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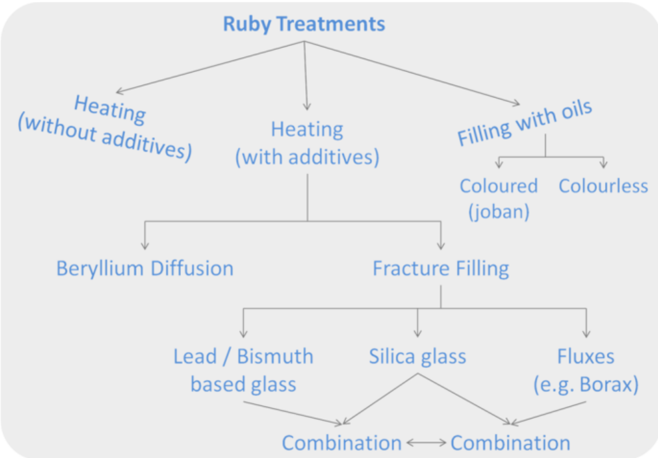
Ruby Treatments - revisited, with Mozambique's perspective

Since the discovery of ruby deposits in Mozambique, especially in the Montepuez area in early 2009, the market is being flooded with these rubies; samples from other deposits are encountered only occasionally. Initially, quite a few unheated samples were seen, but then suddenly huge amounts of treated rubies made their way to the markets. These rubies were heated with fluxes. In December 2009, Volume 56 of the Lab Information Circular, we overviewed various fillers being used in rubies to enhance the clarity and / or colour because of the recent encounters of these rubies. However, today after two years the penetration and quantity of these rubies is much more significant than ever anticipated.



Dyeing white corundum into red is not considered here

Figure1: An overview of currently prevailing ruby treatments

Today, the main concern regarding these rubies is the nature and type of treatments performed. In basic terms, the treatment performed is similar to that have been observed in Mong Hsu (Burma/ Myanmar) rubies for more than a decade since 1990s. These are heated with fluxes, where the flux compound enters into the fissures or fractures of rubies, heals them up and thereby increasing apparent clarity.

Figure 2: The appearance of these Mozambique rubies are improved by heating and flux-assisted healing of fractures.

Figure 1 overviews various treatments applied on rubies in order to enhance the colour and/ or clarity. These have been broadly classified as heating without additives, heating with additives and filling with oils (coloured or colourless). Heating without additives implies, no foreign substance has been added into the stone, while heating with additives implies, foreign substances like fluxes, glasses or colour causing impurities including beryllium have been added into the stone's structure or fissures along with heat.

In the past many months, we at the Gem Testing Laboratory (GTL) observed that these rubies not only contain flux healed fractures, but also glass filled cavities / fissures / fractures. Although the glass filled cavities does not contain significant lead, but glass is a glass. And the intention behind the treatment is same, while the effects and/or results are similar. Hence, in many cases, a single specimen qualifies for two different types of treatments - heated, with flux and glass filling.

Therefore, it is our attempt to produce a simple and basic classification system for describing various treatments on rubies. This becomes even important because of the presence of glass-filled rubies from Madagascar. Although, there is a significant price difference between a glass-filled ruby (Madagascar) and a ruby heated with flux (Mozambique), both may be qualified for the similar treatment type.

Same applies to Longido (Kenya / Tanzania) rubies too.

Figure 3: Rubies from Mozambique (heated with flux)-left and Madagascar (lead-glass filled)-right.

Glass filling vs. Flux-healed fractures - the mechanism

In case of flux healing, rubies are heated at high temperatures of around 1800 - 1900^{o}C in the presence of flux, e.g. borax. The surface of ruby in contact with the flux, at such high temperature, melts and dissolves. On gradual cooling, the alumina of ruby starts to crystallize on the surfaces or within the fissures / fractures of the ruby. This formation of synthetic corundum gradually seals or partially heals up the fissures, thereby improving the overall clarity. On the other hand, glass filling involves the injection of molten glass into the fissures or fractures without healing taking place. As a result, flux-healed fractures are stable while glass-filled not. In case of Mozambique rubies, along with the borax, high amount of silica is also added while heating. This results in the formation of glass in fractures and hence is found present in fissures.

Quite often, these rubies after treatment are washed with hydrofluoric acid, which reacts with silica and dissolves it out from the stones, but not completely from some larger fissures or cavities.

In some cases, these rubies undergo combination of treatments. The stones which do not show significant improvement after heating with borax are then filled with lead-based glass. As a result, a single stone may contain heating residues (flux / borax), silica glass and lead glass. This now mainly depends on the number and size of the fissures, the larger the fissures, higher the chances of it being filled with silica or lead based glasses. Further, some of the Mozambique rubies are filled only with lead glass. In such case, technically and /or logically there is no difference between a Mozambique and Madagascar ruby. Both will be disclosed in the same way with same terminology. This then raises a concern amongst the trade, as there is a substantial price difference between the two ruby types.

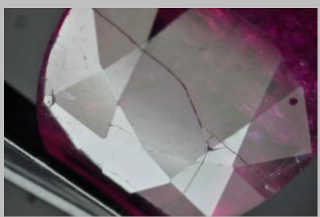
Trade often associates the type of filling with the source origin of stones. As per discussion with many of the trade members from Jaipur, lead-glass filling is done on Madagascar stones while heating with flux on other sources such as Longido, Mozambique or Burmese. However, in our experience looking at thousands of rubies in the last five years, lead glass filling has been observed on other sources too, whether it is Longido, Burma or Mozambique. For a note, we have also seen Indian rubies filled with lead-glass.

A brief description on the features associated with the types of filling in rubies is given in Box A.

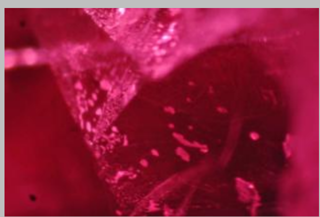
No matter what the source is, one has to understand and accept that glass filling can be done on any ruby from any source. It just cannot be anticipated that if a ruby is from Mozambique or Longido or even Burma, it cannot be glass-filled. Therefore, every ruby should be checked for the presence of lead-glass before giving any assurance. We have seen numerous cases here in the laboratory, when the seller has assured the buyer that a said ruby is not glass-filled, it turns out to be a glass-filled on testing. And this no doubt, affects the goodwill and reputation of the seller adversely.

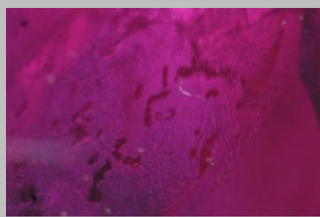
In addition to fracture filling, rubies are also subjected to beryllium diffusion, which is not a major concern at this stage. This treatment is more common for yellow and blue sapphires. Hence, the subject is not covered in detail here and may warrant detailed explanation in the future. Oiling is quite a common feature but is seen in unheated stones and is usually accepted by the trade.

Box A: Features observed in various types of filling in rubies

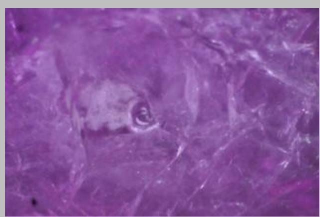

a. Large surface breaks are common features in these filled rubies


b. Surface breaks typically displays flow and net like patterns


c. Some surface breaks display patchy reflections due to trapped/ flattened gas bubbles


d. Trapped / flattened gas bubbles appear opaque in transmitted light

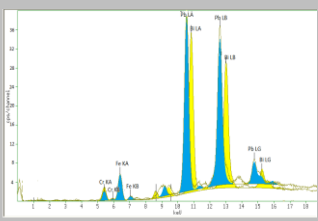

e. Many of these Mozambique rubies have cavities filled with glass, which display duller lustre

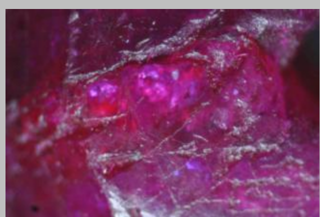

f. Glass-filled cavities often display gas bubbles

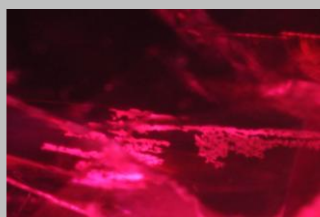

g. Gas bubbles are also a common feature in Lead-glass filled rubies from Madagascar


h. Opaque and reflecting patches / flattened gas bubbles are also seen in lead-glass filled rubies


i. Colour flashes are typically seen in rubies filled with lead / bismuth based glasses


j. Lead (blue line) and Bismuth (yellow line) can be separated by EDXRF analysis


k. Rubies filled with coloured oil (joban) is identified by colour concentration along fractures


l. Colourless oil display dendritic patches along with iridescence

Further, coloured oil (joban) is also used to improve the overall colour and surface lustre but currently, these are seen only occasionally.

In order to provide clear understanding of these ruby treatments, the Gem Testing Laboratory Jaipur uses simple and unambiguous criteria and disclosure policy on its identification reports. This is given in the following table.

Member laboratories of the Laboratory Manual Harmonisation Committee (LMHC) like GIA also disclose these treated rubies with the similar terminology.

Some care has to be taken while using these rubies in jewellery. As the fractures are healed up using a flux and glass, these are prone to damage in high heat and some chemicals.

Conditions	Identification / Report Wordings
A ruby when do not exhibit any signs of heat treatment.	Natural Ruby No indications of clarity enhancement
A ruby when display features of heating.	Natural Ruby Indications of thermal enhancement
A ruby when do not exhibit signs of heating but colourless oil is identified as the filler in fractures.	Natural Ruby No indications of thermal enhancement. Oil in fractures identified which is a common trade practice
Ruby displaying features of heating and presence of foreign residues like flux (borax) in fractures.	Natural Ruby Thermal enhancement and heating residues in (e.g. borax) in fractures
Ruby which displays the features of glass filling using lead, bismuth or silica (subject to identification using EDXRF)	Natural Ruby (Glass-Filled) Thermal enhancement and presence of lead/ bismuth / silica based glass in fractures / cavities
Ruby displaying the features of glass filling using lead, bismuth or silica (subject to identification using EDXRF) along with foreign residues (like borax) in fractures.	Natural Ruby Thermal enhancement and heating residues (e.g. borax) and (lead / bismuth/ silica) based glass in fractures
Ruby displaying features of heating and presence of foreign residues like flux (borax) in fractures and surface cavities filled with a glass (lead / bismuth/ silica).	Natural Ruby Thermal enhancement and heating residues (e.g. borax) in fractures. In addition, glass filled cavities seen
When coloured oil (joban) is identified as filler in fractures.	Natural Ruby Colour enhanced with red colour in fractures
When a ruby displays signs of (inclusions / FTIR spectra) beryllium diffusion.	Natural Ruby Thermal enhancement and indications of diffusion of chemicals (e.g. beryllium) from an external source
When distinct features of beryllium diffusion are seen in ruby.	Natural Ruby (Artificially Coloured) Thermal enhancement and diffusion of chemicals (e.g. beryllium) from an external source

First ever International Colourstone Conference "Mines to Market 2011" ended on a high note

The Gem & Jewellery Export Promotion Council (GJEPC) organised the first ever International Colourstone Conference "Mines to Market 2011" on 2nd and 3rd of November. An overwhelming 350+ participants wherein all the stakeholders of the coloured gemstone industry consisting of the miners, retailers, designers and gemologists were brought together for the very first time in India to witness two exorbitant days discussing the journey of a gem right from their formation to mining, supply chains, their cutting & polishing, their use in jewellery and marketing & branding of these products.....their rarity, legacy, romance and much more. The industry leaders from various sectors of the gem & jewellery pipeline have shown their presence and gave an insight towards development and promotion of the coloured stone trade. The list of prominent speakers for the conference included Alberto Milani (Keynote), Edward Boehm, Richard Hughes, Federico Barlochar, Dr. Federico Pezzotta, Peter Lyckberg, Brian Cook, John Saul, Mark Saul, Ian Harebottle, Gavin Pierce, Shigeru Akamatsu, George Shen, Dr. Lixin Yang, Robert Weldon, Helmut Zimmermann, Andrew Lucas, Louis Bell, Shaltiel Cohen, Glenn Lehrer, Steve Bennett, Wong Chi Ming, Dr. Chuck Lein, Hamilton South and Candy Price. Further details and proceedings of the conference can be seen at m2m.gjepc.org.

Pink cat's-eye quartz, with colour and chatoyancy caused by tourmaline needles

Chatoyant quartz is widely available in gray, yellow, and green colours, and it is often misrepresented as chatoyant chrysoberyl. Recently, we examined a pink cat's-eye quartz that proved interesting because of the unusual cause of the chatoyancy.

The 34.65 ct specimen (figure 4) displayed a broad but distinct chatoyant band with a dull vitreous luster. At first glance, it was reminiscent of chatoyant tourmaline because of its colour. Closer examination with the unaided eye showed colour concentrations in various areas, especially towards the sides. This suggested the presence of a dye along the growth tubes/surface breaks. The specimen appeared anisotropic under crossed-polarizers, with some interference colours perpendicular to the chatoyant band or along the direction of the needles. This suggested a uniaxial mineral, although a clear optic figure could not be resolved due to the dense inclusions. Spot RI and hydrostatic SG were measured at approximately 1.54 and 2.68, respectively. The sample displayed no reaction to UV radiation, and no features were seen with the desk-model spectroscope. These properties indicated quartz, but further analysis was required.

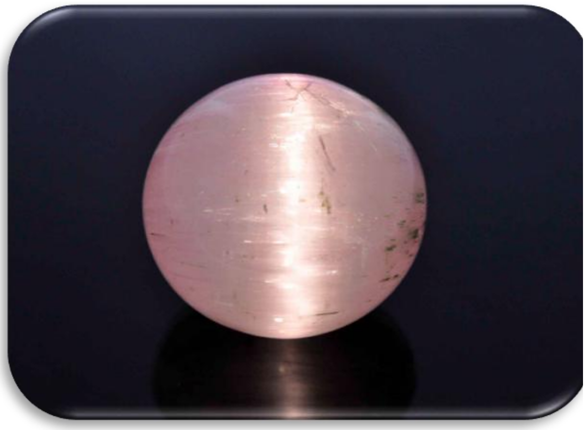


Figure 4

With magnification, the sample displayed long tube or needle-like inclusions (figure 5). From the side, some pink colour was observed along these tubes, which again raised suspicion regarding the cause of colour. Cross-section of the tubes was even darker. At higher magnification, some appeared perfectly triangular (figure 6), a feature typically associated with trigonal minerals such as tourmaline. This was further supported by the colour of the tubes and the absorption along the 'c' axis: the inclusions appeared darker when viewed in cross-section. Hence, the body colour of the sample was colourless but appeared pink due to the coloured inclusions.

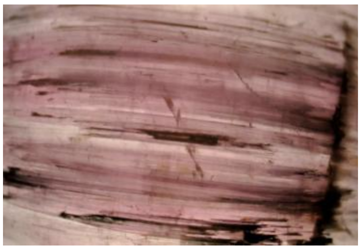


Figure 5

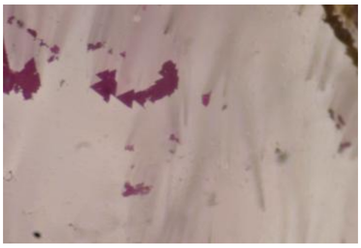


Figure 6

IR transmission spectra were obtained both parallel and perpendicular to the needles. In the parallel direction, two sets of distinct peaks were seen: in the 4800–4200 cm^{-1} region (4594, 4534, 4438, and 4343 cm^{-1}) and in the 3700–3000 cm^{-1} region (3563, 3585, 3480, 3379, 3300, and 3197 cm^{-1}). According to our database and past studies (see, e.g., L. T. M. Oanh et al., "Classification of natural tourmalines using near-infrared absorption spectroscopy," *VNU Journal of Science: Mathematics – Physics*, Vol. 26, 2010, pp. 207–212; G. Choudhary and S. Fernandes, "Spectroscopic examination of commercially available quartz varieties: A gemological perspective," Summer 2011 *G&G*, pp. 146–147), the first set of peaks is similar to those seen in tourmaline, while the second corresponds to those seen in quartz. In the perpendicular direction, only a broad absorption band was observed in the 3700–3000 cm^{-1} region, due to the lower degree of transmission in that orientation.

On the basis of microscopic examination and the infrared spectra, we identified this sample as quartz with inclusions of pink tourmaline. Tourmaline is a common mineral inclusion in quartz, but it is mostly randomly oriented. Thus, this sample was interesting and unusual because of its pink colour (though it was not rose quartz) and chatoyancy. Both were caused by the presence of parallel tourmaline needles.

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