SCREENING FOR SYNTHETIC DIAMONDS: TECHNOLOGIES COMPARED

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Far from abandoning the top of the ranking of the most popular topics, the problem of identifying synthetic diamonds, especially in small sizes, remains constantly topical. Until a few years ago, producing colorless synthetic diamonds free of single nitrogen impurity was very complicated. Now the situation is changed. The HPHT method, the cheapest, was not effective on a large scale and the CVD was still uneconomical to grow the smaller material.

Since 2014, the situation has changed dramatically. Through the addition of "nitrogen getters" such as titanium, aluminum, zirconium and hafnium with the HPHT method it was possible to avoid the presence of single nitrogen which made the diamond yellow, furthermore, the cost of the CVD method has been significantly lowered. The consequence has been the appearance on the market of increasing quantities of colorless HPHT melee at prices well below the natural counterpart and, only very recently, also the availability of small CVD diamonds has started to be accessible at very competitive prices. Obviously the first reaction of the market was absolute panic. The problem does not lie in the ability of gemological laboratories to identify the material, the peculiarity of the issue here is intrinsic in the size of the diamond itself. Analyzing millions of small stones according to standard protocols in the laboratory is not only more complicated, but definitely uneconomical and impractical. Not to mention the diamonds mounted in the jewelry manufactured especially in emerging countries, what guarantees can buyers have that low costs do not derive also from the use of all or part of synthetic diamonds instead of natural ones? The challenge among gemological equipment producers was triggered immediately, but this time the problem was complicated by the fact that a substantial number of potential users had no gemological knowledge. Wholesalers, intermediaries, importers, especially jewelers, a whopping number of people without technical skills with the need to quickly and cheaply test loose and mounted diamonds, with only one requirement: natural or synthetic? In short, a series of required characteristics for the instrumentation has emerged: accuracy, ease of use (they must be able to be used even by unqualified personnel), the possibility of analyzing diamonds both loose and mounted, cost-effectiveness.

At this point we need to introduce a concept that does not seem to be clear to everyone. There is no apparatus based on a single technology ("economic" screening) that allows the separation between natural and synthetic diamond with a 100% accuracy. There will always be a percentage of diamonds, though paltry, that the machine will not be able to classify. This percentage is commonly called "referral rate" and, by definition should include, in addition to ALL synthetic diamonds, those rare natural diamonds that have characteristics that do not make them clearly identifiable as such. For this reason, the most the various devices on the market can all be classified as "screening systems". "Refer" is not a word chosen by chance. It means "refer for further testing" or better, the stone in question cannot be identified with certainty by this apparatus and needs further analysis through the use of other technologies. Once the screening has been carried out, any percentage of "refer" should be sent to the laboratory for further analysis. In any case, the diamonds that receive the "pass" from the screening units must all be natural, this is a requirement that should be considered essential.

Given the importance of the problem it is normal that a number of screening tools can be found on the market. There are different technologies to address the problem, however, very often the producers of these tools fail to mention the principles on which these are based and the reasons may be various. Some believe that the end user is not interested in the technicalities, others instead deliberately prefer not to provide details in order not to favor the competitors, in some cases the "industrial secret" is invoked.

It is our opinion that, although many users have no interest in having this information, the manufacturers should at least indicate the operating principle, this is because not all technologies lead to the same results. It is of vital importance for the user to always know the characteristics and limits of the instruments he uses to avoid incurring errors that can be, above all in this case, decidedly expensive. This article does not contain suggestions on the best technique, also because, regardless of effectiveness, the needs may vary (for example, loose and mounted diamonds) and some techniques lend themselves better than others. It is just a small compendium of the most used technologies that includes their more important pros and cons.

It should be pointed out that some manufacturers have preferred to focus on identifying specific and unique characteristics of natural diamonds and others on those of synthetic diamonds. The only doubt about the latter is that the characteristics of the synthetic can change with the evolution of the product and, perhaps, no longer be detectable by making the apparatus unusable in the future.

Transparency to short wave ultraviolet

This is the most "dated" method. Originally developed for the identification of IIa diamonds for the recognition of possible color enhancement by HPHT method, it has proved to be very efficient also for the screening of synthetic diamonds, as these are also type IIa. The operating principle is quite simple and takes advantage of the property that nitrogen-containing diamonds have to block the transmission of ultraviolet short-wave (SW) wavelength. If the diamond is nitrogen free it will be transparent to these waves, vice versa, if nitrogen is present they will be completely absorbed. The diamond is positioned between the short-wave source and a detector. If the diamond contains nitrogen, it will absorb the SW which will not reach the detector. If the diamond does not contain nitrogen (type II), the SW will reach the detector. There are several devices based on this principle, usually, given the simplicity of the technique they are the cheapest. This is a particularly common method among those who buy diamonds, mainly loose and of medium-large size on the secondary market.

PROS: They are the most economical and compact devices, identifying all IIa stones both synthetic and natural, even excluding the natural ones potentially treated for color enhancement by HPHT.

CONS: All natural IIa stones are "referred" and will need further analysis. It is not suitable for testing small stones or mounted jewelry, only in some models it is possible to test solitaire rings as long as you can place the diamond between the source and the detector and the metal of the mount does not interfere with the transmission of the beam. It is not suitable for lots, the stones must be tested one by one and it is not particularly fast. It cannot be used for simulants (Cubic Zirconia, Moissanite etc).

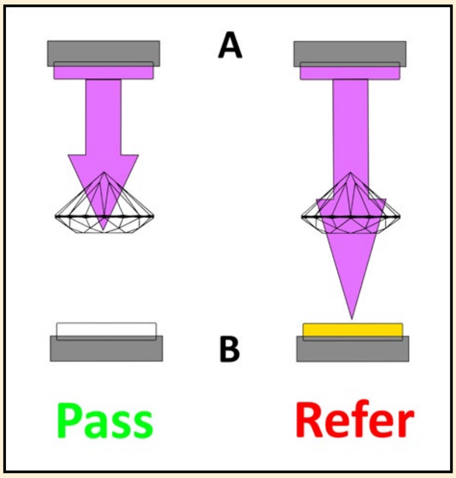

Fig. 2

Operation scheme of transparency to short-wave UV. The "A" lamp emits the beam in the direction of the diamond. If the stone absorbs it completely, it will be reported as a "Pass". If the beam passes through the diamond without being absorbed, the detector "B" will signal it as a "refer".

Visible spectroscopy

This is also a well tested method. It is based on the specific absorption in the visible spectrum caused by a defect in the atomic structure of the diamond, more precisely nitrogen aggregated called N3. It is present in the vast majority (approximately 97%) of natural diamonds and is absent in colorless synthetics as it is not possible for nitrogen to be aggregated in this form within the reduced time necessary for the growth of the synthetic, this is the reason why all the colorless synthetics are type IIa, no matter the production method. The defect causes an absorption peak in the visible area of the spectrum, centered at 415 nm. The apparatus must be equipped with a calibrated source with emission that includes the area to be highlighted (at least between 400 and 440 nm). The beam hits the diamond from which an absorption spectrum will subsequently be analyzed by a spectrometer. If this detects the presence of the N3 center, the diamond will be reported as natural, otherwise it will be reported as a "refer".

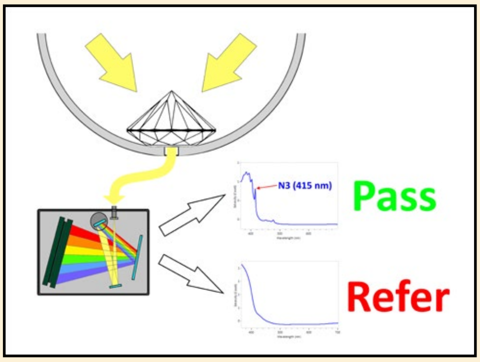

Fig. 3

Visible Spectroscopy. The diamond is hit by the light source and the resulting absorption spectrum is analyzed by the spectrometer. If the N3 center (415 nm) is detected, the instrument will signal "Pass", otherwise the diamond will be considered "refer".

PROS: It is effective and safe. The presence of the N3 center in a colorless diamond is an absolute guarantee of natural origin. It excludes all IIa stones, both synthetic and natural, including natural gases potentially treated to improve color using HPHT. Depending on the models it is also possible to test mounted stones but not small ones. It is effective even with simulants.

CONS: All natural IIa stones are "referred" and will need further analysis. It is not suitable for lots, the stones must be tested one by one and it is not particularly fast. Being equipped with a spectrometer, it is placed on a "high" price range.

Phosphorescence

When it began to be noticed that the vast majority of the synthetic melee HPHT emitted phosphorescence, a conspicuous number of instrumentation producers chose this characteristic as a technique for identifying the synthetic, the reasons are obvious. It would have been possible, by the simple use of an adequate source, to make a distinction between synthetic and natural on a large number of stones simultaneously, loose or mounted. Many of the tools that take advantage of this technique are equipped by an high-sensitivity camera that can also perceive a minimal reaction and a software that detects "suspicious" stones and spots them on a screen. The samples to be analyzed are inserted inside the machine. Once the procedure is activated, a UV source illuminates the samples for a set time of a few seconds. Then the source is automatically switched off, the camera takes the image which is then analyzed by the software. This is the preferred technology for those who have to analyze diamond mounted jewelery in large numbers and loose parcels of considerable size.

Fig. 4

Phosphorescence featured by current production HPHT synthetic diamonds.

PROS: It allows to analyze a large number of stones at a time, drastically reducing time. It is possible to test mounted stones.

CONS: Although rare, there are natural diamonds that exhibit phosphorescence. Not ALL synthetic diamonds are phosphorescent, most CVDs are not. It has been proved (The Journal of Gemmology, volume 36, No.3, 2018, pp 206-208) how, by irradiation, it is possible to "deactivate" the phosphorescence in HPHT synthetic diamonds thus rendering this technique ineffective. The prices of these instruments vary according to their use and the capacity of the sample compartment. Units designed for loose diamonds and small objects have mid-range prices while others that allow to analyze a significant number of pieces of jewelry at the same time may have high-end prices, it also depends on the brand.

Luminescence

It is a technique that is more evolved than that based on phosphorescence but, similarly, bases its effectiveness on peculiar features of the current production of synthetic material. When exposed to a specific light source, diamonds produce a reaction in the visible spectrum that can be used as a discriminant between the synthetic and the natural. This reaction must be "captured" by a sensor or camera and properly processed to provide the required result. The analytical process is very similar to that carried out in phosphorescence instruments. The samples are placed inside a compartment, once the procedure is activated the material is illuminated by the source and the detector catches the reaction. There are several instruments available based on this principle and prices can vary a lot depending on the sensor's technology. This can be a simple detector or a spectrometer, it goes without saying that the more sophisticated the technology, the greater the effectiveness of the instrument. Also in this case it is often possible to analyze more material at a time, depending on the capacity of the instrument.

Fig. 5

Luminescence emission of a colorless synthetic CVD diamond.

PROS: It allows to analyze a large number of stones at a time, drastically reducing time. It is possible to test mounted stones.

CONS: The efficiency of the instrument may vary depending on the detector technology. In some rare cases, CVD materials may not be identified as "refer". As in the case of phosphorescence, the luminescence emission of synthetic diamonds could vary with the future evolution of production, accelerating the obsolescence of the instrument. Sometimes not effective for simulants, it depends on the model. The price of these instruments also varies a lot, usually from the middle to the high end.

Fluorescence spectroscopy

It is similar to visible spectroscopy and its based on the recognition of the structural defect in the diamond caused by the N3 nitrogen aggregate. The difference lies in the source which, in this case is no longer in the visible but in the ultraviolet range. In this case the spectrometer measures the emission of the reaction, not the absorption. The effectiveness is better than visible spectroscopy also for the adoption of coaxial fiber optic probes. These concentrate the emission of the source in an area of just over a square millimeter generating a better spectral response from the diamond. A typical example of greater efficiency can be seen in the case of type IIa natural stones. The classification of nitrogen-free diamond (IIa) is made by FTIR which is credited with a sensitivity level of ppm (parts per million). However, it is quite common that nominally type IIa natural diamonds contain the N3 aggregate, even if in minimal part and are consequently identified as such by fluorescence spectroscopy. The explanation lies in the fact that even a few ppb (parts per billion) of these aggregates are sufficient to stimulate a reaction to high power ultraviolet emission. The wavelengths used for the source are commonly included between 365 and 380 nm to allow a safe use of the apparatus. The analysis is carried out by approaching the probe to the diamond, its reaction will be conveyed to the spectrometer by means of the same probe and, almost instantly, the instrument will provide the response.

Fig. 6

Fluorescence emitted by natural diamonds illuminated by a high power UV probe. The most common color is blue but it is also possible to observe reactions such yellow, yellow-green, pale whitish blue and pink.

PROS: This is a safe and probably long-lived technique given the objective impossibility of aggregating nitrogen in the N3 form in colorless synthetic diamonds. Given the number of natural IIa diamonds recognized as such, the total percentage of "refer" is reduced. It is quite fast (usually 1 second for identification) and it is possible to test mounted stones. Effective even with simulants.

CONS: The stones must be tested one by one even if the procedure is very fast. Being equipped with a spectrometer, these instruments are usually placed on a medium-high price range.

Fig. 7

Screening of mounted diamonds by fluorescence spectroscopy.

Some instruments then use several techniques combined together, for example phosphorescence and the presence of the N3 center. These are mostly isolated cases that do not fit the survey on small stones and parcels given the "slowness" of the analysis and usually have limitations for diamonds mounted in jewelry.

Conclusions

This is an overview of the most widespread technologies on which virtually all commercial screening tools are based. Certainly it is rather complex to orientate oneself for those who do not have specific gemological skills, above all because in some cases the producers themselves have caused confusion in the past. There have been cases of instruments for which the diamond that did not pass the test (refer), was directly reported as synthetic "HPHT/CVD", without taking into account that it could also be a particular natural type. The needs of users can be very different from each other, for example some are interested in testing only small assembled material, for others it may be prevalent to check single large stones, while others need units that can quickly analyze relevant parcels of small stones, sometimes very small. Instrumentation manufacturers are usually able to provide adequate suggestions for each individual use. However, it is our opinion that, without prejudice to the technical details concerning the legitimate trade secret, the producers should certainly inform the possible buyer about the technology underlying the instrument they propose. Last notation on the reported price ranges: the "low" range is between 500 and 3000 euro, the "medium" range up to 7-8000, the "high" one above 8000.

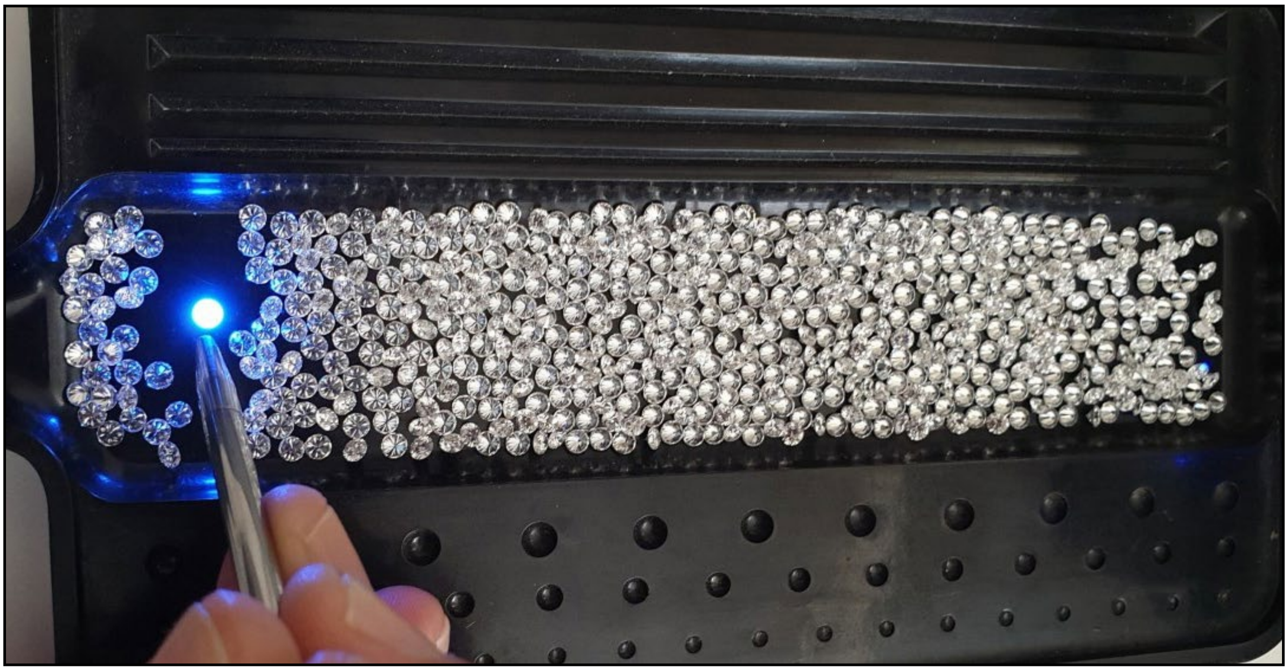

Fig. 8 Diamond parcel screened by fluorescence spectroscopy.

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