Asian Institute of Gemological Sciences Gem Testing Laboratory

"Lead Glass Filled/Repaired Rubies"

By Vincent Pardieu

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In November 2004 a large number of rubies having some uncommon features were brought for testing to the AIGS laboratory in Bangkok. For two months more than 200 of these rubies were analyzed in the laboratory. Lead was detected in their fissures and cavities. Between September and December 2004, a total of 244 of these rubies ranging in size from 3 to 97 carats were examined at the AIGS laboratory including 12 stones over 25 carats. In January and February 2005 these stones did not stop to keep AIGS laboratory busy.

Surprised by the large number of stones suddenly appearing on the Bangkok market, AIGS laboratory has decided to give priority to the study of these stones and began to research this new product.



3 rubies found with "lead rich foreign substance filling in fissures and cavities" at AIGS laboratory

Following several weeks of enquiry we found that the treatment was being performed in Chantaburi, Thailand. At the end of December 2004, two gemologists from AIGS visited the company that was applying the treatment. We were very surprised to discover that the treatment expert, Mr. Mahiton Thondisuk, had been a student at the AIGS.

Very friendly contacts were re-established with Mr. Thondisuk, also known as "Kob" and his partner in this enterprise, Mr. Somkuon Plairahan. Mr. Plairahan had formerly been in Mae Sai, the border town with Burma, where he was an expert in Mong Shu Ruby heat treatment.

In the tradition of AIGS philosophy about sharing knowledge in the gemological field, they were very happy to present to AIGS gemologists their treatment methods.



(Left to right: Ruby expert: Mr. Somkuon Plairahan, AIGS laboratory Director: Vincent Pardieu, Master burner: Mr. Mahiton Thondisuk)

Following the publication of the first version of this study in January 2005, AIGS gemologists have found that several other companies located in Bangkok and Mae Sot were also producing some lead glass "repaired" rubies. It is likely that in the future we will see many stones presenting some unusual features as several companies are known to have begun to work on this new treatment. The glass composition could be very different in the future as some people are just mixing lead oxide powders with different fluxes, silica, crushed glass or quartz in order to try to enhance rubies from different origins.

The present article is the result of the four visits to Chantaburi by AIGS laboratory gemologists in December 2004 and January 2005 and two visits to "Orange Sapphire" company in Bangkok. This last company was famous for its production of sapphire heated with beryllium technology. Several rough and also cut samples from these companies were assembled along with some stones furnished by Bangkok buyers interested in the study of these gems. These stones as well as others brought to the laboratory by AIGS regular customers were also used as a basis for this study.

The purpose of this article is to provide to gemologists, gem dealers, jewellers and persons interested in the gemological field around the world some information to assist in the identification of these gemstones. Mr Mahiton Thondisuk want also to show that Chantaburi burners are not cheaters, he is happy to communicate about Thai technology and contribute to make Thailand and Chantaburi a more famous gem trading center.

PART 1: Interview with Mr. Mahiton Thondisuk from Chantaburi, Thailand

Mr. Mahiton Thondisuk explained to the AIGS gemologists that this treatment was the result of five years of study in association with several Thai scientists from different universities in Thailand. The idea was to develop a glass compound suitable to "repair" fissures in rubies.

"Beautiful natural stones for which human intervention is limited to cutting and polishing are very rare and extremely expensive", explained Mr. Mahiton Thondisuk. "There are many more low quality gemstones compared to the tiny amount of real gem quality stones so that if we can find a good way to repair them we can add them to the trade. In the beginning, I was not thinking that people would actually buy this product: A ruby repaired with glass, it's crazy. So after few months, I went north to do some farming. But some people began to buy these stones so I came back to Chantaburi 2 months ago to produce these stones."

"You know," he added, "I love naturally beautiful rubies, in fact I don't really like to do this treatment but the market is looking for nice looking cheap stones. So I'm doing it...

This is quite amazing to see how we can improve the beauty of these stones! "

It was just after Mr. Mahiton Thondisuk returned to Chantaburi with Mr. Somkuon Plairahan that the lead glass "repaired" ruby gemstones arrived in large numbers at the AIGS Laboratory.

Several isolated gemstones had been seen on the market a few months before and at that time a warning was published by GAAJ (Japan) in March 2004 about lead glass found in ruby. Several short articles were also published by GIA, AGTA and other organizations on their website or in magazines. AIGS laboratory had also presented some photos on these unique inclusions on its website last year. In fact, this treatment is not really a new development. The process was developed in 1982, by diamond cutter Zvi Yehuda from Ramat Gan (Israel) to produce "Yehuda diamonds". The process used for fracture filling diamonds was quite common in the 1990's. Several companies were producing such stones at that time. Many studies were conducted and many articles about them are found in gemological literature or on the internet. In the fall 1994 edition of *Gems and Gemology* an important study of these diamonds was presented. At about this same time AIGS was developing some special radiographic instruments to detect lead glass filling in diamonds.

The fact is that lead glass filling of fissures is back and this treatment is now known in Thailand as "Paw Mai" (new heat treatment in Thai language). A few months ago in Chantaburi this term was used for the beryllium treatment of stones. Most of the stones seen these days are believed to be mainly sent to Asian markets like China and India in which the conjunction of important gemstone traditions and the need for cheap gems has created a niche for this product. But these stones are known to have reached the USA and Europe.

Mr. Mahiton Thondisuk adds: "It is simple, if I give you the choice between two rubies of equivalent beauty at the same price: one synthetic grown in a factory and one grown in nature but "repaired" by human technology after being mined, which one will you choose?"

"We are selling these stones at very good prices... Imagine how much a 20 carat heat treated ruby would cost and compare that to one of our stones of equal beauty? We are so cheaper!"





Large lead repaired Rubies in process to be sold on tables in Chantaburi

This is an important point and in AIGS opinion there is nothing wrong about treatments as long as the customer is provided with the correct information about the stone and the price is related to the real quality of the stone. The important thing is the proper disclosure of the nature of the stone. A very good point with these "repaired rubies" is that they don't currently present any real identification problems, which is a major difference when compared to the beryllium treatment issue.

PART 2: The "lead Glass" treatment, step by step:

Note: Some terminology problems may occur about this treatment regarding to the "Lead Glass" definition as many different formulas can be used: Pure lead oxide, lead oxides mixed with silica or fluxes like borax can be encountered... Temperatures, parameters and result can be very different. Some specific studies will probably be done in the future regarding to this issue. Here is now a presentation of the treatment performed in Chantaburi, Thailand by Master Burner Mahiton Thondisuk:

The most suitable rubies for repair are stones with color potential and that are rich in fissures. This new treatment is performed currently mostly on Andilamena rubies (Madagascar) on which Mr. Thondisuk has had extensive experience but any ruby material with fissures could be "repaired".

It is a multi step treatment involving simple heating and the use of different lead rich compounds to fill the fissures and cavities of the stones. If most of the "repaired" stones seen were large size stones, stones less than 1 carat have also been treated this way.



(Andilamena rough rubies before treatment)

<u>First step:</u> The stones are preformed to eliminate the matrix and other impurities that could disturb the treatment.

Note: As you can see here, small and large stones are treated together and AIGS gemologists have seen many rubies less than 1 carat treated with lead glass. But many stones treated this way do not present fissures and as a result will not show diagnostic features of the treatment.

<u>Second step:</u> The stones are "warmed". In fact, this step is a heat treatment. This step is important to remove the impurities possibly present in the fissures that could create some problems when the glass is added. The heat treatment may also by itself improve the stone color.

This "warming" can be conducted at different temperatures from 900C to 1400C depending on the ruby type. As 900C is not hot enough to melt some inclusions as rutile, many stones can still have an "unheated" aspect. But all stones are heated.

Third step: The stones are then mixed with powders oxide and heated. The composition of the powder is mainly a mixture of silica and lead, but sodium, potassium, calcium and metallic oxides like vanadium or bismuth also enter in some glass composition. It could be interesting to compare the glasses used here with those used for diamonds, but this would need further investigation. There are 2 main types of glass compositions used in this process at the moment but experimentations about new glasses are in process.

There is currently the "basic" formula which is a simple lead rich transparent glass. This formula is used for most of the best quality larger stones. This orange powder will turn into a yellowish to orange glass after heating as you can see in this used crucible.

In this case, the heat treatment temperature is believed to be around 900 degrees Celsius in Chantaburi. But the treatment temperature can be much higher for other burners as other components are used in the composition of the glass.









The second formula also incorporates some other metallic oxides, in order to produce glass, optimizing the color and aspect of rubies. This formula is known in the market as the "popular" formula and is used on "iron stain" rich commercial quality stones.

This formula is in fact a mix of many oxide powders that turns to a pink glass, after melting.

With this formula, the treatment temperature is slightly higher nearing 1000 degrees Celsius.

The powders are added to the stones with care along with some oil so they will cover them. The stones are then placed in crucibles and are then bring to the furnace. The powders will fuse during the heating process and turn into glass. Note: A well balanced glass composition is the key to achieving good transparency and fluidity so the glass will fill the entire fissure. The glass stability is also an important concern to create a durable product. New improved fillers will probably soon be tested and used in order to get better results.

These stones are then heat treated with the glass powder under a controlled atmosphere using electric furnaces. Special precautions have to be used at this step due to the use of lead compounds involving high temperatures. Gas masks, gloves and special compounds are used to clean the clothes and the treatment area.

Heat treatment using such kind of chemicals is very technical and requires special knowledge and security precautions as lead vapors are very toxic.









Rubies covered by colorless lead rich glass after heat treatment in Chantaburi.



Rubies covered by yellowish lead rich glass after treatment in Bangkok

Then, the stones are preformed and heated again using lead glass mixture in order to get a better result. Some stone can be heated several times with several types of oxides up to the desired result achieved.

The stones are finally cut and polished. Sometimes the resulting stones have their surface treated again using several chemicals in order to improve their surface luster.

This last surface treatment may explain why it is easy to detect lead on the stone using EDXRF technology.



Several rubies heated with lead rich glass in Chantaburi (photo 1) and in Bangkok (photos 2 to 6)

PART 3: Identification and analysis of the "repaired rubies":

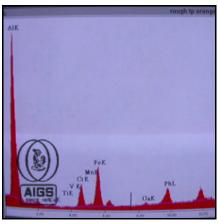
Identification of the lead glass "repaired rubies" is very easy for any laboratory that owns an EDXRF (Energy Dispersive X-Ray Fluorescence), but microscopic observation is in most cases enough for a gemologist that has experience with these stones. Using EDXRF the AIGS laboratory was able to find lead in all the stones studied. It is the ideal instrument to provide a rapid diagnostic result.

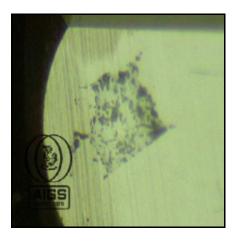
<u>EDXRF:</u> This instrument's main use is to provide some quantitative and qualitative information on the chemical composition of a given stone. EDXRF can detect any element heavier than fluorine and it is especially efficient for heavy elements such as lead. This instrument provides a very fast and reliable diagnostic analysis. The AIGS laboratory is using this instrument on all rubies presented for identification.

On the EDXRF spectrum on the right you can clearly see the peaks related to various elements. Ruby is composed of aluminum, oxygen and some trace elements such as chromium and iron that give the stone its color. The presence of lead (PbL on the photo) is diagnostic that the stone has been treated with the lead glass technology because lead is never found in natural corundum. Lead was found in every stone that was tested and copper was also found in 2 stones with very large filled cavities.

This cavity on a "repaired" ruby was carefully placed to be checked using EDXRF, as the cavity luster is very close to the ruby luster showing that the glass has a refractive index very close to the ruby. The cavity surface is visibly damaged, as the glass is very soft. Such damages could have occurred during the cutting or polishing process. Copper was detected while studying this large cavity along with lead but it could be the result of pollution from polishing powder residue filling damaged areas. (The detection of copper was not successful under other conditions)







Because lead is a very heavy element, we are here in the exact opposite situation compared to the Beryllium issue. Beryllium is a very light element. Its detection is not possible using EDXRF, which makes the detection of the stones, treated using beryllium a complicated matter. This relatively new type of treatment involving lead is very easy to detect.

<u>Ultra Violet:</u> Examination of the stones using a standard SW and LW (short wave and long wave) fluorescent box did not give any diagnostic result.

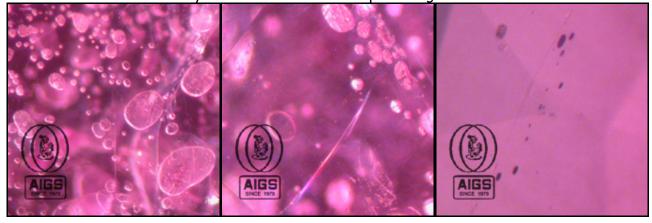
<u>Microscopic observation:</u> An experienced gemologist using a microscope and dark field illumination will be able to identify correctly the "repaired" rubies without any difficulty:

Using dark field illumination, most lead-rich glass filled fissures will display blue/orange flashes as seen on the following photos. This observation can be easier using fiber optic illumination looking near parallel to the fissure. This is a very typical diagnostic feature that is quite similar to "opticon flash effect" in emeralds or in lead glass filled diamonds.

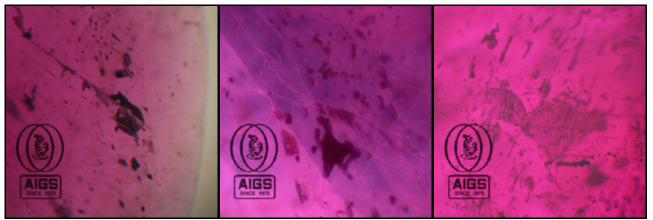
Note: Depending upon the company performing the treatment, the composition of the glass and the parameters used, the "blue/orange flashes" may vary from very obvious to very difficult to observe:



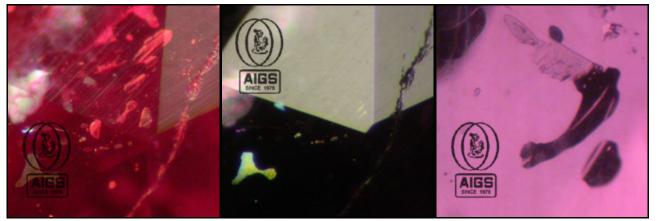
With careful close-up examination of the fissures an observer may also find gas bubbles or "platelets". Gas bubbles were found in many rubies filled with the simple lead glass:



Some "platelets" are present in all the "repaired" rubies that were treated using the so called "popular" formula in Chantaburi and in most stones from Bangkok, these platelets could be remnants of former "iron stains" formerly present in many rough stones from alluvial mining areas.



("Platelets" found in Chantaburi rubies: They are transparent or act as mirrors depending the light orientation.)



("Platelets" found in Chantaburi rubies: They are transparent or act as mirrors depending the light orientation.)

Mr. Mahiton Thondisuk told first AIGS gemologists about "copper platelets". It was in fact a language misunderstanding as he was in fact speaking about "iron stain" present in the stone before the treatment: "Iron stain" is present in many stone from alluvial mines like Andilamena. Orange or

yellowish before heat treatment, it turns to whitish or blackish after treatment:

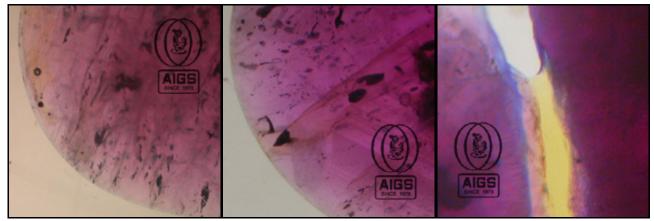


Orange "Iron stain" is very visible in these unheated Andilamena rubies fissures (photo under immersion)

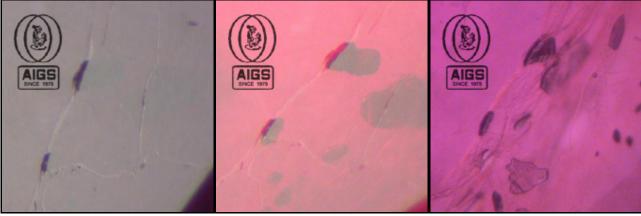


("Iron stain" remains in Pailin blue sapphire heated using traditional heating methods)

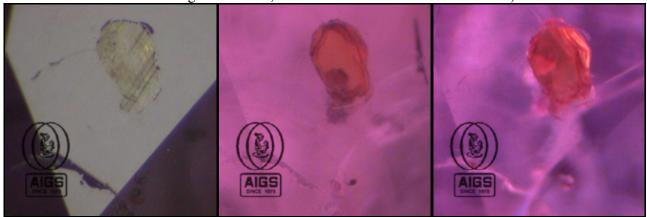
The "platelets" present in many stones does not present the typical shape of flattened glass bubbles but could be in fact the result of the mixing of the glass with some iron rich natural powders. "The number of such platelets can be reduced with careful temperature control" said master burner Mahiton Thondisuk. Careful observation of these platelets shows that they were transparent when observed from most directions, but they can also act as small mirrors inside the gem. In this case they are often seen reddish. Color concentrations in the fissures were not found in any stone from Chantaburi using the immersion technique, but some platelets did seem to have subtle coloration. In rubies enhanced in Bangkok by Orange Sapphire company, some yellow to orange color concentration appears is large fissures and in cavities. The fact that lead glass used in most Chantaburi treatment is pink explains why it is most of the time not visible inside the gem:



(Photo 1 and 2: Bangkok heated rubies presenting some orange color concentration in fissure and cavities Photo 3: Yellow lead rich glass sticking together two rubies after heat treatment using lead rich glass)



(A wide fissure filled with lead glass from a "Bangkok repaired" ruby present a noticeable orange color: Left to right: overhead, transmitted and dark field illumination)



(A cavity filled with orange lead glass in a "Bangkok repaired" ruby: Photo 1: overheard light, photo 2: transmitted light, photo 3: dark field light)



(Other cavities filled with orange lead rich glass in "Bangkok repaired" rubies. Photos: Transmitted light)

An attempt to explain the reason why the stones repaired using the "popular" formula present a stronger coloration than the stones using the "lead only" formula could be in the fact that the transparent platelets could act as mirrors inside the gem. They are normally present in a random orientation inside the gem, following the former fissures planes. They don't block the light path as orientated rutile silk can do. The light passes through some of them and is reflected by others. These reflections could increase the length of the travel of the light inside the gem. As we know, the longer the length of the light passing inside the gem, the more saturated the stone color will be. But this attempt to explain the improvement of the color should be confirmed by more in-depth studies.

Some observers could be disturbed by the fact that the heat treatment temperature is not high compared with the temperature at which many stones are heated nowadays using gas furnaces. Many inclusions may still appear as "unheated" which could disturb an inexperienced observer:

Heat treatment temperature can vary from as low as possibly 800 degrees Celsius to more than 1300 degrees Celsius. As rutile needles begin to resorbe over 1000 degrees Celsius, it is possible to find perfectly shaped needles in lead glass repaired rubies. Burmese star rubies are also known to have been repaired by burners in Mae Sot. As the glass composition, the treatment parameters and the ruby material used are not the same in Chantaburi, Bangkok, Mae Sot and in the other places this treatment is or will be performed, different features are possible.

Inclusions in Andilamena rubies repaired in Chantaburi at low temperature (Under 1000 degrees):

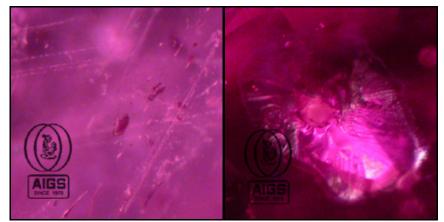


(Intact rutile needles, euhedral crystals found in many lead glass treated stones giving them an "unburned" aspect)

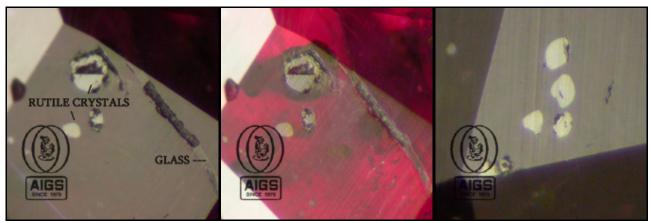
Inclusions in Andilamena rubies repaired in Chantaburi over 1000 degrees:



(Melted crystals surrounded by glassy discoids, typical of heated corundum are also found in many "repaired" rubies)



(Melted crystals, glassy discoids, resorbed needles typical of heated gems are also found in many "repaired" rubies)



(High luster rutile crystals reaching the surface should not be mistaken with lower luster lead glass filed areas)

"FUNNY" IDENTIFICATION STORY: A funny identification story happened during this lead glass ruby study: One of AIGS good friends that have provided us some stones did not warn us that the parcel was not only rubies: One stone with a similar aspect as the others was not ruby... Inclusions in this stone were slightly different from the typical ruby heated with lead glass but not that much as you can discover on the following photos: If "blue/orange flashes" were absent numerous "platelet" looking inclusions were present along with needles. When the stone was drop for immersion study in heavy liquid something was really wrong for corundum as the stone was floating! Refractive Index and EDXRF study confirmed its true nature: Pezzottaite...



(The pezzottaite cabochon submitted to AIGS lab and some of its "red platelet" looking inclusions)

"What about the durability of these gems? Should we buy it?"

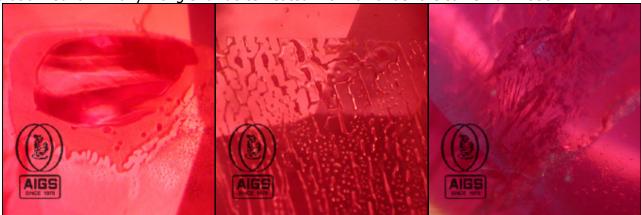
Here are several questions dealers in Bangkok, and from outside Thailand asked AIGS gemologists recently. In order to help them to find the answers, the AIGS laboratory has performed some tests to evaluate the durability of these gems:

• What is the origin of the color in these stones? The gemstone color improvement seems to result from the fact that the fissures that were formerly filled with air or liquid are then filled with a transparent glass. As lead glass and ruby refractive index are very close, the light can then travel more easily inside the gem, and as a result, the overall color looks dramatically improved. The same phenomenon is encountered with emeralds before and after oiling them.

Some questions were raised about the fact that some pink colored glass is used in Chantaburi, and a more yellow one in Bangkok:

• Can we describe these stones as dyed? Regarding the stones we have seen up to now: No. We have observed some light color in some wide fissures and cavities in rubies from Bangkok ovens but it was never enough to honestly say that the stone was dyed. Color concentration in fissures was not detected in Chantaburi stones.

Some red or orange color can be seen in some stones using "day light" tubes like those in common use in Chantaburi's buying offices. But these red looking inclusions described as "copper platelets" by treatment master Mahiton Thondisuk, present similarities with the glassy areas showing similar red coloration found in many Mong Shu rubies heated with flux under the same illumination:



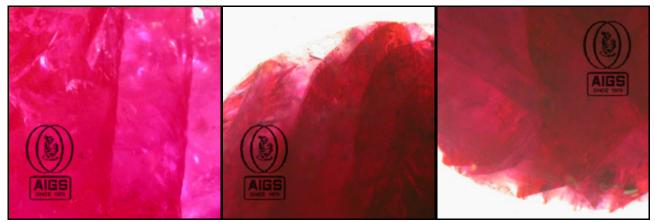
(Glassy inclusions showing some red reflected color visible in Mong Shu rubies heated with flux)

The glass lead glass present in this new treatment or the "flux residue glass" present in Mong Shu act in fact as a mirror, reflecting the color of the stone to finally give the illusion that the glass is colored. The same phenomenon can also be observed when twinning is present: Twinning planes can appear colored under some orientations. Twinning plane cannot be described as colored:



(Twinning planes seen in Mong Shu rubies heated with borax presenting some pinkish to reddish "coloration")

Dyed rubies present on the other hand some very clear color concentration in fissures which is in strong contrast with the light colored to colorless body color whatever is the direction we observe them. The presence of a light pink or yellowish glass visible in wide fissures or cavities in "repaired rubies" is very different in intensity compared to dyed rubies in which fissures are filled with intensely colored red dying agent. In fact this light colored glass found in some important fissures can be more compared to "iron stain" in natural stones: The coloration of the glass is not the origin of the color, but massively filling important fissures, it can modify the stone color if the stone color is weak.



(Typical dyed rubies presenting strong red colored fissures and a very lightly colored body color)

• **Are these stones durable?** If handled with care: Yes, probably...

As this study was performed over a 2 month period, and regarding to the fact that none of the studied treated stones were more than one year old, we cannot be 100% sure about the durability of the glass inside the gem. Now if we compare this glass filling method to the glass filling used in diamonds, the treatment looks to be suitable for "normal wear".

Compared to emerald oiling or impregnation using resins, this current ruby treatment is probably more durable. Glass is more stable than resin and its presence in a fissure will probably lower the probability that the fissure will expand. The fact that the fissures are closed with a lead rich glass is also probably improving the durability of the "repaired" gemstone, but not as much as those fissures filled by flux additives. It is reasonable to assume that these stones are possibly more durable than an impregnated emerald, but less than a ruby heated with flux.

Now we have also to consider that many companies are now trying to perform this treatment and if some will succeed, some other may encounter technical problems. As the "glass" composition might vary a lot in the future, some surprises are probably still to come.

Note: If this treatment technique is currently performed mostly on Andilamena Madagascar rubies, it is likely to spread on other fractures corundum like star rubies, sapphires and even other gemstones which can support temperatures around 1000 degrees and present fissures.

• Are only large rubies affected? No

The stones are heated together in large amounts. The most suitable material is corundum presenting multiple fissures. But small stones and clean material are submitted to the treatment in large parcels along with fissured stones. These stones after cutting will not present filled fissures, so it will not be possible to find diagnostic "blue/orange" flashes. Lead detection using EDXRF can also become a problem: When dealing with small stones, it is more difficult to get diagnostic results as we have less material to investigate. Small stones testing present some identification challenges and several EDXRF tests on different parts of a given stone are recommended: AIGS laboratory has studied several stones under 1 carat presenting lead reaction using EDXRF only when studied on the pavilion as no filled fissures were reaching the stone surface on the crown.

- What should be avoided with these stones? AIGS laboratory has performed several durability tests:
 - One of these stones to a jeweler's torch for a few seconds, and we observed some glass leaving the fissure and some glassy bubbles created on the stone's surface. This stone would then need to be polished again at a minimum to enhance its aspect.
 - Some stones were also boiled for several hours, immersed in detergents and exposed for short periods to ultrasonic cleaning without any apparent damage.
 - Several stones were submitted to "light and heat" fade test in order to study the color stability after long exposition to light. No color modification was observed.
 - Recutting or repolishing should be performed with care as the glass used is very soft and could be damaged during the process. Many stones studied presented damaged or incomplete fillings.
 - The most important threat to the stone's durability and beauty is contact with powerful acids, such as hydrofluoric acid (also known as HF). AIGS exposed several stones to hydrofluoric acid for 12 hours and 48 hours. In all cases the acid dissolved the glass and the fissures in the stones were much more visible. The color and the clarity of the gemstones were then seriously damaged in 3 cases out of 4. The attractive transparent purplish red stones had lost some saturation after immersion in HF and were then presenting unpleasant shiny whitish fissures.

Conclusions:

It is likely than in a close future different types of lead glass filled rubies will be present in the market as several companies and individuals are investing in the process.

The detection of these repaired rubies should not be a problem for any experienced gemologist with dark field illumination microscope or using EDXRF technology. But the fact that the treatment can be performed at low temperature and let many inclusions in their "unheated aspect" we recommend all ruby buyers to check their purchases with care.

Experienced gemological laboratories can provide them rapid identification if needed.

As long as these stones are properly disclosed and priced AIGS feels that these stones should find their place in the gem trade where large size and low prices are of prime importance.

The stone durability under "normal wear" should not be a problem, but it is important to notify that these stones have to be kept away from excess of heat or powerful acids. If some repairs are required special attention similar to that used for glass filled diamonds and epoxy filled emeralds, should be observed. Jewelers wishing to use these stones in jewelry should be cautious, but if handle correctly this product can be used in jewelry without problem.

AIGS Laboratory Reports Comments:

In order to properly disclose these stones to its customers, AIGS Gem testing Laboratory currently describe them as follows:

• On full reports:

Result: Found to be a Natural Ruby

Comments: This stone has been clarity enhanced.

(Color if noticeable) lead rich foreign substance found in fissures and cavities.

On mini reports:

Identification: Natural Ruby.

This stone has been clarity enhanced. Lead rich foreign substance found in

fissures and cavities.

Learn more about "lead glass filled/repaired" rubies

People interested to study more these stones can contact AIGS Laboratory in Bangkok, Thailand:

Labinfo@aigsthailand.com

More inclusions photos are also available on AIGS laboratory website inclusion gallery:

http://www.aigsthailand.com/LaboInclusion.php

Contacts:

To contact M Mahiton Thondisuk you can send email to:

labinfo@aigsthailand.com

We will be happy to transmit your message to him.

To contact Orange Sapphire:

www.orangesapphire.com

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