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To: VDM and CGH

From: John L. Emmett, Troy R. Douthit

Subject: Summary of initial heat treatment tests on Tasmanian sapphire lot CC974

Material

A shipment of sapphire was received from VDM which are as follows:

CGH Receiving #	CC #	Weight, g.	Origin
R-070214	CC974A	36.89	Weld River
R-070214	CC974B	150.1	Frome River
R-070214	CC974C	17.93	Frome River

This material was in the main very small and of very low value. However, VDM requested that we begin some heat treatment experiments on these lots with the hope that what we learn could perhaps reduce the amount of experimentation that will be required to develop heat treatment processes when true mine run lots are available. The tests turned out to be quite informative.

First Tests

Crystal Chemistry uses a series of screening heat treatment tests to determine the response of a new corundum material to heat. These tests are not designed to produce optimal color, or indeed to provide material for cutting tests. However, when complete, they will define the boundaries of what can be done with the stones.

Thus we began a set of experiments to determine the general response to heat in the mid temperature regime. The first screening was conducted at 1550°C, for 10 hours, in pure oxygen. The oxygen atmosphere is used to improve the solubility of tetravalent oxides. At this temperature there is generally significant improvement in clarity, and rutile generally goes into solution resulting in a darkening of the blue coloration. To conduct these experiments, a portion of each of these lots was separated into the following groups: unsorted, translucent blue, translucent green/gray, nearly opaque, or opaque. Each of these groups was photographed prior to heating. The groups were individually heated to 1550°C as described above and photographed again. The second test was performed at 1750°C, for 10 hours, in oxygen followed by another set of photographs. The test runs were 2711, 2712, and 2713. All photographs are in file CC419CR.

Observations

By far the largest amount of this material falls into the opaque group. There was very little increase in transparency as a result of heating to 1550°C. A few stones became a translucent or transparent very dark blue, but overall it was only a few percent. This raises the question as to whether this fraction of the material should be heat treated at all, given that if it was it would dominate the heat treatment costs. Heating to 1750°C further darkened nearly all of the material, rendering it useless for gem purposes. The material is so dark that without cutting it into thin pieces it is not possible to determine if it has any transparency. A careful test of mine run opaque material including cutting should be done to determine if there is any value in this fraction.

The next group, the translucent green/gray stones were then examined. All of the gray or brown banding became blue indicating that it is titanium minerals, primarily rutile. There appears to be increased transparency in nearly all of the material that is not heavily fractured. A single very light pink stone became blue. Overall the impression is that the color is more towards aqua from blue as we would expect from the oxygen atmosphere. Heating to 1750°C darkened the dark blues somewhat. The medium to light blues were unchanged. In a single yellow stone, the yellow color became more saturated.

The translucent blue group exhibited lighter blue coloration after the 1550°C run and a color shift towards aqua, as expected. The increase or decrease in transparency could not be assessed given the frosty surface condition of most blues. After heating to 1750°C the dark blues became much darker blue while the light blues were nearly unchanged. This could possibly indicate that there are additional titanium-containing inclusions that dissolve at higher temperatures than rutile. This translucent blue group could potentially provide some fine cut stones if similar, but larger, material was recovered in actual mine production. The problem is that this is a very small portion of the samples received, and the stones are very small. All photographs are in file CC422CR.

Second Tests

The second tests employed our process P976. This is a two-step process ending in a moderate hydrogen-based reduction. The purpose of this process is to maximize the formation of the blue coloration. Normally this makes basaltic blue sapphire so dark that it is difficult to see light through it. From each of the previously heat treated lots, we separated out stones that were pale blue, pale green, and pale gray. These were photographed, put through process P976, photographed, and put through the same process again and photographed again. The test runs were 2714 and 2715. Photographs are in file CC422CR.

Observations

We observed essentially no color change following either or both of the P976 runs. In all the different types of sapphire that we have studied, we have never before seen this result. That is the reason that we ran P976 twice, as there was always a possibility that we could have made an error in process setup the first time.

Conclusions

The most significant result from these two tests is that the bulk of the translucent material can be heated to 1550°C without becoming very much darker in color. Basaltic sapphire often contains so much rutile and/or ferri-ilmenites that heating to this temperature renders much of the material too dark for gem use. Heating to this temperature is necessary to produce clarity, and to conduct color shifting processes. The fact that the material can be heated successfully to 1550°C without becoming very dark bodes well for developing a successful heat treatment process for the translucent material.

The fact that P976 did not induce additional blue in the pale fractions might imply that we cannot successfully post process the very pale material from the initial heat treatment. If that observation is born out in later testing of bulk mine run material, it will reduce the percentage of gem quality material that can be produced. However, many of these stones would be of good color if significantly larger.

The tests described above have provided useful information for the development of the eventual heat treatment processes. No useful information has been obtained regarding either the possible yield or value of gemstones from these deposits. There are two reasons. First, these samples were clearly not representative of any mine run size or quality distribution. Second, the small size of the sample lots, once sorted, preclude any meaningful statistical analysis. In many cases only one or two stones were produced from a given sample. Finally, it should be noted that we did not see any consistent differences in the response of the material from the two locations, but again the sample size is so small that differences, unless truly dramatic, would probably not have been observable.