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GEM TESTING LABORATORY

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Lead- Glass Filled Rubies- An Overview

It was March 2004, when new type of filled rubies were first appeared and reported by the Gemmological Association of All Japan (GAAJ) laboratory; these were 'lead-glass' filled rubies. Later on, these rubies were analyzed and reported by number of laboratories all over the world; a short reported also appeared in L.I.C. Volume 40, March 2005. At that time no one had actual thought regarding the popularity and penetration of these filled rubies in the trade. But the time has publicized the importance of these rubies....

Initially, the treatment encountered on facetted stones only but with the time, now cabochons or beads or hollow backed rose cuts have also become very popular. This can very well judged by the range of stones submitted for identification at GTL (figure 1). In the recent times, we have received large numbers of bead strings of lead glass filled rubies in various sizes and cuts.

Figure 1

At the initial stages, these rubies were encountered only in limited numbers but nowadays these are widely available. In the period of August 2007 to January 2008 almost 40 % of rubies tested were identified as lead glass filled. Therefore, one can imagine the penetration of these rubies.

As per discussions with several trade members on the topic, they are happy to deal in this stuff but only on full disclosure. The correct identification is utmost necessary in order to protect the traders and the consumers. As per one member, "There is a huge price difference between lead-filled ruby and a heated ruby (with borax). If the price of a latter one is Rs. 100 then the price of lead-filled would be around Rs.10 or less, hence if a trader purchases a heated ruby (with borax) and turns out to be lead-filled will result in heavy losses". However, there are no reports of such incidences where one has suffered heavy losses, but this could happen any time if not dealt carefully.

Every new treatment / material when appear in the trade creates a lot of confusion and this was also not an exception. Much research and discussion have been conducted to understand this treatment, which now seems to be understood.

The starting material is opaque, highly cracked, brownish coloured corundum, which was not useful for gem purpose. Most of this material was used only as teaching samples or abrasives. Hence, the price of original material is very cheap. 'Lead-glass' filling remarkably changes the appearance of these rubies (figure 2).

Before After Before After

Figure 2

Most of the corundum used for this treatment is from East Africa, especially Madagascar. But, recently we have encountered some Burmese rubies filled with lead glass in addition to borax. As per an article by Vincent Pardieu, who interviewed the treatment experts in Bangkok, revealed that the treatment is performed in various steps.

1. Stones are preformed to eliminate matrix and other impurities.

2. The stones are "warmed" heated at temperature ranging from 900° to 1400°C to remove impurities from the fissures.

3. Stones are mixed with some oxide powder and heated. This powder is a mixture of silica and lead but calcium, potassium, sodium, bismuth may also be present. This powder on heating fuses and forms glass, which penetrate into fissures.

In some cases, the cavities / fissures are so large that they give impression of a composite material and glass component act as the fusing medium. As a result, AGL laboratories have started certifying these filled rubies as "Composite Ruby". Often, there are cases when glass portions are higher than the ruby areas.

As the identification is concerned, a proper use of a 10x lens and torch should enable to identify this type of filling. However, there are cases when the number of fissures is not much, creating difficulty in finding out the surface breaks only with a 10x loupe. In such case, the use of microscope with suitable illuminations becomes very important.

- Try to look for trapped/ flattened gas bubbles, which appear as bright patches in reflected light while transparent in transmitted light (figure 3.a, b & c). This is one of the important identifying features for this treatment.
- The surface breaks will exhibit strong colour flashes from violet to blue on changing the direction (figure 3.d); the effect is best visible under dark field illumination.

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- In addition, some white crystallites may be seen in cavities.
- One has to take care while observing the internal features of these filled rubies as these are heated generally at lower temperatures; some mineral inclusions remain undamaged like rutile needles and platelets which also exhibit strong interference colours as seen in unheated samples. Hence misidentification is possible if not observed carefully.
- Further, confirmation can be done using EDXRF, which exhibits a strong Pb peak.

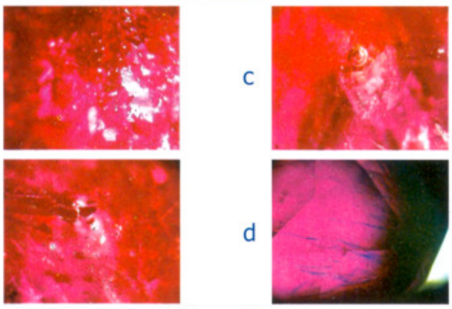


Figure 3

Another question surrounds these filled rubies is that whether these are durable or not and in order to answer this several durability tests were performed at GIA, as reported by S. F. McClure et al., "Identification and durability of lead glassfilled rubies," Spring 2006 Gems & Gemology, pp. 22-34. As per the results obtained on durability testing, these lead-glass filled rubies tend to damage when exposed to (i) high temperature heat applied during jewellery repair and (ii) solvents like ammonia, bleach or concentrated lemon juice. However, under normal wearing conditions, these filled rubies have good resistance against damages.

We have regularly encountered these filled rubies since it first appeared in the market and recently we have seen lead-glass filling in a colour-change sapphire which appeared brownish green (figure 4.a) in fluorescent light and brownish purple (figure 4.b) in incandescent light. Hence we would not be surprised to see this treatment in other colours of sapphires as well.

Figure 4

Due to the technological development in the treatments, these unsaleable rubies have flooded the market with saleable product creating more popularity of rubies. If disclosed properly and completely, now larger sections of consumers can have access and enjoy the pleasure of rubies!

Chaman Golecha resigns from GTL!!

Mr. Chaman Golecha has resigned from GTL after serving it for two years in December 2007. He joined GTL in December 2005 after completing MDGI and FGA. During the past two years, he was regularly involved in certification, educational and research activities at the GTL. He was very keen in learning and exploring various gem related subjects and as a result he became an important member of the GTL team. Further, his efforts in compiling articles that were published in various national and international journals were of a great support in showing GTL's presence at the international level in these two years. GTL wishes him all the very best for his future endeavours and hopes that he will make a valuable contribution to this trade.

A Crude method to test SWUV transparency

A helpful test for yellow-colourless-light coloured sapphires

Identification of sapphire usually does not pose any problems and can be easily accomplished by standard instruments. However the separation of natural from synthetic counterpart sometimes become challenging when light coloured clean sapphires (yellow to colourless) are encountered.

The separation between the natural and synthetic sapphires (especially light coloured - colourless/ yellow) is commonly done using magnification features. Presence of gas bubbles / unmelted powder along with 'plato lines' are observed in synthetics while inclusions like crystals, fluid inclusions, zoning features, parting planes, etc. in natural sapphires. However, often there are cases when these stones are free of inclusions and hence the separation becomes lot more challenging and time consuming

There is a need for a quick and effective method of separation of natural and synthetic yellow and colourless sapphires. One such method is SWUV transparency; this method has been known for quiet sometime, but not commonly used by student gemmologists and is considered to be a laboratory's instrument

What is SWUV Transparency?

SWUV transparency is different from UV fluorescence; certain materials are transparent to some specific kinds of rays while opaque to some. For example, flesh / muscles are transparent to X-rays while the bones are opaque. But the same is not true with visible light/eye; both appear opaque. Also, diamonds are transparent to X-rays but Synthetic Cubic Zirconia is not; but both are transparent to visible light. Hence, it is not necessary that the materials, which are transparent to visible light, are also transparent to all various types of rays. The same principle goes with SWUV. Some materials may be transparent to SWUV (like flame-fusion colourless synthetic sapphires) and some materials are opaque (eg. natural sapphires or other synthetics).

This method has been documented by Yu R.M. and Healy D (1980) "A Phosphoroscope", Journal of Gemmology, Vol 17, No. 4, p.250 and revisited by Elen. S & Fritsch. E. in "The Separation of Natural from Synthetic Colourless Sapphire", Gems and Gemology, spring 1999, p 30- 41. These articles have presented an instrument "Phosphoroscope" to check SWUV transparency of gems, which uses a 'phospor' plate as the base for stones and as the UV rays fall on this plate, it fluoresce strong white.

Instead of building a complex instrument or purchasing it, presented here is a crude way of checking SWUV transparency using a standard gemmological UV lamp coupled with cheap daily use scraps!!

What is required?

- A SWUV lamp (with proper viewing cabinet and accessories like protective glasses, gloves etc.)

Figure 5

- An ordinary cosmetic mirror
- A (broken) piece of fluorescent day light lamp (tube light) (Keep the white powdery material intact on the surface of the broken piece of lamp as it consists 'phosphor')

How to do the test?

- Place the UV lamp in a dark room preferably with proper viewing cabinet (else rest it such a way that the UV light falls below).
- Hold the piece of broken tube light such that the powdery side is at top. When switched on, the piece should glow due to the white powder.
- Place the stones table down on this glass piece with powder surface.
- Now place the mirror below such that one can view the reflection of the stones on the mirror.

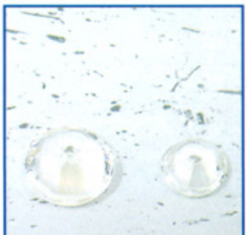
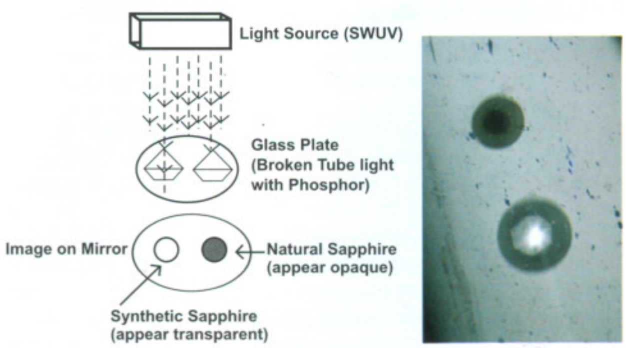


Figure 6

Result:

Samples that do not allow the UV radiation to pass through and hit the powder will appear opaque (figure 7.b, top image.), while samples which allow the rays to pass through them will appear transparent with the darker perimeter on the mirror (figure 7.b, bottom image). This difference depends on the amount of impurities present in the sample under test. Hence, a synthetic sapphire, which is a pure form of Al_2O_3 will appear transparent and a natural sapphire which consist of some impurities of Fe, Ti, or Ga will appear dark. Similarly, hydrothermal or flux stones containing higher percentage of impurities will appear dark and cannot be differentiated from natural counterparts by this method. In addition, if the flame fusion stones have undergone some sort of treatment the results become unusable. It is always advisable to compare any unknown material with the known natural or synthetic samples at the initial stages.



a b

Figure 7

While checking for light yellow/colourless sapphires:

- If a dark spot is observed: **Natural** or **Synthetic** (not very helpful)
- If a bright spot is observed: **Synthetic** (conclusion)

The point to remember is when a bright spot is observed, the sapphire is confirmed synthetic but when a dark spot is observed it may be natural or synthetic. Such a method of checking SWUV Transparency is very easy to construct, cheap and highly effective when used properly. However certain precautions are to be taken as follows:

- Take precautions not to expose unprotected skin or eye to SWUV radiations. Use protective eyewear and gloves.
- The broken piece of tube light may be sharp and dangerous. Be very careful and students are specifically advised to use, store and dispose off the pieces carefully.
- Highly included and opaque stones may provide wrong results.
- Always start with the known set of stones and compare the results with unknown samples. Do not skip the routine gemmological tests.

This method if applied properly and with care will prove to be a fast and effective method of confirming light coloured synthetic sapphires. If you have any problem in understanding the process or the principle, feel free to contact us or visit the laboratory with intimation to us.

GTL introduces new certification category....
"Certification of Bulk Jewellery"

Certification has now become an integral part of the gem and jewellery trade. This is due to the improved awareness regarding the gemstones and their certification amongst the jewellers as well as consumers. Now consumers are looking for a proof of authenticity with the jewellery they purchase so that they have confidence in what they are buying.

In this regard, we introduce a new category where you can get the stones certified when studded in Jewellery pieces at much cheaper and affordable fees. This category is, *"Certification of Bulk Jewellery"*.

- This category would be applicable only for jewellery pieces studded with at least five stones.
- In case of larger number of stones, if a depositor wishes to certify only a portion of stones in a jewellery item, testing will be done by randomly selecting the stones.
- *Certification Fees (in Rs.)*

	Member	Non-member
5 to 20 stones	75/ stone	100 / stone
above 20	50 / stone	75/ stone

- A single certificate will be issued for one Jewellery item
- Heating or Fracture filling will not be detected in this case
- Bead strings will not be considered in this category but a separate category already existing.
- Time taken will depend on the number of stones / Jewellery pieces and workload with GTL.

Two Synthetic Quartz ..

Synthetic Quartz in various colours (amethyst, citrine, rock crystal, green, etc) is available in various colours for many years now. However, identification of quartz can be done by observing 'bull's eye' optic figure but natural and synthetic separation is a challenging task for traders or even gemmologists, especially when these are 'free of inclusions'; often, separation is made on the basis of zoning patterns and IR spectroscopy. Recently, Mrs. Shyamala Fernandes gave us two interesting synthetic quartz specimens for study (figure 8. a & b).

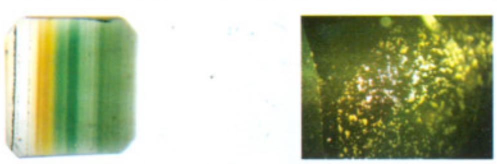
a b

Figure 8

One of the specimens was a multi-colour (figure 8.a), with bands of green and yellow as two main components, while the other one was green with patches of yellow colour (figure 8.b). The specimens were readily identified as Quartz on the basis of 'bull's eye' optic figure; further testes were performed for records. Refractive Index was measured at 1.540 1.548 with birefringence of 0.008 and hydrostatic SG at 2.64. Both specimens exhibited a strong degree of pleochroism with bluish green and greenish yellow as two colours; no spectrum was visible under desk model spectroscope and both were inert under ultra violet light. All these features were consistent for quartz, but did not indicate whether natural or synthetic

Magnification revealed the true nature of the specimens:

Specimen 1: The 'parti-coloured' specimen showed obvious colour zoning with green and yellow as dominant zones (figure 8.a), but on immersion, these zones became even more prominent and exhibited green (various shades- light & dark), yellow and colourless zones with sharp edges / boundaries (figure 9.a). In addition to the colour zones, seed plate with 'bread-crumb' inclusions was also seen (again figure 9.a, left corner of the stone), hence confirming the origin of the stone. Further, these whitish/ yellowish 'bread-crumb' like inclusions were also visible scattered throughout the stone (figure 9.b).

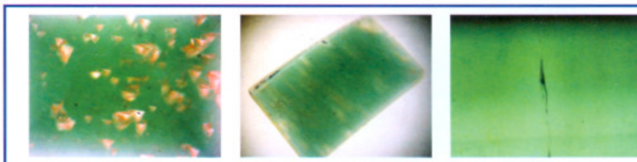


a b

Figure 9

These colour zones were oriented parallel to the seed plate while perpendicular to the 'optic axis'. This orientation of colour zoning is typically associated with those of synthetic quartz, as the crystal growth takes place along the 'seed plate'; in some directions, they appeared to be wavy.

Specimen 2: The face up view of green coloured specimen was even more interesting. The specimen exhibited yellow coloured patches when viewed through the table. On magnification, these colour patches appeared 'triangular' in shape (figure 10.a), which appeared as zones along the 'optic axis' direction; this followed the 'three-fold' symmetry of quartz crystal. At angles, these zones appeared wavy, similar to 'flame' like zoning (figure 10.b). In addition, the specimen also had 'spicules' running in one direction along the optic axis direction (figure 10.c).



a b c

Figure 10

FTIR analyses of both the specimens exhibited absorption till 4000cm^{-1}(figure 11), which is typically associated with synthetic quartz (citrine, green varieties). Natural quartz commonly displays absorption band/peaks in the region of 3000 to 4000 cm^{-1}.

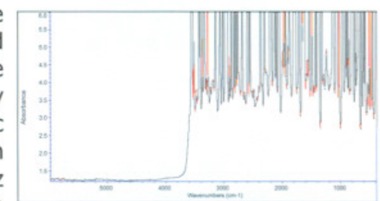


Figure 11

However, standard gemmological tests easily identified these synthetic specimens but lack of some characteristic features like 'breadcrumbs' or careful observations may lead to misidentification of these synthetic quartz.

The 'multi-coloured' specimen was said to be originated from Pakistan but here after testing and observing the features, we can only speculate that Pakistan is only a route of these synthetic quartz varieties through China or Russia.

Figure 12

An imitation 'Moldavite'

Moldavite- a variety of natural glass is known for its dull green colour is one of the most popular materials demanded in the world gem trade.

Recently, we have encountered some man-made glasses (figure 12), courtesy Mrs. Shyamala Fernandes, being sold as Moldavite. This man-made glass displayed appearance similar to that of rough piece of moldavite with wavy to 'spiky' to 'pimply' surface. Hydrostatic SG was measured at 2.50, which is slightly higher for a Moldavite; under UV light, the specimen appeared slightly yellowish along the ridges. Weak bands/ lines were observed at around 550, 590 and 680 nm under desk model spectroscope. Under magnification, spherical gas bubbles were seen as against elongated in moldavites. However, standard gemmological properties of this material are not conclusive enough to separate from moldavite hence, FTIR spectrum was used to differentiate between this man-made glass and a 'Moldavite'.

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