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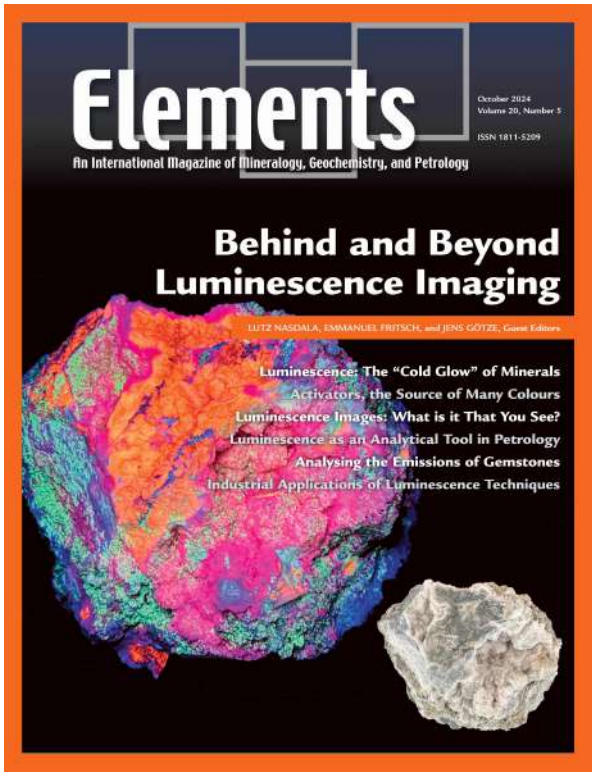

Analysing the Luminescence of Gems

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Photoluminescence associated with the web-like dislocation network in a perfectly colourless natural type IIa diamond. 220-nm UV excitation, image width = 0.75 mm.

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Luminescence imaging and spectroscopy have become essential in gem testing, as most gem minerals and materials exhibit specific luminescence when properly excited. With a range of techniques introduced in gem testing laboratories in the past quarter century, such as luminescence imaging and photoluminescence (PL) emission and excitation spectroscopies, there are many applications to gem materials for establishing their identity, separating natural from synthetic gems, and detecting potential treatments. Further, these techniques often give clues towards the identity of emitting defects. Luminescence-based testing has recently gained attention even outside the gemmological laboratory as many simple luminescence-based instruments are offered to the gem and jewellery trade to separate natural from synthetic diamonds or from their imitations.

KEYWORDS: gems; diamond; ultraviolet; photoluminescence; melee-screening; defects; spectroscopy; imaging

INTRODUCTION

Luminescence has been traditionally considered just a basic observation method for gemmologists, testing with simple ultraviolet (UV) lamps emitting at approximately 365 nm (long-wave ultraviolet; LWUV) and 254 nm (short-wave ultraviolet, SWUV; Boehm 2002). Luminescence has been used in gem testing for about 100 years (Michel and Ridel 1925). Yet, since the mid-1990s, the use of luminescence in a much broader sense has virtually exploded in gemmology. It all started with the introduction of the DiamondView™ "deep UV" (generally 200–280 nm wavelength; here, ca. 225 nm excitation) luminescence imaging system for the visual distinction of synthetic from natural diamonds (Welbourn et al. 1996). The patterns revealed are different, as for example, some synthetic diamonds show cubic growth sectors that are absent from all natural diamonds (FIG. 1). The advent of high-pressure high-temperature (HPHT) treated diamonds in 1998 (Smith et al. 2000) then required differentiating impurity-free diamonds with almost the same properties: initially brown diamonds treated to near-colourless, from their naturally near-colourless counterparts. Only luminescence spectroscopy was sensitive enough for this challenge, with the help of laser-excited, liquid-N_2 temperature emission spectroscopy (Dobrinets et al. 2016). Today, these techniques have found new applications in many gem materials (Zheng and Shen 2023).

While the nearly three decades of exploration and development of advanced luminescence methods in gem testing have led to much progress in the analysis of gem materials, the increased use of luminescence in gemmology also reveals that, in many cases, very little is truly understood about the activator mechanisms involved (for activators, see Waychunas and Kempe 2024 this issue). The evolution mentioned above has led progressively to the design of instruments specific to the field, well adapted to the size and shapes of gems, as well as entire pieces of jewellery. Although the classic UV lamps (see above) remain the staple for practicing gemmologists, their disadvantage is that LWUV lamps typically have a rather low output power compared with devices used in advanced luminescence methods, and that they emit slightly in the visible range as well, with a deep violet glow that renders the observation of violet luminescence impossible. With the advent of light emitting diodes (LEDs) since the 1990s, nearly monochromatic 365- and 254-nm UV sources became available. LEDs also have the advantage that they are easily collimated and focused. Today, high power 365-nm LEDs are available that emit significantly more UV than classic Hg LWUV lamps. On the shortwave side, LED devices are currently not powerful enough to be viable alternatives to the classical SWUV tube.

THE DEVELOPMENT OF SPECIFIC INSTRUMENTS

Laboratories delivering reports on gems have progressively moved from the classical UV lamp to other designs including more powerful excitation sources, which unveil weak emissions and create better resolved emission spectra to identify the emission. Higher-power UV excitation on a wider spectral range is obtained with gas-discharge arc lamps. Some of the most versatile are continuous wave (cw) Xe arc lamps that emit from about 200 to 2400 nm. Xenon lamps are great sources for deep UV, which is most easily selected from pulsed Xe sources instead of cw sources. Excimer lamps are alternative sources for short

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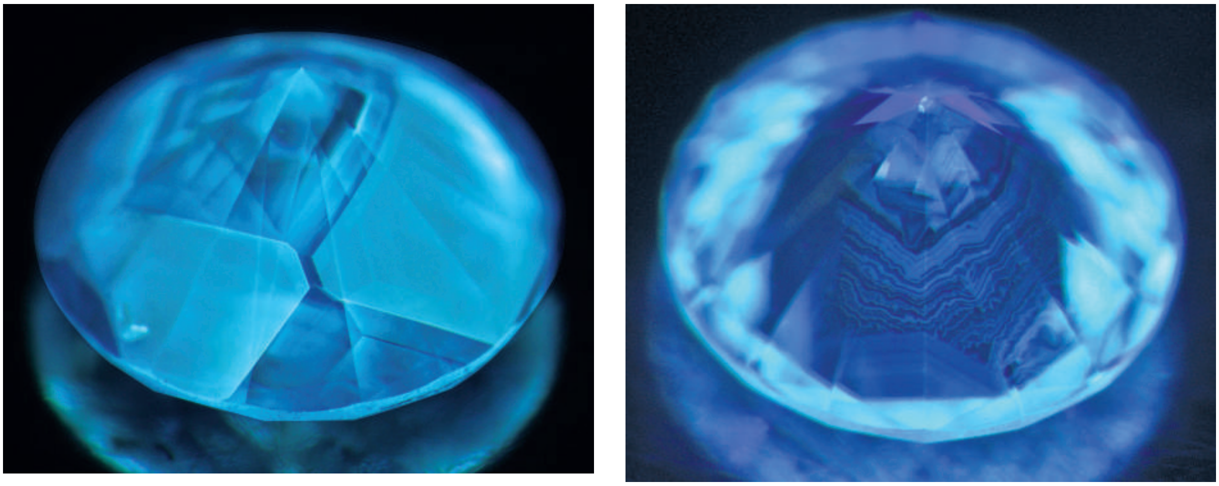

Figure 1 This UV luminescence image (220 nm excitation) of a high-pressure, high-temperature (HPHT) synthetic diamond (**Left**) contrasts with that of a natural diamond of similar appearance in daylight (**Right**). The presence of both octahedral ("chalky" greenish blue) and cubic (blue) growth sectors in the left image unambiguously indicates a synthetic diamond. On the right, there are undulating horizons of cuboid growth, typical of and unique to natural diamond. Photos recorded using a DFI system. Sample diameters = 3.5 mm (**left**) and 2.8 mm (**right**).

UV wavelengths, the most useful in gem testing, emitting at 222 nm; deep UV excitation close to 225 nm is particularly useful for colourless to near colourless diamonds as it excites practically all of these diamonds, whereas classic UV lamps excite only about a third of them. Lasers are the highest-power and truly monochromatic luminescence excitations. While the most popular wavelengths in the visible and near infrared domains are relatively affordable, UV lasers are expensive and often not very intense; pulsed UV lasers are available with higher output powers. Widely available simple diode lasers emitting between 400 and 407 nm are affordable tools to excite luminescence, and are today often used to excite luminescence in diamond and some coloured gems.

While simple visual observation of the luminescence of gem materials can be useful, the true wealth of information hidden in luminescence imaging can only be unveiled using magnification and cameras. There are four points to be considered to gain the most information from luminescence imaging, namely (1) excitation wavelength(s), (2) colours, (3) intensities, and (4) distribution patterns. Integrating the excitation light sources within a microscopy system is the ideal way to exploit the potential of luminescence imaging. In this case, either direct visual observation using a microscope or imaging through a camera can be used. Because the intensity of luminescence is often low, sensitive cameras with cooled detectors can help to visualize emissions otherwise too weak to be perceived by the human eye. Through luminescence microscopy, one can obtain a lot of information from luminescence colours, intensities, and distribution. Introduced in 1996, the camera-based DiamondView^{TM} instrument was the first luminescence imaging system made for gem testing. It used ca. 225-nm-deep UV excitation to observe the growth textures (internal morphology) in diamonds (Welbourn et al. 1996). This instrument also documents phosphorescence, which has some value to distinguish natural from synthetic diamonds.

The UVisio system developed by GemTechLab in the late 1990s used a Hg arc lamp to excite luminescence using 365- or 425-nm light. The main use of this system was to identify and quantify fillers in emerald (the Cr-coloured green variety of beryl) and other gemstones (Devouard and Notari 2009).

Recently, the method of time-resolved luminescence has been introduced to visually distinguish colourless synthetic diamond from natural ones. In this technique,

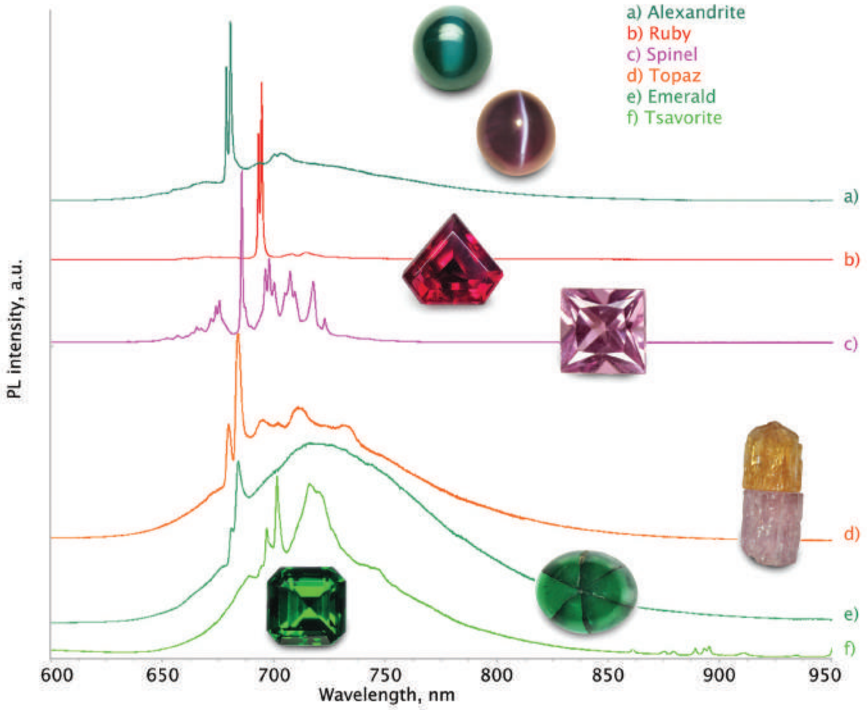

Figure 2 Emissions (and absorptions) related to Cr^{3+} vary depending on the crystal structure in which Cr is incorporated; thus, their detection is instrumental in quickly identifying a gem species. **Top to bottom**: PL spectra (excited at 532 nm) of chrysoberyl (alexandrite), corundum (ruby), pink spinel, orange and pink topaz, beryl (emerald), and grossular (tsavorite). Abbreviation: a.u. = arbitrary units. Sizes of stones range between 5 and 15 mm.

it is basically measured if a luminescence persists for long or short periods, that is, if the luminescence dies off slowly or rapidly after excitation. Long luminescence is an indication of synthetic origin in certain circumstances.

The easiest way to elucidate the cause of luminescence is via PL spectroscopy (see Nasdala and Fritsch 2024 this issue). In most gem testing laboratories, PL is performed on commercial Raman spectroscopy systems, as Raman was a routine testing method well established prior to the use of luminescence methods (Kiefert and Karampelas 2011). Simple integrated PL instruments for diamond testing (such as the ones using the presence of a naturally occurring defect in most diamonds—the so-called N3 defect emitting at 415 nm) have been introduced recently to distinguish colourless to near colourless diamond from synthetics and imitations.

The spectrometers used to measure PL of gem materials should—in the best scenario—be highly sensitive, using a wide spectral range and a spectral resolution of 0.2 nm or better to be able to detect as many defects as possible and to properly resolve all spectral features. In our experience, PL peaks in diamond can be as narrow as 0.08 nm; therefore, a high spectral resolution is essential. PL spectra are significantly enhanced when they are recorded at low temperature, which is typically done by cooling the gem with liquid N_2. Indeed, the bandwidths of many PL features in gems—particularly diamond—narrow significantly at low temperature, and therefore weak features are revealed. Certain defects are even undetectable in the PL spectra at room temperature and only the cooling process renders them detectable. Effectively, the cooling of gems for PL spectroscopy significantly enhances the sensitivity of the testing process.

For excitation spectroscopy (see Nasdala and Fritsch 2024 this issue), a Xe lamp coupled to a monochromator is often used to produce near-monochromatic light at any desired wavelength between about 250 and 800 nm. Subsequently, the intensity of a specific emission is measured by scanning the excitation wavelength, nm by nm.

Pushing the analysis technology one step further, the diamond fluorescence imaging (DFI) system introduced in the market in 2015 was developed as a microscopy- and spectroscopy-based high-power UV luminescence microscope, using six different excitations between 215 and 400 nm plus a 405-nm laser to trigger luminescence. The system is coupled to a camera for long-exposure imaging and to a spectrometer to enable the user to perform both luminescence imaging and spectroscopy at the same time, and to record Raman spectra using the 405-nm laser. Such a combined system enables the analyst to very efficiently screen gemstones, which is particularly important for very small gems (occasionally as small as 0.4 mm diameter), known in the trade as "melee" (Hainschwang and Notari 2015) and usually submitted to gem labs in batches of hundreds to thousands of stones. It is particularly useful to rapidly screen natural from synthetic diamond melee. For such tiny specimens, luminescence is the most useful technique, if not the only one applicable.

SOME IMPORTANT APPLICATIONS OF LUMINESCENCE ANALYSIS IN GEM TESTING

Gem Species Identification

The visually observed luminescence reaction, even with a classic UV lamp, may be indicative of particular gem materials, synthetics, or imitations. One example is benitoite, a rare gem found mostly in San Benito County, California, USA, which exhibits a characteristic blue luminescence caused by titanate groups (octahedral TiO_6) under SWUV excitation, while it is inert under LWUV (Gaft et al. 2004). Using luminescence microscopy with several excitations covering the entire UV range, the observation of luminescence colour and luminescence distribution will help to quickly identify most natural corundum and diamond and other gem materials, such as tsavorite (a green variety of grossular) and certain tourmalines.

A more conclusive and very rapid method for the identification of some gem species is PL spectroscopy, in particular for those with Cr^{3+} as an activator. In many cases, even low concentrations of Cr will result in significant and easily detectable PL when appropriate laser excitation is used (Hainschwang et al. 2013). The best-known examples of Cr-bearing gems include alexandrite (a variety of chrysoberyl), corundum (ruby and sapphire in all colours), spinel, orange to pink topaz, emerald, and tsavorite (FIG. 2). Their PL spectra identify the species conclusively as the

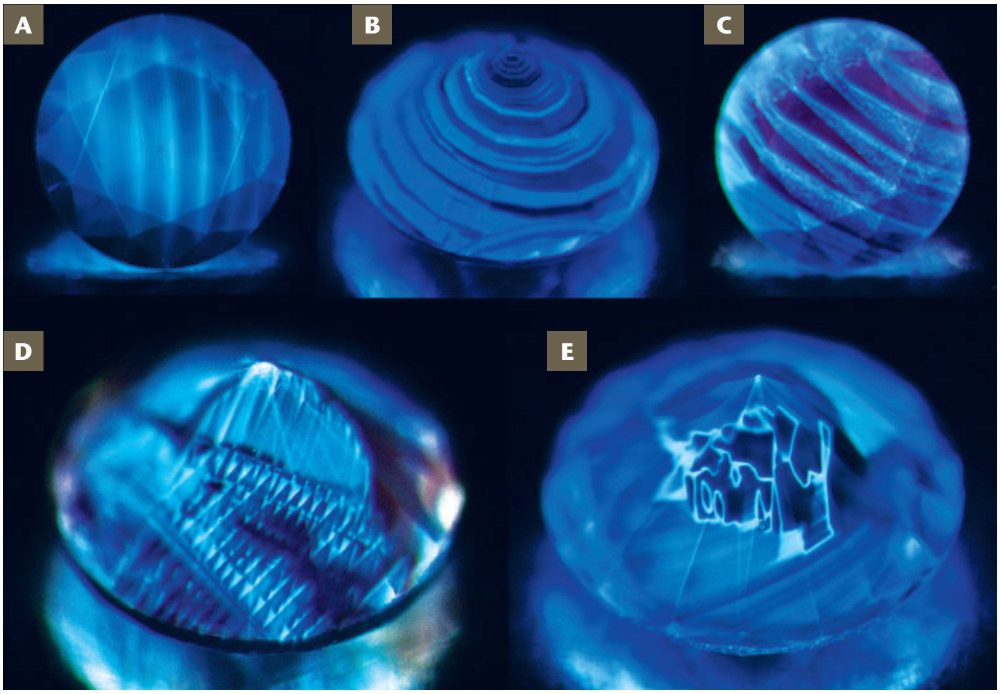

FIGURE 3 In gem corundum, blue to green luminescence in SWUV can be indicative of heat treatment above 1500 °C or of a synthetic origin. The curved luminescence pattern (**A**) is indicative of a flame-fusion synthetic blue sapphire. Beyond this useful information, these examples also reveal spectacular growth patterns, such as growth zoning in successive layers (**B, C**), sectorial zoning (**D**; each "tooth" of the "dinosaur jaw" represents equivalent growth sectors), or even a strange writing-like pattern, possibly caused by dislocation loops (**E**). All stones except stone (**A**)—which is a Verneuil synthetic blue sapphire—are natural blue sapphires that have undergone high-temperature (1600 to 1700 °C) heat treatment. Sizes of stones range between 2.0 and 3.2 mm.

exact peak position(s) and shape(s) of the Cr^{3+}-related emission(s) depend on the structure into which the Cr ions are integrated. This completely non-contact and non-destructive technique, testing a gem at a short distance, can be useful for the rapid identification of gems in cultural heritage objects (Nasdala et al. 2023).

Synthetics and Treatments Identification

Photoluminescence can give valuable information about various gem treatments, and also whether the gem is synthetic (grown in a laboratory) or of natural origin. In general, the Fe concentration should be low, as iron tends to quench luminescence (see Waychunas and Kempe 2024 this issue). We will limit examples to only two important gem species: sapphire and spinel. Diamond is dealt with separately further below.

Sapphire. While the identification of corundum by PL spectroscopy based on Cr^{3+} emission is straightforward, the distinction of natural corundum from its synthetic counterparts is more demanding, as the PL spectra of synthetic and natural corundum are generally identical. One efficient way of distinguishing natural from synthetic corundum is via luminescence imaging. When titanate groups are present, then the luminescence is blue (Vigier et al. 2023) but may appear rarely green to nearly white from the presence of other emitting defects. This luminescence is most distinctly developed in Ti-bearing corundum after heat treatment (>1500 °C) as Ti diffuses from rutile inclusions into the corundum lattice, forming titanate groups; therefore, it serves as a good indicator of heat treatment in blue sapphires (FIG. 3B–3E).

This luminescence is also commonly seen in flame-fusion synthetic corundum (Vigier et al. 2023). The often-complex growth history of natural corundum, compared with the rather simple growth of flame-fusion and Czochralski synthetic corundum, can be visualized by luminescence imaging. The varied reactions of different growth sectors may produce spectacular and yet useful images to identify the growth process (FIG. 3). The most common synthetics exhibit either homogeneous luminescence or rather simple patterns such as the curved growth horizons of flame fusion corundum (FIG. 3A). Caution must be exercised as flux grown synthetic corundum can exhibit complex growth-related luminescence that may resemble the type of patterns one observes in natural corundum. Some flux grown synthetic corundum commonly exhibits green luminescence, a fluorescence colour that is very rare in natural corundum.

Spinel. Photoluminescence spectroscopy is a powerful and fast method to distinguish unheated from heated natural spinel, and also both flame-fusion and flux-grown synthetic spinel. Albeit a rarely worthwhile treatment, some spinel heated above 700 °C will exhibit a modified colour, often shifting towards a more orange hue in pink to red spinel. The PL spectra of heat-treated natural spinel and synthetic spinel exhibit broadened peaks owing to some lattice disorder (due to partial inversion in the occupation of Mg and Al sites; Widmer et al. 2015). The Cr^{3+}-related PL emissions are the most sensitive means to measure this effect as they are very narrow and rather intense in most spinel (FIG. 4).

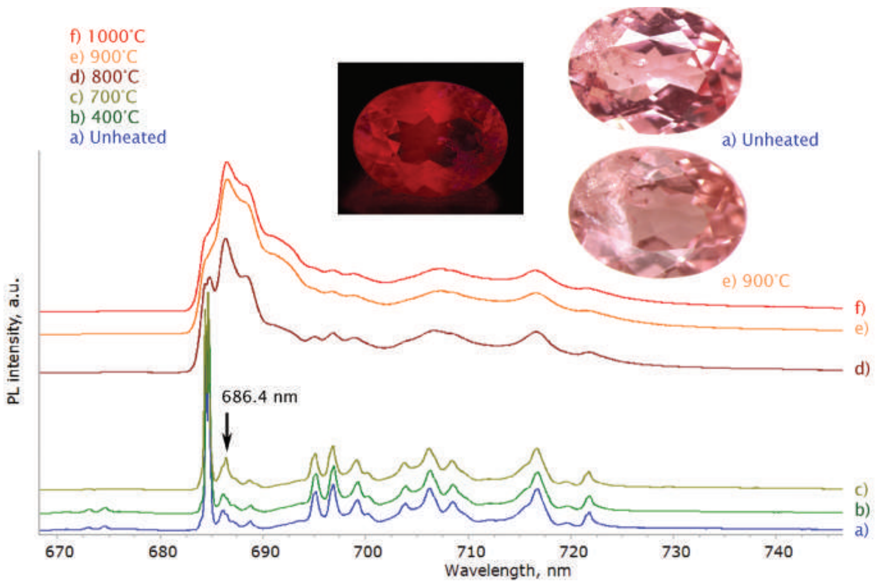

FIGURE 4 The colour of spinel can be changed by heating (in this case, the orange colour is slightly enhanced). To detect this treatment, PL spectra (532 nm excitation; analysis at –196 °C) of Cr^{3+}-coloured pink spinel have been recorded before and after heating. The red luminescence colour—here shown under LWUV—remains unchanged. The emissions are sharp for the untreated stone, whereas they are considerably broader after heating above 700 °C. Abbreviation: a.u. = arbitrary units. Sample dimensions (length × width) = 7.0 × 5.1 mm.

Diamond Testing

Luminescence in diamond exists in all possible hues, from violet to red to white. With adequate, powerful UV sources, virtually all diamonds luminesce. As mentioned above, the internal morphology revealed by luminescence (growth zonation) indicates the growth history of the crystal, and based on this, natural diamonds can be distinguished from synthetic diamonds (Eaton-Magaña and Shigley 2016; Eaton-Magaña et al. 2017). Additional details can be gained by using different excitations.

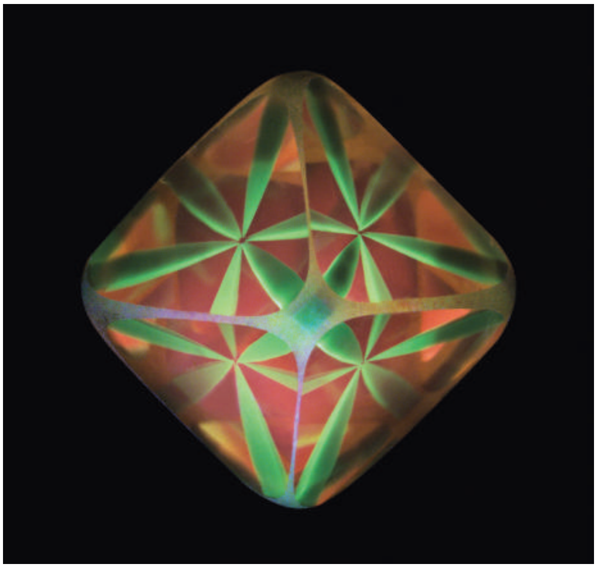

FIGURE 5 The 11.38 ct "Rhodesian Star" diamond is a spectacular example of how luminescence varies among different growth sectors. The cuboid sectors luminesce green from Ni defects (the so-called S3 centre), whereas the octahedral sectors luminesce orange pink from unidentified centres. Image recorded using broadband excitation centred at 320 nm of the DFI microscope.

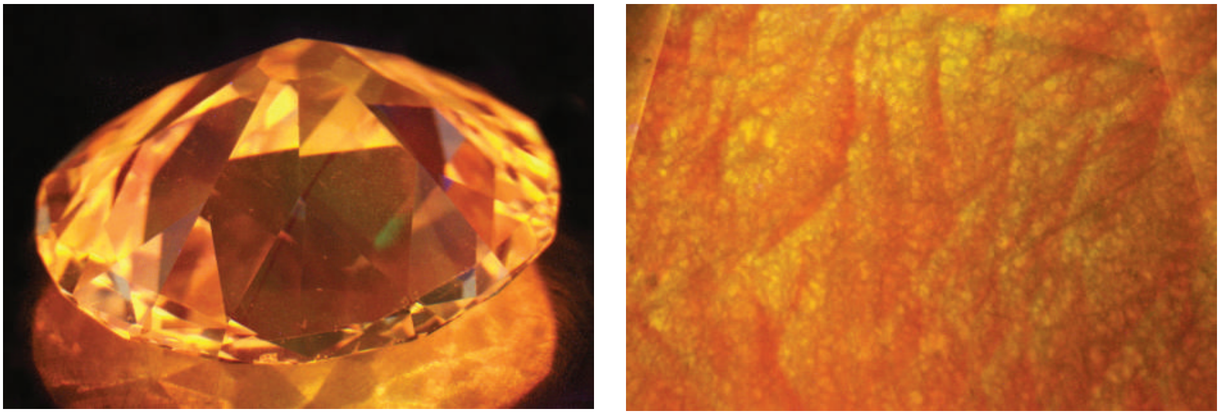

Figure 6 (Left) This 13.20 ct brown pink type IIa (chemically pure) diamond has a strong orange emission under ca. 220 nm UV excitation due to a very low concentration (on the order of 1 ppm) of the NV^0 centre. (Right) This detail enlargement (width = 4 mm) shows a polygonised network of dislocations, visible only with luminescence. This pattern reveals that the crystal has undergone major deformation, a characteristic of natural type IIa diamonds.

Luminescence imaging also illustrates the very diverse growth history of natural diamonds. In natural diamonds, the most important growth modes are octahedral and cuboid (Lang 1974). The contrast in emission may be spectacular (Fig. 5): cuboid growth sectors incorporate more nitrogen, hydrogen, and nickel than octahedral growth sectors; nickel–nitrogen defects present in such sectors—known as S2 and S3—cause a distinct green luminescence. A broad luminescence band in the orange to red spectral range (unknown identity) is responsible for the orange-pink luminescence of the octahedral growth sectors (Hainschwang et al. 2014).

While growth sectors in diamond can be efficiently visualised by luminescence imaging, post-growth processes and associated defects can be demonstrated by this technique as well. In diamond, this is mostly post-growth plastic deformation, which causes dislocations and vacancies (missing carbon atoms) in the diamond lattice. While in type Ia diamond, such defects manifest themselves as lamellae or bands parallel to the octahedral directions (e.g., Gaillou et al. 2010), in type II diamonds, the dislocations appear as very complex polygonal networks (Fig. 6; De Corte et al. 2006). Such dislocation networks contrast with their luminescent "matrix" that may appear in a range of colours such as blue, green, yellow, and very rarely orange (from the NV^0 centre, a nitrogen atom next to a carbon vacancy with a global neutral charge), as shown in Figure 6.

Further, thousands of PL spectroscopic features have been documented for natural and synthetic diamonds, of which only a small fraction has been assigned to specific defects (e.g., Zaitsev 2001). Photoluminescence emission spectroscopy is an extremely sensitive method in diamond analysis to detect activators even in extremely low concentrations (below ppm), especially when spectra are recorded with the sample kept at −196 °C by liquid-N_2 cooling. A range of excitation wavelengths may be needed to obtain sufficient data (Lindblom et al. 2003). This method has become routine in many laboratories, first to identify HPHT-treated diamonds (as discussed above) and nowadays more broadly to diagnose the nature and treatment of natural and synthetic diamonds of all colours. For the distinction of natural and synthetic diamonds, a few typical defects are expected, even if these defects are not exclusive for synthetic diamonds and may be found in some rare natural diamond as well: for synthetic diamonds grown by chemical vapour deposition, a sharp doublet is induced at about 737 nm by Si-V centres (a silicon ion next to a carbon vacancy), the Si coming presumably from the silica windows in the reactor. For diamonds grown by the HPHT method, a Ni-related doublet at about 883 and 885 nm can sometimes be seen, the nickel coming from the Fe–Ni metal catalyst often used to grow such synthetics.

Defect Identification

On the fundamental analysis of gem materials, the identification of activators is of great importance, as seen above with the case of synthetic diamonds. The simple observation of a luminescence colour may give an idea of the defect(s) involved, but in most cases, a PL emission spectrum is mandatory to confirm the identity of the defect(s). We present one simple example of how an unknown luminescence activator may be identified and attributed to a specific defect.

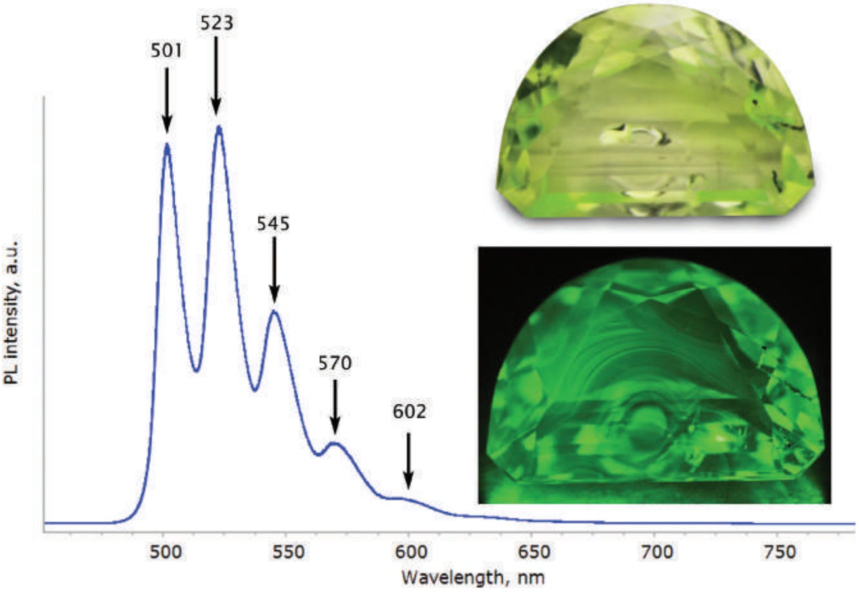

Figure 7 The green luminescence colour of this 0.82 ct hyalite opal is due to uranyl $[(UO_2)^{2+}]$ groups; the deep UV luminescence is contrasted enough to reveal the botryoidal nature of opal deposition (bottom right). The yellow body colour of the stone changes to yellow green under regular daylight because of its luminescence (top right). Accordingly, the classic strong green emission spectrum of the uranyl group (excited with 405 nm laser) is detected in this opal (left). Abbreviation: a.u. = arbitrary units.

Green luminescence in gems is not uncommon; it can be caused by a range of defects such as Mn^{2+} in a tetrahedral site found in genthelvite, willemite (Czaja et al. 2021), and spinel (Rodríguez-Mendoza et al. 1995), or H3 and H4 nitrogen vacancy defects, as well as S2/S3 nickel nitrogen defects in diamond (Zaitsev 2001), rare earth elements in fluorite (Czaja et al. 2012), and last but not least, uranium. The uranyl ion $(UO_2)^{2+}$ causes very bright green luminescence, stronger in shortwave UV in many gem minerals including adamite, aragonite, opal (e.g., Fritsch et al. 2015), and chalcedony. This luminescence is detectable at extremely low uranyl concentrations, well below those for which radioactivity is easily measurable.

The uranyl PL spectrum is very characteristic (Fig. 7), with several typical bands forming a vibronic structure between 500 and 600 nm, thus the uranyl activator is identified in this case with emission only. The width increases in disordered structures, but the positions of the main bands are always similar (Nasdala et al. 2022). In opal, very low uranyl concentrations cause luminescence that can make the material look green under indirect sunlight, and the luminescence under UV excitation attains intensities higher than most other luminescent gemstones.

CONCLUDING REMARKS

The luminescence of gems has evolved over the past quarter century from a basic observation-based approach with a standard UV lamp to a much more sophisticated approach with specific instruments for both microscopic observation and spectroscopy. Excitation now includes lamps with a broader spectral range (in particular in the UV), and often higher power, and also a variety of lasers. Camera-based imaging delivers a higher sensitivity to weak emissions. Considerable progress has been made with spectroscopy, although many useful gem emissions are still not understood fully. Luminescence-based methods do not replace older absorption-based techniques in any way, but they are invaluable additions in particular for current challenges, including testing of melee-sized gems and the separation of natural from synthetic diamonds.

The future of luminescence testing of gem materials will likely be directly linked to the development of cheaper and/or more powerful excitation light sources plus more sensitive detectors. Currently, particularly UV lasers are very expensive and LED excitation is limited to wavelengths of >360 nm; while short wave UV diodes exist, their output is not satisfactory compared with their high price. More powerful and economical short-wave lasers and LED sources would enable the development of more affordable instruments using such excitations. In parallel, a better understanding of the atomic nature of activators in gems is expected, supporting gemmological laboratories.

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