Spinel and its Treatments: A Current Status Report

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Introduction

Spinel has historically been one of the most highly revered gemstones. However, over an extended period of time, its popularity had suffered as a result of many factors, including its classification as "semi-precious" and a general confusion with another dominant red gemstone: ruby. More recently though, spinel has been making a strong comeback and so its popularity is once again on the rise.

Articles of important new sources and even a book devoted to this beautiful and colorfully diverse gemstone have helped to focus attention back onto spinel (see e.g. Smith et.al., 2007; Senoble, 2008; Pardieu et.al., 2009; Krzemnicki, 2010; Yavorskyy and Hughes, 2010). In addition to exhibiting a vibrant array of shades and nuances of color, spinel has also traditionally been spared the controversy of treatments that have encumbered many other gem varieties, such as ruby, sapphire, emerald, quartz, topaz and tanzanite among others.

Fortunately, spinel remains a gemstone that is generally free of treatment considerations. However, today some treatments are starting to be encountered (Robertson, 2012). This article is a review of those treatments and the gemological characteristics that help to distinguish them.

Spinel Treatments

It should be noted that for those stones presently in the marketplace, treated spinel is not commonplace and they are only rarely encountered by the major labs around the world. However, the author has noticed a general increase in the attempts to improve spinel quality and color, utilizing a variety of treatment practices.

Clarity Enhancement

The practice of filling fissures to reduce their visibility and improve the apparent clarity of a gemstone is probably the single most prevalent treatment applied to gemstones. Oils and other

Figure 1: Spinel offers a beautiful array of colors that is matched by a few other gemstones.
(Photos: Bilal Mahmood and Kelly Kramer)

materials may be introduced into a fissure, thereby making that fissure less reflective and reducing its visibility.

As a result for all colors of spinel, the filling of fissures is going to be the most common form of treatment or enhancement that one may encounter.

Detection of clarity enhancement is most readily accomplished with magnification, using a microscope or jeweler's loupe. An iridescence is generally visible along a filled fissure. In addition, areas of higher relief may also be evident where the filling of a fissure was incomplete or the filler has been partially removed (Figure 2).

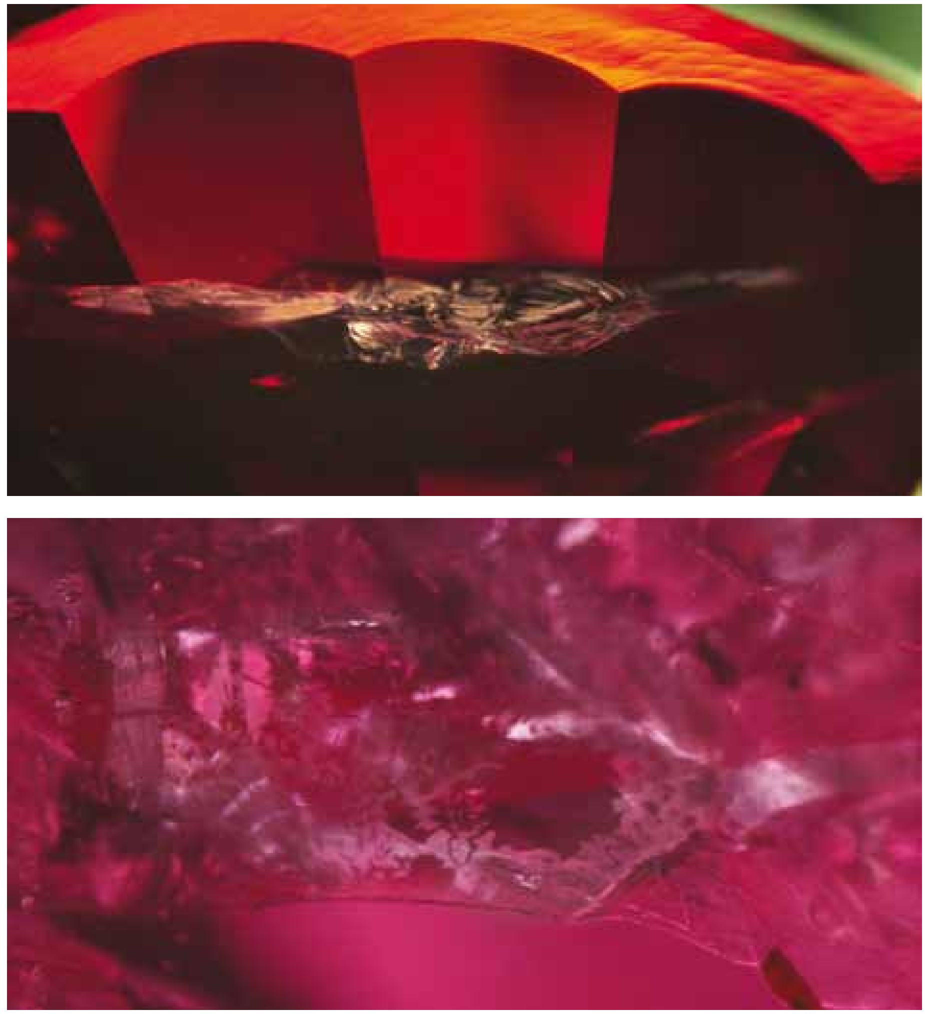

Figure 2: The filling of fissures in spinel using an oil is the most common form of enhancement that is likely to be encountered. Such clarity enhancement can be readily seen where the filling has been incomplete or the filler has been partially removed, leaving behind areas of higher relief that stand out against the lower relief areas where the filler remains. *(Photomicrographs: Christopher P. Smith, 35x)*

Heat Treatment

Throughout most of the history of gemology, it was thought that heating did not improve the quality of spinel. In the author's 25 years of experience, in the very rare occurrence when a red spinel was encountered in the lab that showed evidence of heating, this was thought to be an unintentional treatment where a spinel inadvertently was mixed in with a parcel of rubies that were being heated.

For those stones encountered in the past, this inadvertent treatment is still considered to be the case. However, in 2005 there started to be talk in the industry that heating was being used to improve the quality of some spinels from Tanzania. As a result, research was conducted that demonstrated how indeed heating could be used to alter the quality of some spinels (Saeseaw et.al., 2009).

As described, these researchers concluded that this treatment was not being performed to improve color, since their experiments showed either little to no color modification or a less desirable color resulted from the heating. However, they did find that the transparency of certain spinels could be significantly improved by heating at temperatures between approximately 950°C and 1150°C.

Additionally through this research, it was shown that Raman and photoluminescence spectroscopy provided a very effective means of identifying this treatment. The researchers demonstrated that the lattice of spinel heated at or above approximately 750°C went from what is classified as an ordered structure to a disordered structure. For more detail, the reader is referred to Saeseaw et.al., 2009. This change in the lattice may be seen in the broadening of the primary Raman band of spinel (from approximately 6.8 – 10.6 wavenumbers in unheated spinel to more than 30 wavenumbers in heated spinel), as well as a significant modification in the band structure of chromium emission (Figure 3). It is important to note that the Raman and photoluminescence spectra of spinel heated above this temperature are consistent with the spectra of synthetic spinel.

Relatively Higher Temperatures

Since the awareness of the potential for heating spinel, most major labs have been routinely testing the spinel submitted using either singly or in combination, photoluminescence and Raman spectroscopy.

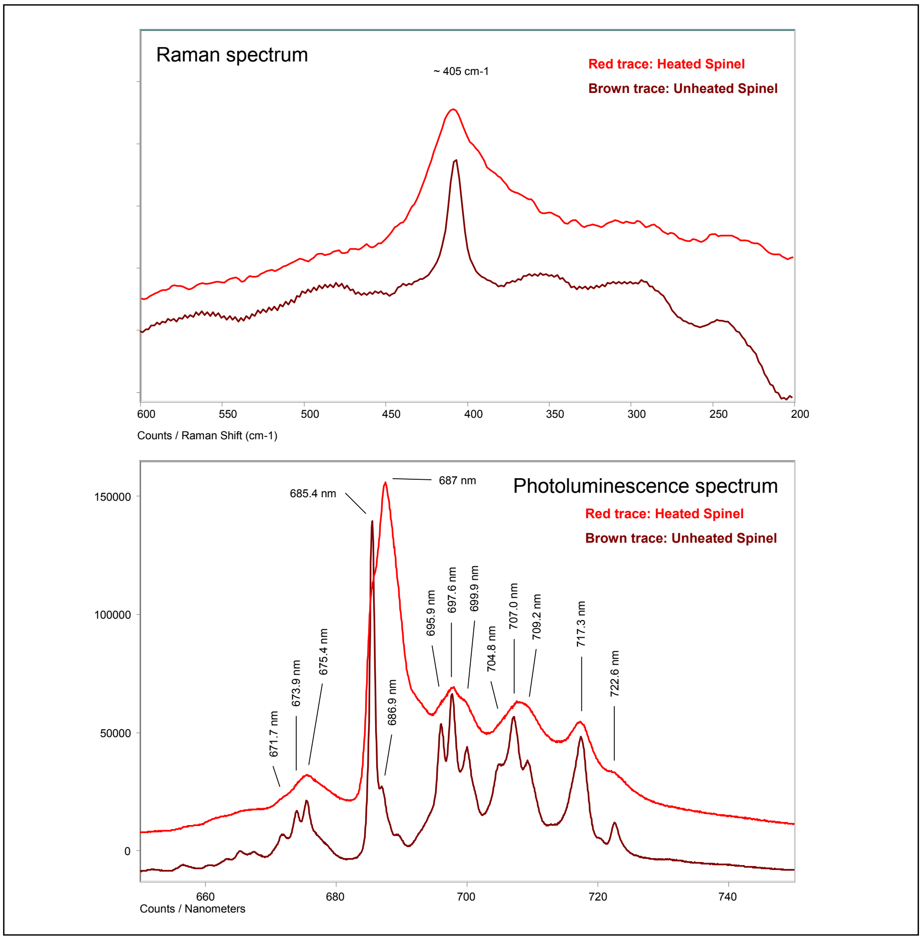

Figure 3: Raman spectroscopy and photoluminescence are very effective methods for distinguishing if a natural spinel has been heated to temperatures of approximately 750°C or more. At approximately this temperature, the lattice of spinel transitions from an ordered to a disordered structure. The traces of this disordered structure may be seen in the broadening of the primary Raman band positioned at approximately 405 cm-1 Raman shift, as well as the chromium (Cr) emission of the photoluminescence spectrum. Shown here are two natural spinels: one unheated and one heated.

To date, only a handful of spinels tested by the AGL have been determined to be heated in this manner. For these stones, not only were the photoluminescence and Raman spectroscopy helpful in determining their heated status (Figure 3), these stones additionally exhibited inclusion features that revealed evidence of being exposed to heat treatment (Figure 4).

A similar limited number of stones have been identified by other major labs, such as the GIA (McClure, 2012). However, other researchers have been in contact with groups that are attempting to increase the usage of this treatment technique (A. Peretti pers. comm., 2012).

Relatively Lower Temperatures

More recently, the AGL has examined parcels of spinel that were not consistent with the type of spinel heating described previously, yet due to various inclusion features, they still appeared to have been heated (Smith, 2011; Robertson 2012). This included most colors of gem-quality, transparent spinel with the exception of "cobalt-blue." As questions were raised to the suppliers of these parcels by the author, they admitted that the spinels had indeed been heated.

The features observed by the author were more consistent with the type of inclusion features that are reminiscent of rubies, pink sapphires and yellow sapphires, etc. which had been heated at relatively low temperatures (see e.g. Smith, 2010). These consisted of atoll-like discoid stress fractures (Figure 5), low-relief secondary fissures extending from healed fissures (Figure 6) and stress fractures surrounding various mineral inclusions (Figure 7), as well as others.

Unexpectedly, the Raman and photoluminescence spectra of these spinels were consistent with an ordered lattice and did not reveal the modifications that so readily had distinguished the heated spinels described previously (Figure 3).

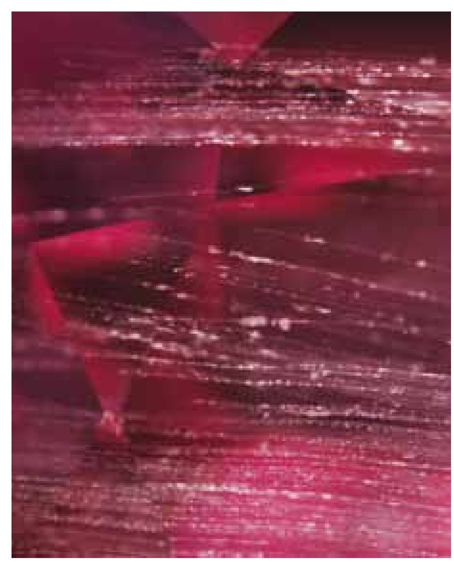

Figure 4: Concentrations of fine stringer-like inclusion features are common to certain sources of spinel. In the heated spinels submitted to the American Gemological Laboratories (AGL) to date, such stringer-type inclusions revealed an atypical or disturbed appearance. *(Photomicrograph: Christopher P. Smith, 42x)*

Discussion

Spinel is a fascinating and beautiful gem that is once again receiving the attention and admiration that it deserves. The broad array of color and exciting new sources have helped to catapult this gem in popularity over the past decade. As demand have grown and top-quality gems are scarce, it is not surprising that attempts are now being made to improve certain colors and types of spinel.

At the moment it is still safe to assume that the vast majority of spinel in the market is free of treatments. However more recently, attempts to improve the quality and subsequent value of spinel through treatments seems to be increasing. The oiling of fissures is an old treatment procedure that can and is applied to virtually any gemstone with surface reaching fissures. Careful observation using

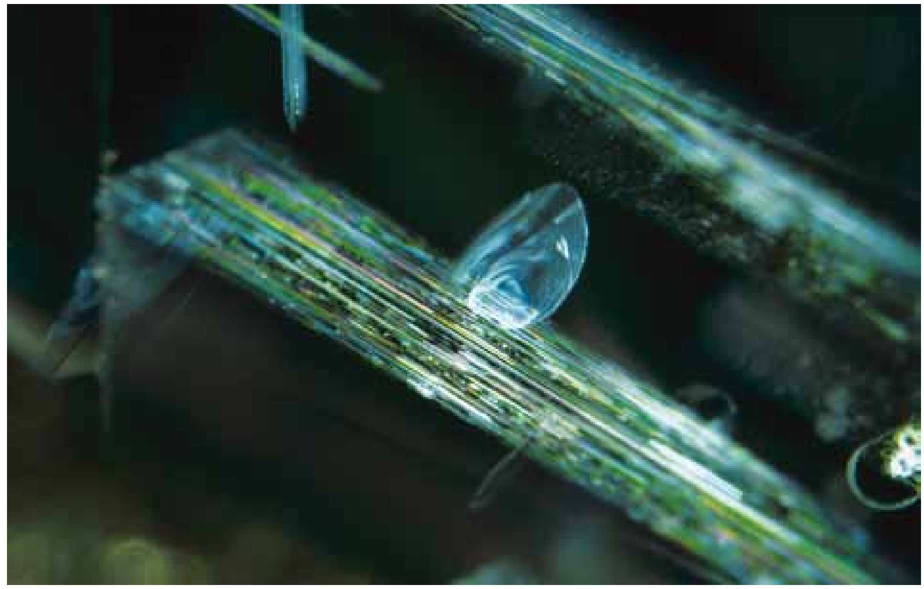

Figure 5: A more recent type of heated spinel encountered by the AGL revealed thermally induced inclusion features more consistent with what may be seen in rubies and sapphires heated to relatively lower temperatures. These included discoid or atoll-like induced stress fractures with a partial healing rim as seen extending from needle-like inclusions. (Photomicrograph: Christopher P. Smith, 75x)

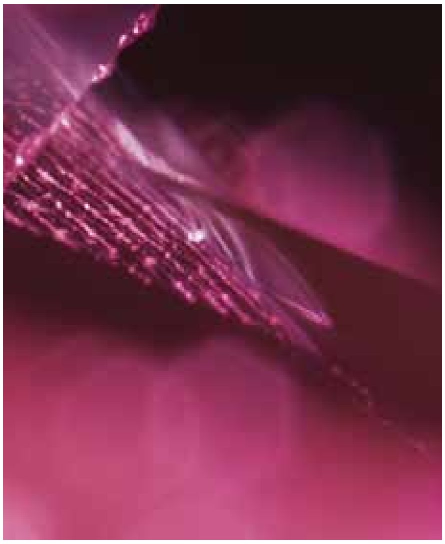

Figure 6: Another feature of the spinels heated at relatively lower temperatures was the occurrence of very transparent, subtle stress fractures extending from the constituents of healed fissures. *(Photomicrograph: Christopher P. Smith, 75x)*

magnification will permit this treatment to be identified by any jeweler or gemologist with a little training.

The heating of spinel seems to be the most recent trend. Although a few years ago it was shown that spinel heated from approximately 950°C to 1150°C may be improved in transparency, this form of treatment has so far not been widely encountered.

Even more recently, a newer heating procedure has been seen. This new treatment does not exhibit the modified Raman and photoluminescence spectra that permit the relatively higher temperature heating to be readily detected. However, a number of inclusions were observed that can help to identify this treatment. For stones free of inclusions, the detection of this relatively lower temperature heating is significantly more difficult.

In the course of discussions with those performing the treatment, the specific heating conditions were not given. However, based on the fact that the Raman and photoluminescence spectra were not altered, it is presumed that the treatment is either below approximately 750°C and/or shorter in duration than the 6 hours of heating employed by previous researchers (see again Saeseaw et.al., 2009). In further discussion with the treaters, they indicated that the spinels were being heated to improve their color and not their clarity. Although the author witnessed this relatively lower temperature heating in a broad range of colors, the treaters indicated that only some stones improve significantly. Others improved only slightly or not at all. Presently research is continuing with samples before and after heating and the findings will be presented once completed.

Although the author examined a number of parcels consisting of several hundred stones that had been heated in this manner, it is unclear at the moment how prevalent this treatment may be and the author is not suggesting that this form of treatment is widespread.

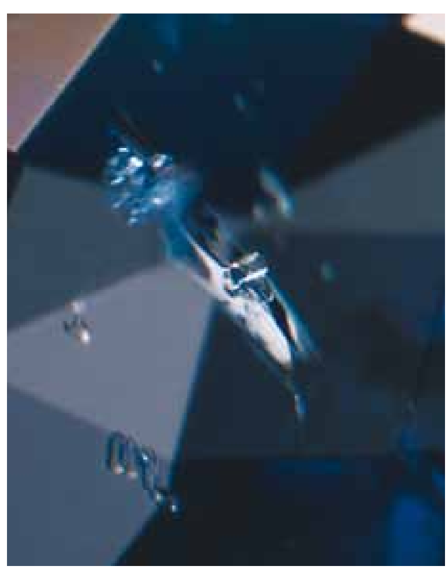

Figure 7: Mineral inclusions of the spinels heated at relatively lower temperatures also revealed evidence of this treatment. Shown here are inclusions of apatite with thermally induced stress fractures. *(Photomicrograph: Christopher P. Smith, 65x)*

As an ending note, it is unfortunate that as a gemstone which has traditionally been exempt from controversy involving treatment, spinel must now be added to the long list of gemstones that may be heated. Furthermore, this is another reminder that as gemologists, we can no longer take any gem for granted as being "untreated."

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