THE GEM & JEWELLERY EXPORT PROMOTION COUNCIL
Sponsored by Ministry of Commerce & Industry Govt. of India

GEM TESTING LABORATORY

Volume 56 — Lab Information Circular — December 2009

Ruby Treatments – where do we stand?

In the March 2008, Volume 50 of the Lab Information Circular (LIC) we overviewed the 'lead-glass' filled rubies due to the higher penetration of these treated materials in the trade. Since then, the type of treatment in these rubies has been modified to such an extent that exact identification and disclosure has become quite challenging even for a well-equipped laboratory. This is mainly because of the range of materials used as a filler substance especially when present inside the fractures or fissures.

As also discussed in the Volume 50 of LIC, the starting material is opaque to translucent, highly cracked and brownish coloured corundum which turns to near transparent red of ruby; such material mainly originate from Madagascar. However, with the discovery of some new deposits in the eastern part of Africa, especially in Mozambique, the nature of treatment also modified and as a result various different types of fillers are observed. Here, in this issue we shall discuss various types of fillers used and the problems associated in identification and disclosure.

The list of fillers used for rubies is quite exhaustive and therefore only few of them are mentioned below which are being encountered at GTL.

1. *Fluxes (e.g. Borax):* Various types of fluxes are being used as additives during the heat treatment processes; the most common one being borax which is being used with the heating process of Burmese rubies.
2. *Glasses (Silica, Lead, Bismuth based):* Silica glass has been used for filling of fractures since 1990's. This was followed by the introduction of lead-glass in 2004 and then Bismuth-glass recently.
3. *Combination of Fluxes and Glasses:* Flux substance like Borax along with the lead and / or bismuth is added while heating rubies. This enables the mixture powder to melt and penetrate into the fissures or any opening in the stone. In addition to lead or bismuth, tantalum oxide is another substance which has been detected in some of the stones.
4. *Colourless oil / resin:* Oil is also used as fillers in rubies but this is of not so much concern as this does not affect the appearance of the stones to a great extent and is usually acceptable. However, resin filling is also performed but not seen frequently.
5. *Coloured Oil (Jhoban):* The oil coloured by organic or inorganic dyes have been used to enhance the colour of rubies and other stones for several decades. However, in the recent past coloured oil is observed only in few stones.
6. *Inorganic dyes:* This is mainly used on white to light coloured, translucent to opaque corundum turning them red and other colours.

Out of the above mentioned materials, fluxes and glasses and the areas of major concern. This is due to the fact that a number of different types of fluxes and associated elements are added during the heat treatment process. Some of them form glass like compounds and the stones thus are described as 'glass filled'. In addition, aluminium oxide is also said to be used as a filler material but there is a question mark on this since aluminium oxide will not melt and penetrate in the fissures at lower temperatures at which heating is usually done.

The use of wide range of fillers has created a lot of confusion amongst the trade members as to correctly classify the material. Exact identification of the nature of filler material is also important with respect to the cost of the stone. A ruby when filled only with flux (like borax) usually fetches a good price as it is widely accepted as a byproduct of heating procedure. However, if lead or bismuth glass is observed, the prices fall down considerably and the visual appearance of both these treated rubies is similar; main reason behind this is the huge difference in the price of the starting materials.

Because of wide range of materials used as fillers, exact identification and disclosure becomes very difficult and much challenging. Identification of various fillers at GTL mainly involve the microscopic examination and chemical analyses (using EDXRF); in some laboratories x-radiography is also used to detect the presence of lead and/or bismuth glass. Few of the characteristic features observed in fracture filled rubies:

Figure 1. a

Figure 1.a: Film and flow patterns forming a net like pattern along with bubbles in Flux- filled ruby

continued to page 2...

continued from page 1...

Figure 1.b: Sheet and mirror like re12 filled ruby

Figure 1. b

Figure 1.c: Presence of gas bubbles (left), patchy reflections (centre) and blue flashes (right) in lead / bismuth glass filled rubies

Figure 1. c

Figure 1.d: Presence of flow structures forming a net like pattern (left) and colour flashes (right) in a ruby filled with flux as well as lead / bismuth glass

Figure 1. d

Figure 1.e: Presence of colourless oil will display a characteristic dendritic pattern (left) and iridescence (right)

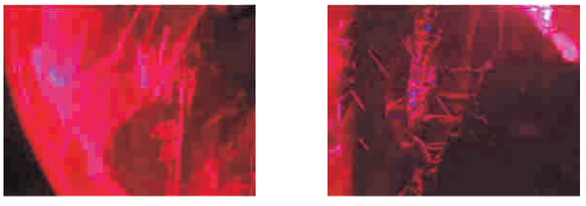

Figure 1. e

Figure 1.f: Colour concentrations along the surface breaks like fractures and / or twin planes in a ruby / corundum filled with coloured substances like jhoban or inorganic dyes

Conclusive identification of presence or absence of lead and / or bismuth is done with the help of chemical analyses using EDXRF. This also helps to determine whether some other element is present or not. Characteristic peaks are observed for individual elements.

Figure 1. f

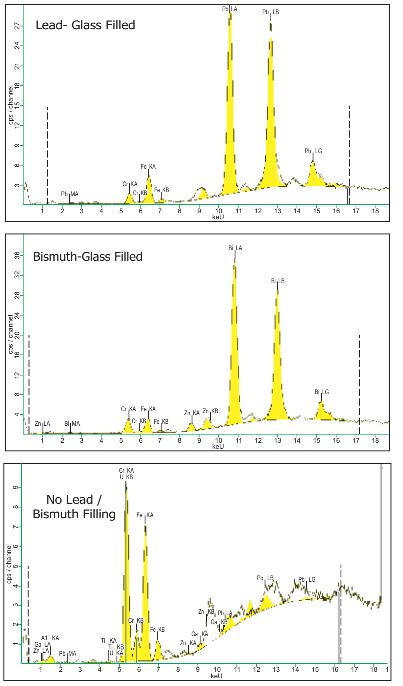

Figure 1. g

The separation is quite distinct between the glasses and fluxes as carried out by magnification features and EDXRF analyses described above. But the fact is various heavier elements are being used along with borax which penetrate into the fissures and act as a high refractive index substance; this improves the appearance of the stones drastically similar to the lead and/ or bismuth glass filling. Identification of these various elements is possible with EDXRF, but the problem lies in the disclosure.

The disclosure of any form of treatment is made to explain the consumer or a trader what has been done to the stone. This can only be done when the language is simple and short and self explanatory. Therefore, each time a new element is detected in the fissure cannot be mentioned on the identification reports. A laboratory has to maintain simpler and uniform wordings while disclosing a treatment. For example: if a ruby is identified as heated where some flux material is observed (irrespective of the exact nature), GTL mentions the following on identification reports: "Indications of heat treatment. Presence of foreign substance (e.g. Borax) in fissures / voids".

On similar grounds, the American Gemological Laboratory (AGL) has started to mention "foreign residue" in place of "flux-type treatment". This neutrality in the identification reports is essential in order to avoid the confusion, because each time the trader comes across the presence of a new element in his rubies, the worry and confusion will only increase. The same applies to the glass filled rubies where each and every element cannot be added in the report wordings.

Another major problem associated with these treated rubies is the extent of filling. In some cases, the amount of glass is more than that of ruby. In such cases, the glass is not only present in the fissures of ruby but act as a binding agent for several smaller pieces of rubies. This has given rise to a debate all over the world gemmological laboratories and trade associations to correctly classify these treated rubies. This is also the most debatable point on many internet discussion forums.

In this case, small pieces of rubies are taken and joined together with the help of glassy substance to form a single piece, but it is not durable enough. On acid wash or after a use of few months these filled areas tend to crack revealing the true nature of rubies. In acid wash for longer periods of time, the glass disintegrates and looses the bonding with ruby pieces. A similar discussion is on new rubies originating from Mozambique. The starting material is highly cracked and crumbles in fingers; this material is said to be heated with fluxes where the ruby becomes intact and much resistant to knocks.

In these two matters, many individuals have started to designate these rubies as "Composite ruby" or "Reconstituted ruby" rather than glass filled. The American Gemological Laboratory (AGL) has already certifying these strongly filled rubies, "Composite Ruby". Some are in the opinion of calling these as synthetic overgrowth as individual pieces are joined together either with the help of glass or flux. However as per one of the treaters (anonymous), "the treatment of these Mozambique rubies is similar to those for Mong Hsu rubies started to appear in early 1990's".

Traders often associate the type of filling with the source origin of stones. As per the discussions with many traders in Jaipur, lead-glass filling is commonly done on Madagascar stone while flux (borax) on other sources like Longido or Burmese. But, in the experience of the laboratory, lead-filling is done on stones from all various locations, even Burma. Therefore, if a ruby is said to be Burmese, it always has a possibility of being lead- glass filled.

As the disclosure is concerned, majority of labs are still calling these treated rubies as 'glass-filled rubies', 'treated rubies' or 'natural rubies' with comment on glass filling with extent of filler. However, Laboratory Manual Harmonization Committee (LMHC) and CIBJO are still to take a decision whether to call these heavily filled rubies "Composite".

"Smoky" gray beryl

Recently, we examined a 71.57 ct step-cut stone in figure 2.a. It had a gray color with moderate saturation and slightly brownish gray reflections near the corners; one of the corners showed relatively stronger reflections. When it was tilted in standard lighting against a white background, subtle zones of pale brown color were observed at some angles.

Figure 2. a

The color appearance and brown zones were reminiscent of smoky quartz. Although the specimen displayed a uniaxial optic figure, it did not show the characteristic "bull's-eye" pattern of quartz. This did not rule out quartz, but did raise sufficient doubt to warrant further testing. The results were surprising: The refractive indices were 1.590–1.598, with birefringence of 0.008, values that are consistent with beryl. Although beryl occurs in a variety of colors—green, blue, red, pink, yellow, orange, brown, and colorless are all known—gray is quite unusual.

The stone had a hydrostatic SG of 2.81, which is high for aquamarine but low for pink beryl (e.g., 2.66–2.80 and 2.80–2.90, respectively; see M. O'Donoghue, Ed., Gems, 6th ed., Butterworth-Heinemann, Oxford, UK, 2006, pp. 163–164). No absorption features were visible with the desk-model spectroscope, and the sample was inert to long- and short-wave UV radiation. It displayed weak gray and pinkish brown dichroism (figure 2.b). No features were observed with the microscope, other than some angular and planar growth zones.

Figure 2. b

FTIR spectra were typical for natural beryl, while qualitative EDXRF analysis revealed the presence of Al, Si (major) Ca, Mn, Fe (traces), and Cs (minor). The presence of Cs would explain the relatively high specific gravity compared to aquamarine.

The cause of color in this unusual specimen remains unknown. John Sinkankas's Emerald and Other Beryls (Chilton Book Co., Radnor, Pennsylvania, 1981) noted that a beryl that had been previously heated in oxidizing conditions turned deep gray when subsequently heated in reducing conditions, although the cause of color was not determined.

This write up was first appeared in the Gems & Gemology, Fall 2008, pp 271-272

An interesting sapphire crystal from Winza, Tanzania

This write up was first appeared in the Gems & Gemology, Fall 2008, pp 271-272

Recently discovered rubies from Winza in central Tanzania have gained popularity for their bright red color and transparency (Gems & Gemology Summer 2008 Gem News International, pp. 177–179). Sapphires have also been reported from this deposit (V. Pardieu and D. Schwarz, "Field report from Winza," Rapaport, Vol. 31, No. 26, 2008, pp. 173–175), but they have not received as much attention.

Figure 3

At the Gem Testing Laboratory in Jaipur, we had an opportunity to characterize a 2.82 ct sapphire crystal (figure 3) that was brought to our attention by Mrs. Shyamala Fernandes. She obtained the crystal from Jacob Hoyer of Italy, who purchased it directly from a Winza miner.

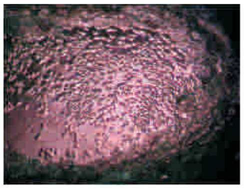

Figure 4

The crystal's appearance immediately pointed to sapphire due to its characteristic pyramidal habit, which was terminated by a pinacoidal face; it also exhibited faint horizontal striations along the pyramidal faces, in addition to tiny hexagonal to sub-hexagonal growth hillocks (figure 4) on the pinacoid that were observed only at high magnification.

The most interesting feature of the crystal was its color zoning. The area nearest the pinacoid was purple-pink, which gradually shifted to colorless and then blue (again, see figure 3). The latter color increased from light to deep blue to almost black toward the lower end of the crystal, making it appear opaque. When magnified, this color distribution seemed to be restricted to zones with sharp edges that mostly followed the pyramidal directions within the crystal (figure 5, left and center). In addition, the purple-pink area displayed zones of alternating saturation along with some blue zones that were oriented parallel to the pinacoidal face (figure 5, right, also illustrated in M. S. Krzemnicki and H. A. Hänni, "New Tanzania mine uncovers source of exceptional rubies," InColor, Spring 2008, pp. 46–47).

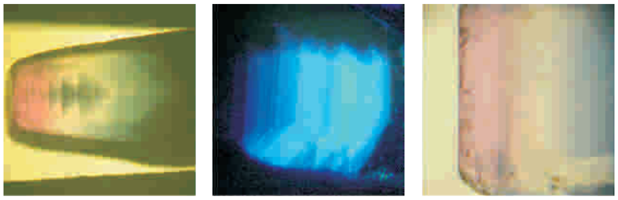

Figure 5

When the crystal was viewed along the c-axis, we observed a transparent purple-pink core that was surrounded by a dark blue to black rim (figure 6). This effect reminded us of Mong Hsu rubies where the central core is dark blue and the outer rim red; in this crystal, the zoning was reversed. However, we could not determine whether this core was colorless and the purple-pink color visible because of the zone at the tip of the crystal, or if the core itself was purple-pink.

Figure 6

No mineral or fluid inclusions were seen due to the dark color of most of the crystal; only the color zones described above were seen where it was relatively transparent.

Since its discovery, the Winza deposit has produced some fine rubies, along with interesting specimens such as this sapphire. With further exploration, a wider range of material may be expected.

Created and Edited by : Gagan Choudhary, *Asst. Director (Tech. & Training)*

Contact for further details : Meenu Brijesh Vyas, *Asst. Director (Tech. & Training)*

Radhamani Amma, *Asst. (Coordination & Info.)*

Kailash Chand Daurata, *Ju. Executive*

GEM TESTING LABORATORY
Rajasthan Chamber Bhawan
M.I. Road, Jaipur, India
Phone : 91-141-2568221, 2573565
Fax : 91-141-2567921
Email : gtl@gjepcindia.com
Website : www.gtljaipur.info