NEWS FROM RESEARCH

SEPARATION OF NATURAL RED ANDESINE FROM TIBET AND COPPER-DIFFUSED RED ANDESINE FROM CHINA

Ahmadjan Abduriyim and Ross Pogson

Abstract

The question of a natural red andesine occurrence in Tibet has been the source of great controversy since 2006. In an attempt to put this matter to rest, an international group conducted field research on Tibetan andesine in October 2008 and in late September 2010. The alleged deposits are located at the village of Zha Lin and in the Yu Lin Gu Valley in Bai Nang County, ~3 km from the previously investigated Nai Sa village–Bai Nang mining area and about 55 km of southeast of Shigatse. Meanwhile, commercial treatment by diffusing copper into andesine was reportedly ongoing in China. The starting material for this process was said to be pale yellow andesine from Gu Yang County, Inner Mongolia, China. This article presents additional details on the morphological appearance and the geochemical and powder X-ray diffraction properties of these Tibetan samples as well as Cu-diffused samples from Xi’an and Shen Zhen in China. Some of the natural-color Tibetan samples proved indistinguishable from Cu-diffused Inner Mongolian andesine using laboratory techniques, although destructive analytical methods may establish feasible identification criteria.

INTRODUCTION AND BACKGROUND

To address whether natural red plagioclase feldspar-andesine occurs in Tibet, the author and five gem dealers and a gemologist visited an alleged deposit called Bainang (or Bai Lang) in 2008. The deposit is located near the village of Nai Sa, ~55 km southeast of Shigatse (or Xigazê; see figure 1; Abduriyim, 2008, 2009a,b). Copper-bearing red andesine of gem quality has been found in the mine, which is owned by Li Tong of China. The internal features and chemical composition of the Tibetan samples collected on the 2008 expedition closely resembled the allegedly copper-diffused Inner Mongolian red andesine available on the market, to the degree that doubt was cast on the natural origin of the Tibetan stones (Hughes, 2010; Rossman, 2011).

In an effort to resolve the controversy, another field trip to Tibet was carried out in late September 2010. They studied the reported Tibetan andesine occurrences at Zha Lin village and Yu Lin Gu (Abduriyim and Laurs, 2010; Abduriyim et al., 2010; Hughes, 2010; Leelawatanasuk, 2010). These areas are located ~3 km from the Nai Sa–Bainang mining area, which the author visited in 2008. Due to the objection of a local monk who controls access to the land in this area, the party was unable to visit the Bainang mine. The villagers interviewed by the expedition said the orange-red stones have been known in this part of Tibet for several decades or longer.

A brief excerpt of the research on andesine from three localities in Tibet and the known Cu-diffusion treated andesine
from Xi’an and Shen Zhen was published in the summer 2011 issue of Gems & Gemology. The geologic field information gathered by the expedition group was also included in that comprehensive article.

This article introduces more detailed photos and figures laboratory testing of the samples the author collected, including surface features, chemical and gemological properties, and structural order as revealed by power X-ray diffraction.

MATERIALS AND METHODS

All samples described in this article were obtained during the author’s 2008 and 2010 expeditions. These were personally collected by expedition members (for samples from Zha Lin and Yu Lin Gu, see figure 2a,b) and collected near the Nai Sa village. The Bainang mine area (see figure 2c). In Guangzhou, the group obtained two additional lots of red andesine that were reportedly copper-diffused in China using Inner Mongolian pale yellow andesine as the starting material. These were treated either in Xi’an in 2004–2005 or in Shenzhen in 2007–2008 (figure 3a,b).

Standard gemological properties (RI, SG, UV fluorescence, Chelsea filter reaction, spectroscopic observations, and microscopic characteristics) were obtained for 198 pieces of reddish orange to orange-red andesine from Zha Lin and Yu Lin Gu. Of these, 95 were polished with two parallel windows and cleaned with acetone and aqua regia in an ultrasonic bath to remove surface contamination. In addition, 50 known-treated red andesines (from Xi’an and Shenzhen) were characterized.

Figure 1. The reported andesine mines visited by Abduriyim’s 2008 and 2010 expedition are located in south-central Tibet, ~55 km from Shigatse.

Figure 2. A pile of andesine rough samples obtained by Abduriyim’s 2008 and 2010 expedition are shown here. (a) The samples are from Zha Lin (0.12–1.09 g), (b) Yu Lin Gu (0.20–3.20 g), and (c) Nai Sa–Bainang (0.11–2.92 g). Photo by A. Abduriyim.
Quantitative chemical analysis using a JEOL JXA-8500 electron microprobe was carried out at NIMS (Tsukuba, Japan) on the polished surfaces of eight orange-red and red samples (two each obtained from Zha Lin and Yu Lin Gu during the 2010 expedition, and two each of the known-treated stones from Xi’an and Shen Zhen). Five spots were analyzed on each sample. The operating conditions included an accelerating voltage of 15 kV, a beam current of 7 nA, and count times of 20 seconds on peaks and 5 seconds on backgrounds.

LA-ICP-MS trace-element data were obtained for 80 orange-red to red Tibetan samples (20 from Zha Lin and 40 from Yu Lin Gu obtained in 2010, and 20 from the author’s 2008 expedition to Naı’nan and Besnang); 45 known-treated red samples (22 from Xi’an and 23 from Shenzhen); and 12 pale yellow samples from Inner Mongolia. Data were collected with a Shimatsu LabX XRD-6000 X-ray diffractometer, using CuKα radiation (λ = 1.5406 Å) with beam conditions of 40 kV and 30 mA. Diffraction data were collected over a range of 7–70° 2θ with a step interval of 0.02° 2θ and a count time of 1 second/step. The lattice parameters were calculated by the least-squares method using CellaCalc software, with the well-known parameter of ∆2θ = 2θ[131] − 2θ[131] to determine structural states based on the spacing between the (131) and (131) peaks of the powder X-ray diffraction pattern (Ribbe, 1972). To test whether the ∆2θ parameters were changed by heating, two pale yellow Inner Mongolian samples were selected for copper diffusion experiments. Each sample was cut into two halves.
two pieces. Half of one sample (INMG-DF-001a) was buried in ZrO₂ powder mixed with 2–3% copper powder and diffused at 1160ºC for 50 hours; half of the other stone (INMG-DF-002a) was packed into Al₂O₃ powder mixed with 2–3% copper powder and heated at 1050ºC for 24 hours. The remaining halves were retained as control samples.

RESULTS AND DISCUSSION

Gemological Observations

The andesine samples collected from Zha Lin and Yu Lin Gu were generally indistinguishable from one another and from those obtained in Nai Sa village. All of the andesines from these localities were rounded. Most of the stones were transparent to translucent, and their overall gemstone quality was not high (again, see figure 2). In fact, very few of the samples would be expected to yield high-quality cut gemstones. Most of the pieces were under a centimeter, and the largest reached ~2 cm. Their color was mostly pale orangy red or red, though a rare few had a pure red color. Compared to andesine from the Nai Sa–Bainang area, the Zha Lin and Yu Lin Gu samples generally were a weaker red. Some showed green or bluish green zones or patches; any surface-reaching fractures were surrounded by a narrow colorless zone (see figure 4). In addition, a few of the Yu Lin Gu samples showed a distinct yellow color zone near the surface. Most also had a narrow colorless rim along the surface, and some stones showed concentric color zoning with green-blue and red bands.

None of the Tibetan samples exhibited a good crystal form. They had a smooth water-worn appearance and generally round, elongated faces. A few had broken cleavage surfaces. Some of the andesines also featured embayed areas that appeared to have been created by chemical etching, while some areas displayed a melted glassy surface (figure 5a and b).

The samples’ refractive index and specific gravity fell in the range for the andesine variety of plagioclase feldspar. They gave a weak chalky orange reaction to long-wave UV radiation, and dark red or inert under short-wave UV. Under a Chelsea color filter, the andesine remained red. Some red stones appeared green in diffused transmitted light, and a...
Figure 5. (a), some of the Tibetan samples from three localities have a smooth water-worn appearance but with chemical etching pits, cavities and channels. (b), colorless or white viscous glassy residue can be seen on the surface. Photomicrographs by A. Abdurizim; magnified 12x and 20x.

Figure 6. Some internal features noted in the Tibetan andesine included plenty of twin lamellae which are parallel to {010} (a), hollow channels (b), irregular dislocations and turbid milky clouds (c), and rare schiller from the presence of native copper inclusions (d). Copper platelets are well known in Oregon labradorite, but those platelets tend to be much larger than Tibetan samples (e). Photomicrographs by A. Abdurizim; magnified 15–25x.

Figure 7. The known-treated samples from Xi’an and Shen Zhen showed numerous pipes (a), tube-like structures and discoid fractures (b), a recrystallized white residue filling the lath-like hollow channels (c), dislocations (d) and cloud-like inclusions (e). Photomicrographs by A. Abdurizim; magnified 20–25x.

weak red-green pleochroic stone appeared red in white incident light. Examination with a gemological microscope revealed that most of the Zha Lin and Yu Lin Gu samples contained prominent twin lamellae that were parallel to the {010}, parallel lath-like hollow channels, and pipe-like tubes (figure 6a and b). They also contained irregular dislocations
and color patches caused by milky turbidity from fine granular inclusions (figure 6c). A few stones also displayed healing fractures with no difference of light reflection on the surface. Some contained native copper grains or platelets, which produced reddish orange schiller after polishing (figure 6d). Similar copper platelets are known in Oregon labradorite, but those platelets tend to be much larger (figure 6e). None were seen in the known-treated samples from this study.

The known-treated samples contained pipe and tube-like structures, discoid fractures, a recrystallized white residue filling the lath-like hollow channels, dislocations, and cloud-like inclusions (figure 7). Overall, these resembled the internal features seen in the Tibetan andesine. Distinct concentric color fluctuations from the rim to the core were present in some of the known-treated stones, however (e.g., figure 8). These layer-by-layer color zones appeared more distinct than in the Tibetan samples.

**Surface Residue Features**

Microscopic observation of the samples from all three reported Tibetan localities revealed surface residues in
Separation of Natural Red Andesine from Tibet and Copper-Diffused Red Andesine from China

The residues ranged from transparent to translucent and were colorless/white, yellow, orange, and black. The colorless/white fragments or glassy residues were identified as crystalline alpha quartz and feldspar by Raman spectroscopy, though some residues did not display clear spectra.

Some known-treated andesines showed very characteristic surface residues such as viscous melted glassy material (colorless or white) and gas bubbles. Plenty of tiny spotty numerous particles covered the full surface of these materials, a highly magnification observation by SEM is need. The fractures and cavities were covered by black residue (figure 10). Raman analysis was unable to show any spectra that could be assigned to any crystalline mineral from some white glassy residues on the surface, suggesting that these residues are in amorphous structure.

Chemical Composition

Microprobe analysis of two andesine samples from Zha Lin yielded a composition of $\text{Ab}_{46.4} \cdot \text{An}_{49.0} \cdot \text{Or}_{3.3} \cdot 3.8$ (expressed as mol.% albite:anorthite:orthoclase). They plotted in the andesine field, near the border with labradorite (figure 11). One samples from Yu Lin Gu showed a similar range of major elements but with more variable anorthite contents ($\text{An}_{51}$), which placed them at the border of andesine-labradorite.

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Figure 14. Trace-element data of the Tibetan andesines (blue) and known-treated samples (red) was processed by multivariate statistical analysis into this principal component diagram. Ten isotopes (\(^{14}K, ^{49}Ti, ^{57}Fe, ^{69}Ga, ^{88}Sr, ^{107}Ag, ^{137}Ba, ^{139}La, ^{140}Ce,\) and \(^{208}Pb\)) were categorized into two parameters as component 1 and component 3. There is ~30% overlap between the two groups. 3-dimension view by plotting with 3 components in different angles. "DF" copper-diffused stone.
field. However, five spots were analyzed on another sample (TB-YLG-NP-001) from Yu Lin Gu. The concentration of An mol.% showed very flexible values at 60.17%, 59.03%, 57.74%, 58.34% and 58.02%; some positions may indicate a lamellae growth-zonation feature as solid-solution. Detailed chemical mapping needs to be carried out on the entire sample area (see table 1).

With the exception of the An₅₈ sample, these data overlap the compositions given by Rossman (2011) for samples from Tibet and "China." A similar composition was documented in the Nai Sa–Bainang andesine studied by Abduriyim (2009b). Microprobe analyses of four known-treated samples gave compositions of Ab₄₆-₄₉:An₄₈-₅₀:Or₂₉-₃₃, placing them in the andesine field at or near the border with labradorite, indistinguishable from the Tibetan samples. The Zha Lin and Yu Lin Gu andesine contained overlapping copper contents (0.04–0.15 wt.% CuO), similar to the range in the known-treated samples (0.07–0.11 wt.% CuO).

LA-ICP-MS analyses of samples from all three Tibetan localities showed a similar range of Li, B, Mg, P, K, Sc, Ti, V, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, Rb, Sr, Y, Zr, Ba, and Pb, as well as the rare-earth elements La, Ce, Pr, Nd, and Eu (see table 2). Except for the trace element of silver, the andesine samples collected from Zha Lin, Yu Lin Gu, and Nai Sa–Bainang were generally indistinguishable from one another. Copper ranged from 270 to 1200 ppmw and tended to be higher in stones with a redder color. Li contents showed corresponding increases with Cu, while Ni concentrations were higher at the surface than in the interior.

Traces of Ag were recorded only in samples from Yu Lin Gu, ranging from 35 to 2900 ppmw. The concentration of Ag at the outer edge of the samples, where a colorless rim was visible, was much higher than the average inside the stones. Also, the amount of Ag appeared to diminish toward the center. In contrast, the distribution of Cu and other trace elements throughout the samples did not show any specific trends (figure 12). Some stones from Zha Lin showed a very low amount of Ag at the surface (~0.12–0.26 ppm by weight) but none in their interior. Ag was not detected in any of the Nai Sa–Bainang stones or in any known-treated or Inner Mongolian materials.
The known-treated samples showed trace-element contents similar to those of the Tibetan stones, but the Cu content decreased from the rim toward the core in most of them (figure 13). Barium contents were slightly lower (99–142 ppmw) than in the Tibetan andesine (123–166 ppmw).

Multivariate statistical analysis of the trace-element data yielded the principal component diagram shown in figure 14. Ten isotopes ($^{39}$K, $^{49}$Ti, $^{57}$Fe, $^{60}$Ga, $^{88}$Sr, $^{107}$Ag, $^{137}$Ba, $^{139}$La, $^{140}$Ce, and $^{208}$Pb) were categorized into two parameters as component 1 and component 3, and data were plotted into this principal component diagram. The blue symbols represent the 80 samples from the three reported Tibetan localities and the 12 pale yellow samples from Inner Mongolia. The red symbols indicate the 43 known-treated samples. Although there was ~30% overlap between the two groups, the known-treated samples tended to have lower values for the component 3 parameter (<0.2) and relatively high values for the component 1 parameter (up to 0.53).

**Powder X-ray Diffraction**

Using the parameter of $\Delta2\theta = 2\theta_{131} - 2\theta_{131}$, combined with anorthite content, we can determine the Al/Si distribution among the four tetrahedral sites in the plagioclase structure (Ribbe, 1972). The diffraction data for five Zha Lin andesines gave values of $\Delta2\theta = 1.9554^\circ$–$1.9724^\circ$, while five samples from Yu Lin Gu yielded $\Delta2\theta = 1.9572^\circ$–$1.9700^\circ$ and 10 from Nai Sa–Bainang were calculated as $\Delta2\theta = 1.9539^\circ$–$1.9668^\circ$ (figure 15). Of the known-treated samples, five from Xi’an were in the range $\Delta2\theta = 1.9574^\circ$–$1.9886^\circ$ and five from Shenzhen gave $\Delta2\theta = 1.9720^\circ$–$2.0047^\circ$. The copper-diffused samples’ tendency to have a higher parameter is reflected by the degree of Al/Si disorder at the tetrahedral site. The temperature at which this disorder formed is probably higher than 800–900°C. The 12 pale yellow samples of Inner Mongolian andesine showed a significantly lower parameter of $\Delta2\theta = 1.9161^\circ$–$1.9506^\circ$ compared to the Tibetan and known-treated samples.

Our copper diffusion experiments confirmed that the $\Delta2\theta$ parameter can be altered significantly by high-temperature heating. Both diffused samples showed red and green colors (figure 16a and b), and the parameters changed from $\Delta2\theta = 1.9264^\circ$ to $1.9764^\circ$ and from $\Delta2\theta = 1.9506^\circ$ to $1.9811^\circ$. The high temperatures associated with laboratory diffusion of Cu into plagioclase are reflected in the increased disorder measured by powder X-ray diffraction. Nevertheless, the cooling history of the reputed Tibetan andesine is unknown, so it is difficult to interpret how the order/disorder data relate to the possibility of laboratory treatment.

**CONCLUSIONS**

Most of the samples obtained in Zha Lin and Yu Lin Gu during the 2010 expedition were rounded pebbles of lower quality than the attractive gems represented as Tibetan in...
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Note: Chemical composition data analyzed by EPMA is an average of 5 analyses on per sample, and data shows a value range of numbers sample in each locality. * was studied in Abdurrahim 2003.

Table 1. EPMA chemical composition data of 8 samples from Tibet and China.
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Table 2. LA-ICP-MS analysis of 80 orange-red to red Tibetan samples (20 from Zha Lin and 40 from Yu Lin Gu obtained in 2010, and 20 from Abdurrijim’s 2008 expedition to Na Se Bainang); 45 known-treated red samples (22 from Xi’an and 23 from Shenzhen), and 12 pale yellow pieces from Inner Mongolia. Results are in ppmw.

http://www.gia.edu/research-resources/news-from-research August 1, 2011
the marketplace. The andesine from the Nai Sa–Bainang area generally had a stronger red color, and 4–6% of the production had a high commercial quality.

The gemological properties between the Tibetan and the known-treated samples obtained by the expedition overlapped. Both the Tibetan and the known-treated andesines may host glassy surface residues consisting of colorless or white patches of silica and feldspar. Raman spectra confirmed that most of the silica fragments in Tibetan andesine have a quartz crystalline structure, but some glassy residues of known-treated materials are amorphous silica. Known-treated materials from Xi’an and Shenzhen displayed a viscous-like melted residue on the entire surface and a black residue covering the fractures and cavities. The reddish orange schiller effect due to native copper grains can be seen in a few of the samples from Zha Lin and Yu Lin Gu, but the known-treated materials did not show such a phenomenon.

The chemical properties also overlapped, except for the significant amounts of Ag in the Yu Lin Gu samples and a possible depletion of Ba in the known-treated stones. Multivariate statistical analysis of the chemical data in a principal component diagram gave a 30% overlap in the Tibetan and known-treated stones. Powder X-ray diffraction data showed some overlap between the two, but copper-diffused samples tended to have a higher parameter, which is reflected by the degree of Al/Si disorder at the tetrahedral site.

Argon isotopic studies by Dr. George Rossman indicated that the samples from Zha Lin and Yu Lin Gu (none of which had glassy surface residues) had not been exposed to the high temperatures associated with diffusion treatment (Rossman, 2011).

Our research has shown that to date there is no single analytical technique to separate the natural Tibet material from the treated stones from China. This separation might be made using a combination of several advanced techniques, such as microscopic observation of internal features, LA-ICP-MS chemical analysis, X-ray powder diffraction Δ2θ parameter measurement (or single-crystal X-ray diffraction), and argon isotope analysis, but most of them are expensive, destructive, or not widely available, making them impractical for individual specimens.

The field investigation group in 2008 and 2010 expedition found strong field evidence of a genuine andesine deposit at Nai Sa–Bainang and Zha Lin, but could not confirm the authenticity of the Yu Lin Gu occurrence. No primary andesine-bearing source rocks were found in either area, and a more thorough geologic investigation of the region is still needed.

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