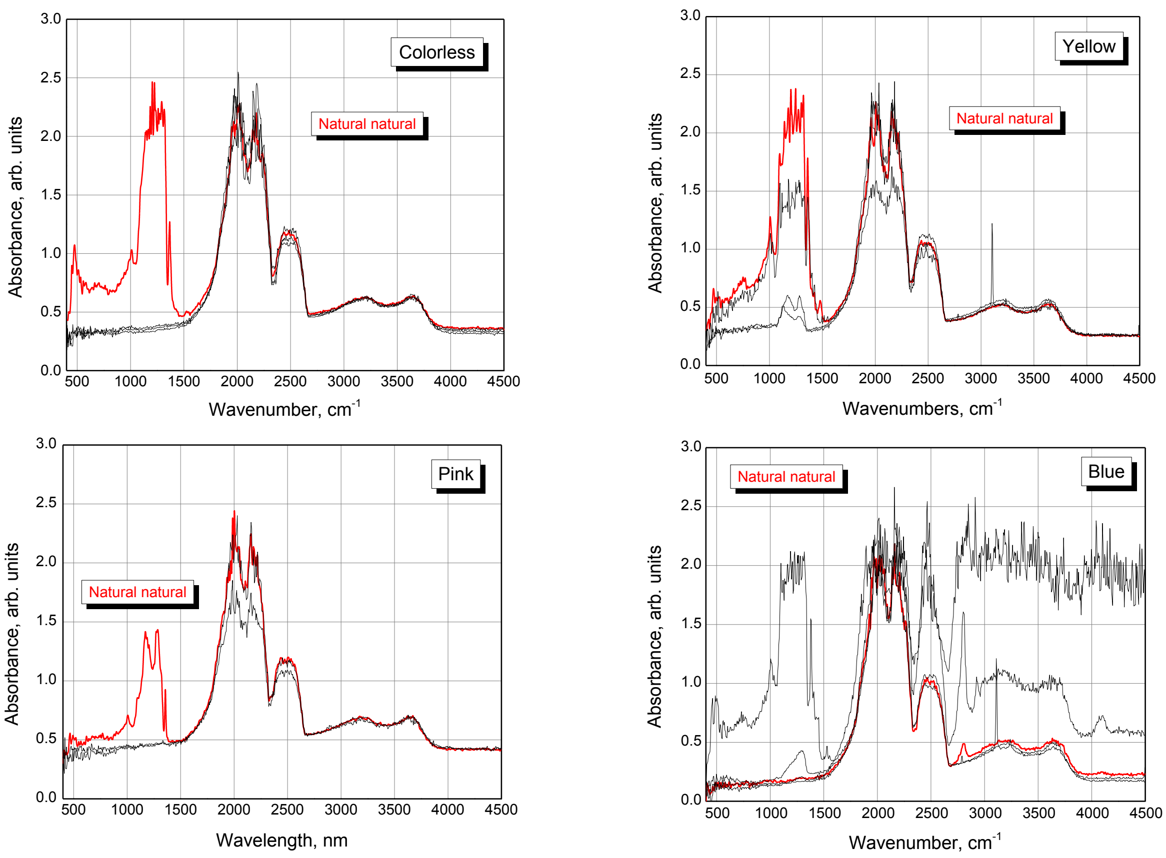

Fig. 1: Colorless (b), yellow (b), pink (c) and blue (d) diamonds

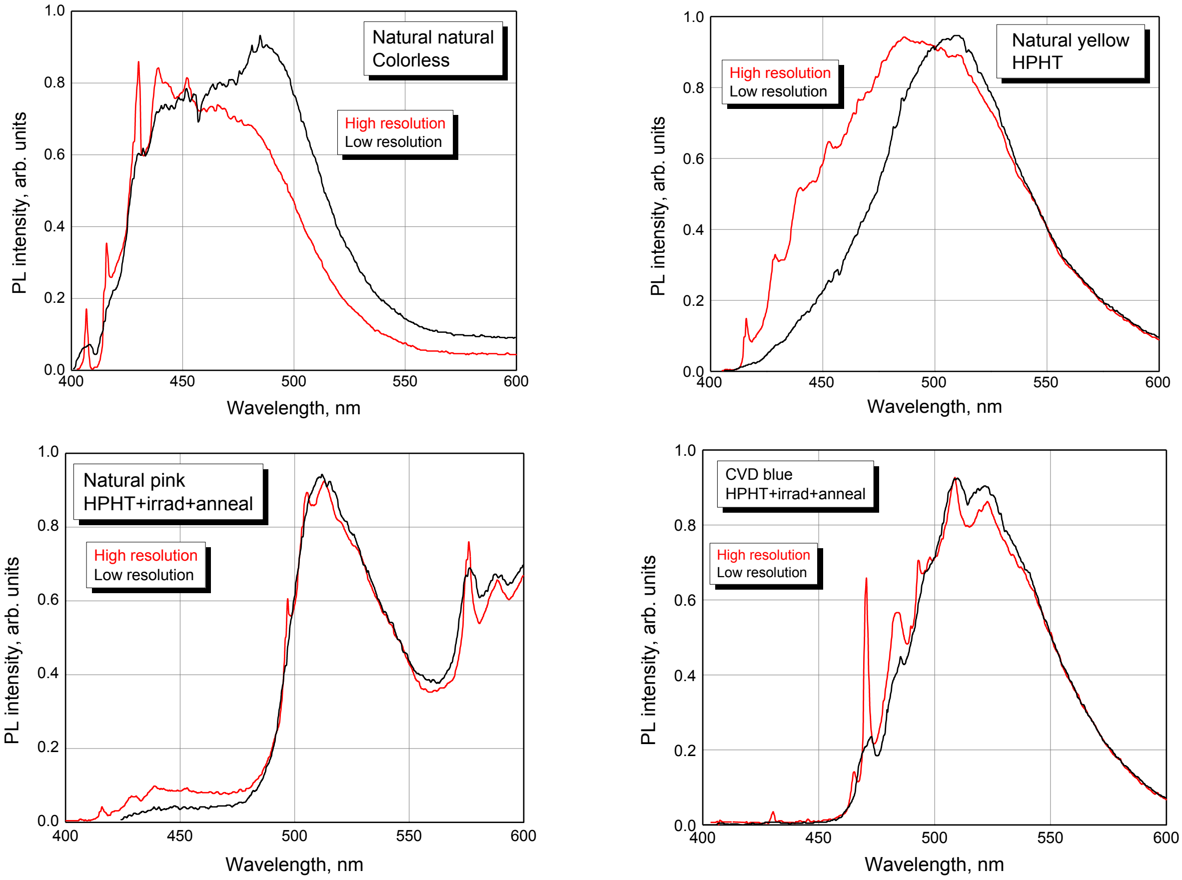

Fig. 2: Exemplary spectra showing comparison of high resolution and low resolution fluorescence spectra for diamonds of different color

Yellow diamonds— IR spectrometry can reliably differentiate between yellow natural (mostly type Ia) and yellow synthetic diamonds (mostly type Ib for HPHT-grown and type IIa for CVD-grown). However yellow natural diamonds and HPHT-treated yellow natural diamonds are mostly of type IaAB and they cannot be reliably differentiated by IR spectroscopy alone.

Pink diamonds— In most cases, natural pink diamonds are of type Ia, whereas pink treated and synthetic diamonds are of type IIaor very low N type Ia. Thus IR spectrometry is an effective screening method for pink diamonds.

Blue diamonds— Most natural blue stones are low boron type IIb diamonds. This reliably distinguishes them from most HPHT-grown and irradiated blue diamonds. However, CVD-grown blue diamonds may show IR spectra identical to those of natural blue diamonds.

It is seen that in most cases the natural untreated diamonds can be reliablyseparated from their synthetic and treated counterparts based on IR spectroscopic characterization only. Yet, the IR absorption, if used alone, may fail in recognition of HPHT-treated colorless and pink diamonds of type IIa, yellow HPHT-treated diamonds and blue CVD-grown diamonds.

3.2 Fluorescence Spectroscopy

Fig. 2 shows fluorescence spectra of the studied diamonds measured in different regimes. Actually, every spectrum is individual. It is especially obvious in case of high resolution spectra, which reveal multiple fine features characteristic of particular diamond. However, even the low resolution spectra are individual enough in order to provide reliable separation of diamonds of different origin and different treatment.

Comparison of fluorescence spectra obtained on diamonds of same color but of different origin and color treatment is shown in Fig. 3. It is seen that almost everywhere the diamonds of natural origin and natural color can be separated from their synthetic and color-treated counterparts. The most distinguishing feature here is the band with maximum at about 485 nm. Yet, some synthetic and treated diamonds may show fluorescence bands similar to that observed in natural/natural diamonds. However, these “questionable” diamonds have different IR spectra. Thus in combination, fluorescence

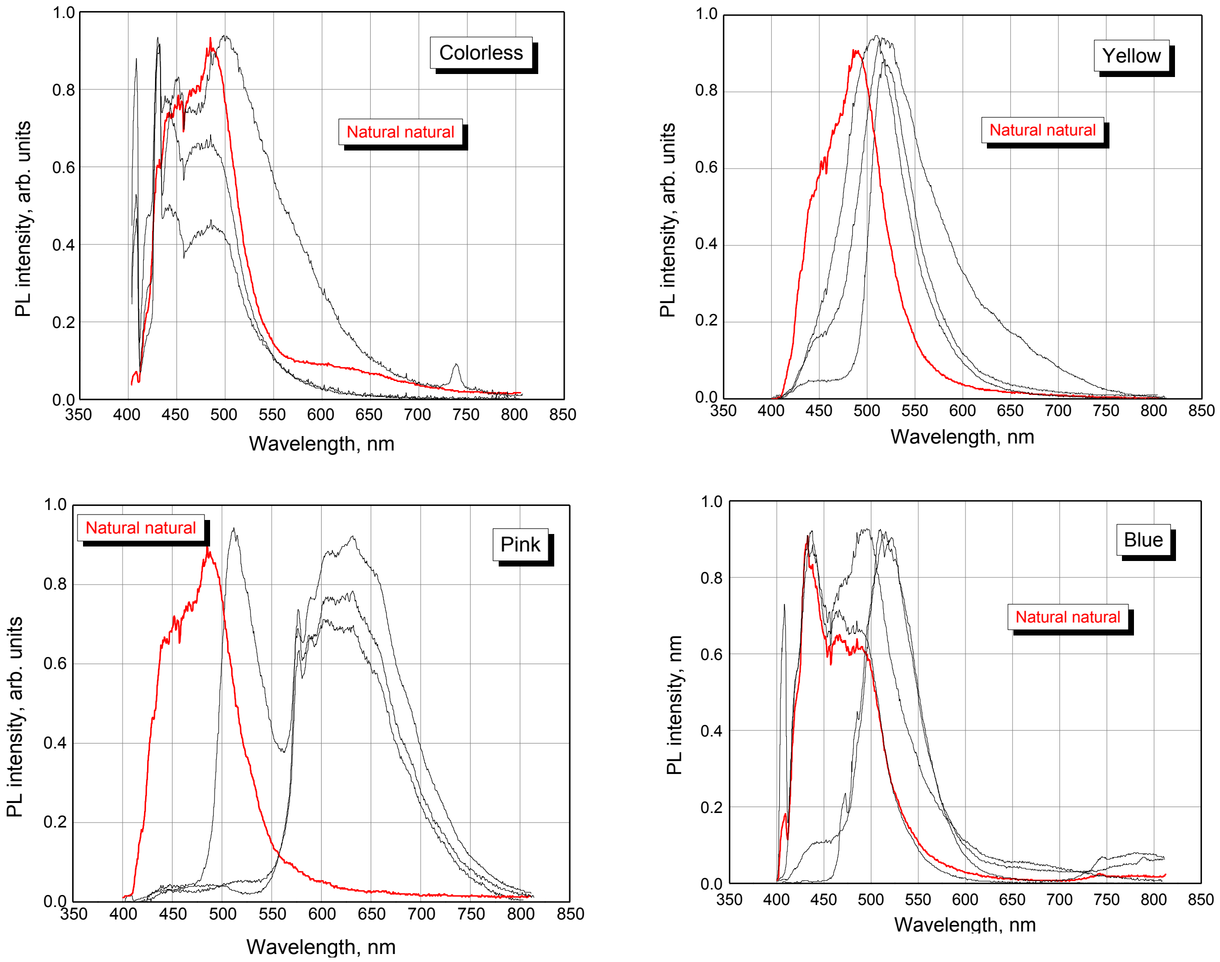

Fig. 3: Low resolution fluorescence spectra.

and IR spectroscopy can provide reliable screening in practically any case.

4. CONCLUSION

We show that combination of simple IR absorption and UV-excited fluorescence spectroscopies is a method of reliable identification and confirmation of authenticity of natural diamonds. The spectroscopic data, when added in digital, or spectral form to diamond certificates, could solve the problem with fraudulent trade in twin diamonds. We have demonstrated the usefulness of this approach using simple IR absorption spectrometer and home-made fluorescence spectrometer. Thus we anticipate that the design of an instrument to be used for the spectroscopic characterization will not be a challenge. This instrument will be inexpensive, it will work in fully automatic regime and it will not require any advanced qualification to operate it.

We propose to introduce the Spectroscopic parameter of a diamond as its 5th C (in Cyrillic, “C” stands for “S” - Spectroscopy). This additional C will give possibilityto every jeweler to verify the authenticityof the diamond he works with using cheap and handy instrument.