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Identifying Treatments!!

This note is based on the presentation given by Gagan Choudhary, Asst. Director at the workshop on "Identifying Treatments" organized by Gem Testing Laboratory, Jaipur on 19th of April 2007.

Treatment refers to any type of modification done on a gem material other than cutting and polishing. In the current gem trade, almost every gem material is being treated in one or the other manner. Some of the common types include Bleaching, Coating, Foiling, Spraying, Impregnation (Fracture Filling - Coloured / Colourless, Filling of Pores - Coloured / Colourless), Heating (Without and With Additives - Diffusion Treatment), HPHT, Irradiation, Lasering, etc.

Out of many gem materials the most controversial ones are those performed on Corundum and Diamonds, as some of these are quite difficult or impossible to detect by classical testing and affect the price to great extent. One has to use sophisticated and expensive advanced techniques.

Here, the emphasis is being given on treatments performed on corundum, as this is the most common gem material being encountered in local market. Certain common treatments performed on corundum include:

- **Heating**
- **Diffusion** (Surface / Beryllium)
- **Glass Filling** (Silica/ Borax/ Lead Glass)

Heating....

Heating is done to modify (change / enhance) the colour and/ or clarity (remove inclusions/ add inclusions). The stones are exposed to a high temperature varying from 1000°C to 1800°C with or without oxygen for few hours to several. The changes after treatment in corundum are variable from specimen to specimen depending on the following:

- Source origin of the stone
- Atmosphere (Oxidising or Reducing)
- Presence of Chemical Impurities & Inclusions
- Rate of heating and cooling
- Maximum temperature reached and maintained

Following are the examples of few of the changes after heating.

Figure 1

a. Srilankan Sapphire; before (left) & after (right)

b. Kenya Rubies; before (top) & after (below)

c. Kenya Rubies; before (left) & after (right)

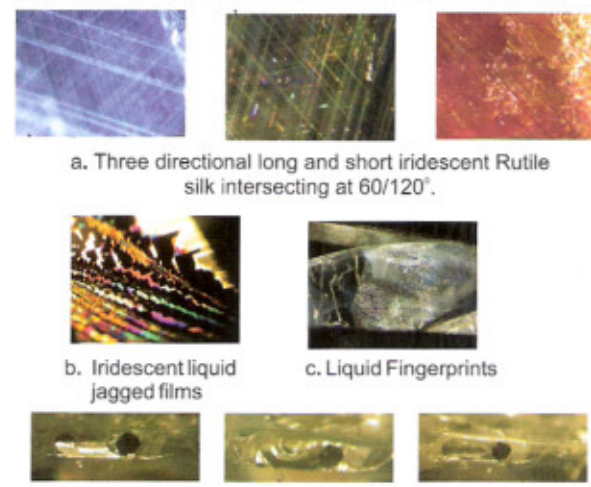
1 2 3

d. Some Srilankan Sapphires are not stable and may change the colour as in case 1 (before treatment) changes to case 2 and then looses the colour on exposure to light and heat as in case 3

Identification.... of the treated nature of corundum involves the observation of internal features like the pattern of rutile silk, discs, liquid inclusions, crystals, fingerprints, etc.

Figure 2

Few of the Features indicating Untreated Nature...



a. Three directional long and short iridescent Rutile silk intersecting at 60/120°.

b. Iridescent liquid jagged films

c. Liquid Fingerprints

d. Negative crystal with CO_2 inclusion, where the position of bubble changes as the stone is tilted. On gentle heating of stone under microscope, the size of bubble reduces and finally disappears. This is one of the features that conclude the stone being not exposed to high temperature.

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When stone is exposed to high temperature heat, the direct effect is on the internal features which help in its detection. Some of the features are affected by the temperature applied while some still escape through, which then creates confusion in the identification. Some of the features helping in identification of heat include.....

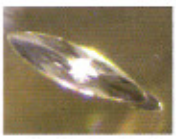
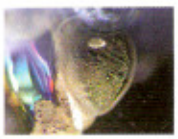
Figure 3

Indications of Heating.....



a. Three directional Rutile silk when exposed to high temperature, melts and beads up while some forms diffused zones.

b. Liquid fingerprints dry up giving cloudy pattern.

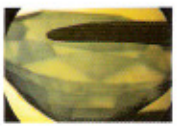
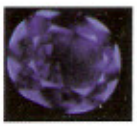

c. Crystal inclusions often form a glassy stress with high reflection or white droplets and edges or white stress around them.

Heating with Additives...*Diffusion*

Diffusion treatment involves introduction of foreign substance into the structure of stone and has been divided into two categories as *Traditional Surface Diffusion* and *Advanced Beryllium Diffusion*.

Figure 4

Features indicating Surface Diffusion....

a.Colour confinement to facets and edges clearly visible when immersed in liquid and observed in transmitted diffused light.

b. Under UV light patchy fluorescence is commonly seen

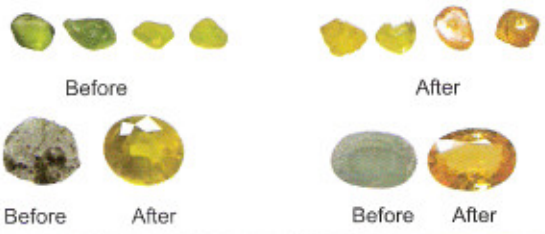
Beryllium diffusion...the changes are dramatic and any colour can be produced. The reason for this being the stones are heated at very high temperature of around 1900°C, where beryllium enters into the lattice and forms colour centers.

Identification....of Beryllium treated stones is very difficult with classical laboratory techniques as only small percentage of these treated stones possess the identifying features.

Figure 5

Various shades of Beryllium treated corundum...

Few of the changes after Beryllium treatment...



Before After

Before After Before After

The treatment can conclusively be identified by advanced sophisticated techniques like Laser Induced Breakdown Spectroscopy (LIBS) or Laser Ablation -In Coupled Plasma - Mass Spectroscopy (LA-ICP-MS). The operating cost of these techniques is very high thus the cost of testing becomes higher than the cost of the stone.

Figure 6

Features identifying Beryllium Diffusion....


a. Blue Halos or Spots formed due to internal diffusion of titanium oxide around a crystal inclusion

b. Orange / Yellow rim is the conclusive feature in beryllium treated stones

Heating with Fillers..... It includes filling of fissures/ cracks with a foreign substance thereby enhancing the clarity of the stone. Material used for filling is a basically a glass; this is divided as :

- Borax / Silica glass
- Lead glass

In case of Borax, the aim is not the clarity improvement but is a result of heat treatment processes where the flux material penetrate into the fissures. On the contrary, lead glass is intentionally filled into the fissures to enhance the clarity. The effects in this case is much better as compared to the tradional borax filling therefore the original material used is of much lower quality and hence cheaper.

Figure 7

Changes....

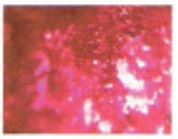
Both images : untreated ruby (left) & glass filled (right) from Madagascar

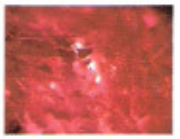
Identification.....of glass filled rubies can be done on the basis of observation of fissures / cracks which are breaking on the surface. Look for the *flow pattern* with in the crack/ fissure; this is generally visible due to improper filling, also seen as patches; *reflections* from the crack; *thickness* of the filled material; *gas bubbles* along crack.

Figure 8

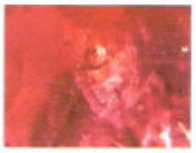
Features indicating fillers.....

Fissure in this ruby showing thick & opaque nature of borax.

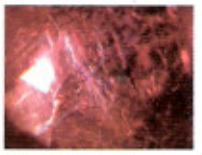




Patchy filling of lead in this filled ruby gives high reflection in oblique illumination while appear dark in transmitted



Note the large gas bubble in the filled cavity/ fissure.



Blue flashes is one of the identifying feature in lead filled ruby.

In some cases the conclusion may not be drawn on the internal features; in such cases the filler can be detected using EDXRF spectroscopy, where it exhibits the peaks for lead. Although, most of the methods of treatments discussed are identifiable, but still there are some features, which are overlapping in natural and treated counterparts making the conclusive identification very difficult.....

EDXRF.....now at GTL

Energy Dispersive X- Ray Fluorescence spectrometer has been installed at GTL in April 2007. This equipment is already available with most of the laboratories all over the world and is now with GTL. After a work of two months we are now able to conclusively detect lead glass filled rubies. This enables us to mention the lead filling on gem identification reports and making the certifications much clearer. This service has been started from 11th June 2007. Still, a lot of work is to be done on the machine to utilize it to its full strength. The installation of this machine will help improving the quality of gem identification reports as well.

GTL...Annual Awards Function....2007

19th April 2007, Gem Testing Laboratory, Jaipur has celebrated its annual awards function at Mohanlal Sukhadia Hall, Rajasthan Chamber Bhawan at 4:00 P.M.

Chief Guest, Shri Prakash Chandra, Director General (Investigations), Rajasthan, Jaipur kindly consented to present the certificates and deliver the valedictory address.

Other distinguished guests from the local Gem and Jewellery industry were present; some of them were Shri Rajiv Jain (Regional Chairman, GJEPC, Jaipur Region), Shri Vijay Kumar Chordia (Convener, Technical and Education Committee, Jaipur), Shri Mehul Durlabji (Co-Convener, GTL), Shri K.L Jain (Honrary Secretary General, RCCI), etc.

The two trade awards: **Durlabhji Education Trust** Award for the Best student of the Year (Overall) went to Kashish Sachdeva and

Bhuramal Rajmal Surana Award for Best Student of the Year in Practicals won by Rishi Kant.

Following candidates stood out best in their respective batches in the year 2006 - 2007:

1st Overall

1. Apeksha Jain	-	Batch 37
2. Kashish Sachdeva	-	Batch 38
3. Priya Mohan	-	Batch 39

1st in Practicals

1. Ananya Gupta	-	Batch 37
2. Smeet Rajen Zaveri	-	Batch 38
3. Rishi Kant	-	Batch 39

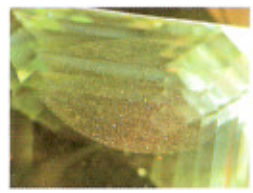
Glasses...with interesting features...

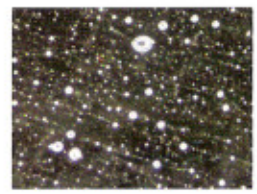
A constant development in technique and manufacture of synthetic gemstones and glasses often leads to products which may surprise a gemmologist. Recently at GTL, we have encountered several glass specimens exhibiting some unusual/ interesting features like strong colour zoning similar to kyanite, circular bands, fingerprint like patterns, etc.

Case 1: A blue stone, colour very similar to sapphire or kyanite exhibited a strong eye visible colour zones that are commonly seen in kyanite. However careful observation revealed a wavy pattern of zones, which raised suspicion. Testing of the stone using instruments concluded it to be a glass. Under U.V. lamp, the stone gave a strong surface related yellow-green fluorescence; refractometer gave a constant reading at 1.515 and the spectroscope revealed a strong cobalt spectrum On immersing the stone in water, wavy blue, green and colourless zones were observed. These colour zones were associated with the swirl marks which were evident on careful examination.

Case 2: A colourless carved specimen in the form of "*Shivling*" was tested. The expected material was either glass or rock crystal, therefore we started looking for gas bubbles or swirls using a fiber optic light but surprisingly, some concentric circular whitish bands / zones became visible at the base of carving; these bands closely resembled to those seen in synthetic sapphires by flame fusion method. On turning the specimen and viewing from sides (direction perpendicular to those of curved bands), the zones appeared straight. These zones were giving the impression of a cross section of concentric cylindrical tubes. At higher magnification, these zones appeared to be planes of white pinpoint inclusions; however, exact nature of these pinpoints could not be resolved. One more striking feature of this specimen was the reaction under shortwave ultraviolet light, a strong violet blue fluorescence was observed confined to the circular zones; the edges appeared blue while the central area violet.

Case 3: A greenish blue- "aquamarine" shade stone was tested and as expected could be an aquamarine or a glass. A fingerprint like inclusion was seen just below the table but surprisingly, weak swirl marks were also seen creating a doubt regarding the origin.

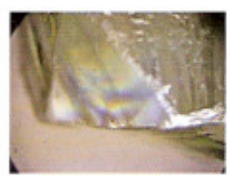




When magnified, the fingerprint appeared to be composed of some rounded and elongated inclusions similar to phase inclusions, but careful examination revealed that those droplets are basically fine spherical and elongated gas bubbles. Although, glasses are very common but they often surprises a gemologist now and then.

Quartz with Play of Colour

Recently, a green coloured broken rough with some natural faces was tested. Some of the faces appeared triangular in shape with triangular growth features and strong twinning similar to that seen in quartz. The material was concluded as quartz using specific gravity and FTIR analyses.



The striking feature in the specimen was the colour flashes as seen in labradorite/' spectrolite feldspars. The cause of this play of colour appeared to be fine dotted and fibrous inclusions in three directional zones at 60/ 120°, which also caused a sheen effect.

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