Corundum C-Axis Device for Sample Preparation

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Abstract

As a part of GIA's on-going project to establish a comprehensive corundum database a need was recognized for accurate polarized (O and E) absorption spectra. For research material this necessitates crystallographically orienting, grinding and polishing specimens prior to recording the spectra. This note describes a tool developed to facilitate this work which provides for crystallographic orientation and mounting of the sample for grinding and polishing. Absorption spectra where the measurement beam axis is accurately parallel (E-field perpendicular) to the corundum optical axis is possible to within 1 degree, as verified by x-ray diffraction. Similarly, flat optical faces parallel to the optical c-axis may be created for spectrophotometer measurements that are E-field parallel to the c-axis. A tool and method for finding the optical c-axis accurately and preserving this through the grinding and polishing process is described.

Introduction

Approximating the location of the optical c-axis in corundum can be accomplished using a simple dichroscope for samples with adequate color saturation or using the polariscope. These methods provide some level of control for finding the c-axis but may produce an error of several degrees. For faceted material or rough with jagged edges, finding the c-axis on the polariscope may require immersion in methylene iodide. The dichroscope and polariscope do not provide the ability to attach the sample to a fixture for grinding and polishing while preserving the c-axis alignment. The tool developed by GIA is based on a previous tool developed by Dr. John Emmett and on the Western Electric conoscope (Heising, 1946). GIA has added laser alignment of the lenses and a method of accurately introducing a second dop for the purpose of transferring the aligned sample from the tool to the grinding and polishing steps, while preserving the c-axis parallel to the transfer dop.

Tool Description

Figure 1 illustrates the optical path of the tool. A fiber optic illuminator supplies light to a filter wheel for selecting the desired illumination color. The filter wheel contains three interference filters which are manually selected by the operator and centered at 450 nm, 550 nm, and 650 nm, with 80 nm bandwidth (FWHM). The filter is chosen by the operator to maximize the light transmission through the sample. Fresnel lens (FR1) is located 1 focal length away from the fiber optic, and approximately collimates the fiber optic output. A polarizing filter (PF1) polarizes the light. A second Fresnel lens (FR2) focuses the light into the immersion cell at the location where the corundum is located. After passing through the corundum sample, the light diverges and is approximately collimated with a Fresnel lens (FR3). The three Fresnel lenses are identical and have a diameter of approximately 50

millimeters and a focal length of approximately 50 millimeters. A second polarizing filter (PF2) is oriented so that its polarization axis is perpendicular to PF1. The operator looks through the system and adjusts the pitch and yaw of the sample until the visually observed figure is centered on a reference fiducial (cross-hair) which is on FR3.

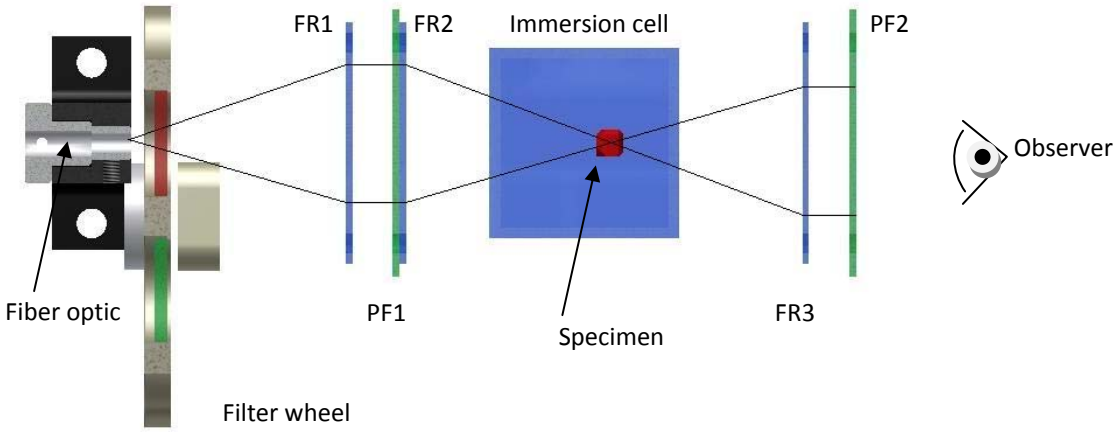

Figure 1 C-Axis Tool optical path. The immersion cell is a 40 mm cube (inside dimensions). From the fiber optic to PF2 the distance is ~170 mm. The observer is ~230 mm from PF2.

The immersion cell is resting on a platform that is driven vertically with a rack and pinion drive (Figure 2) to either drive the cell up or down. This allows for the immersion cell to be removed from the path without disturbing the sample.

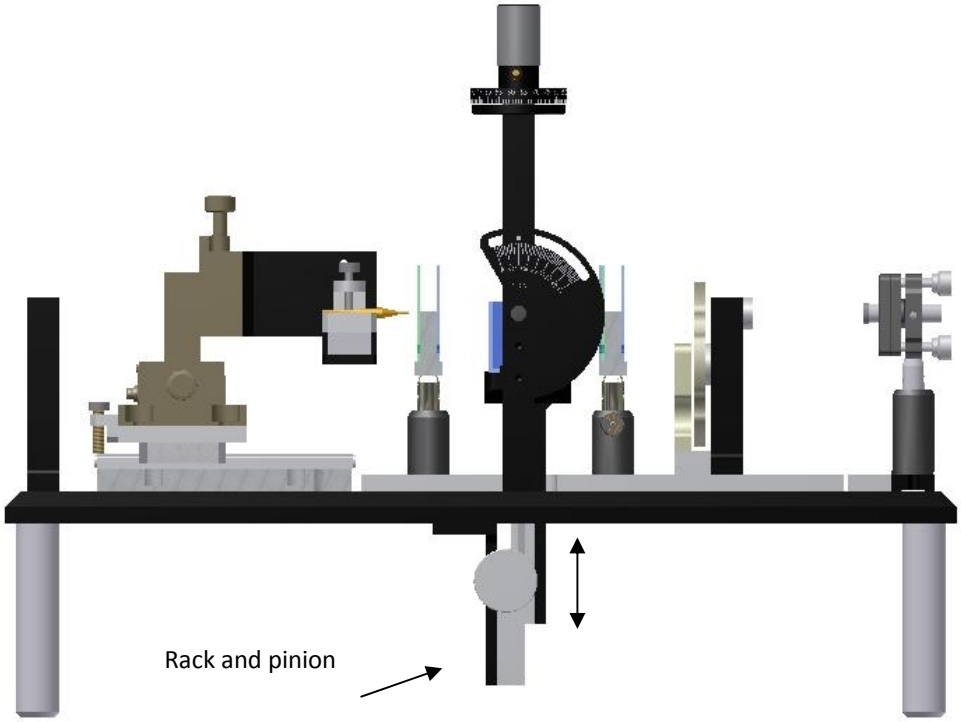

Figure 2 C-Axis Tool side view. Black base plate is approximately 560 mm in length.

Optical Alignment

With the fiber optic, filter wheel, FR1, PF1, FR2, immersion cell, FR3, and PF2 removed, a low-power red alignment laser diode is aligned so that it is centered on two alignment targets. The first being the center of the fiber optic and the second being a similar mount approximately 260 millimeters away from the specimen location. The red laser beam serves as the reference for aligning the lenses FR1, FR2, and FR3, as well as aligning the mount for the transfer dop. While observing the aligned red laser on the far field target the lenses can be aligned in centration and the tilts can be aligned by observing the back reflected light at the laser output face. The reference fiducial is marked directly on the surface of FR3 by finding the center of FR3 using a conventional microscope and marking the surface with lines through the center of the lens. As the lenses are Fresnel lenses, the exact center of the lens is easy to locate.

The lens mounts reference a precision reference rail (Figure 3) which enables the lens mount assemblies, once aligned, to be removed and repositioned against the rail without losing alignment. With the lens assemblies removed, the alignment laser is allowed to strike the face of a specially prepared alignment dop. The face of the alignment dop has been polished to a mirror surface that is perpendicular to the mechanical axis of the alignment dop. With the alignment dop mounted in the kinematic removable vee-block mount, the supports for the vee-block are aligned in pitch and yaw to retroreflect the laser back to itself. This establishes the mechanical axis of the dop to be parallel to the optical axis of the lenses. The alignment laser is only used for the alignment of the lenses and dop mount and not during orientation of corundum samples.

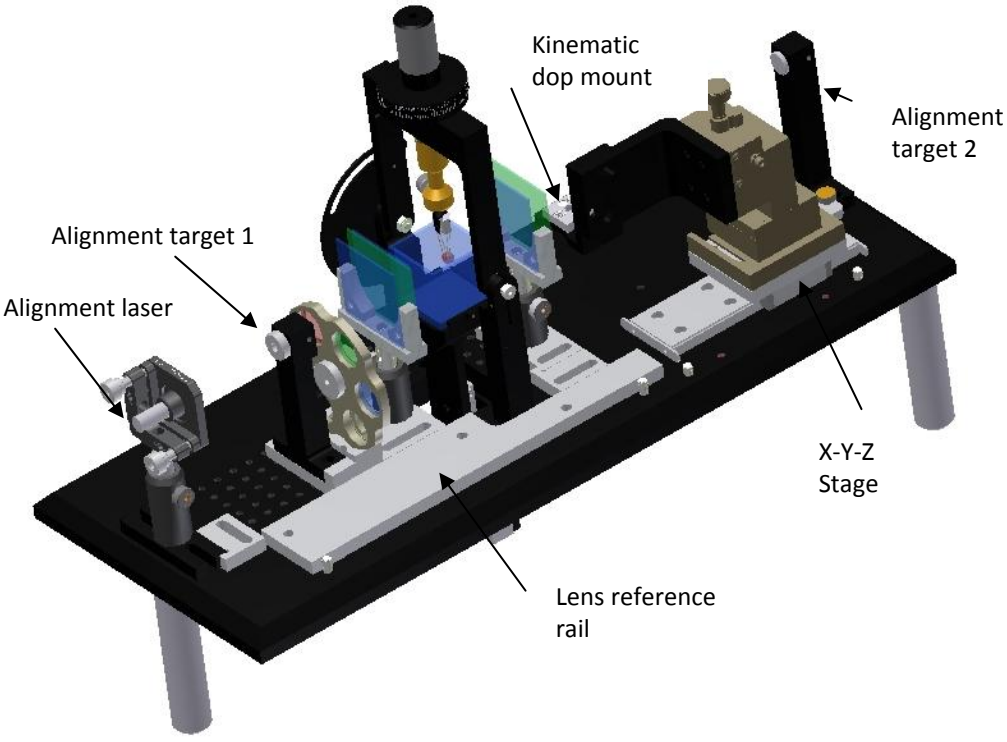

Figure 3 C-Axis Tool laser alignment features.

Operation

The approximate optical axis of the corundum sample is found using the dichroscope or polariscope. The sample is then placed in a standard dop transfer fixture and attached to a dop using rapid curing adhesive with the optical axis approximately perpendicular to the dop axis. Alternately, the sample may be mounted in a gem clip but this approach is not rigid and the sample may accidentally be misaligned.

This initial dop is placed in the spindle of the C-Axis Tool. The immersion cell is raised and lowered using the rack and pinion slide. This allows the immersion cell to be out of the way during dop insertion, removal, and attachment of the transfer dop. With the initial dop inserted in the spindle the immersion cell is raised to immerse the specimen.

The spindle of the C-Axis Tool rotates about its axis 360° (yaw) and tilts (pitch) +60° and -20°. The fiber optic provides illumination through the lens path and through the sample. The user views the cross-hair on FL3 while rotating and tilting the spindle until the figure is centered on the cross-hair (Figure 4).

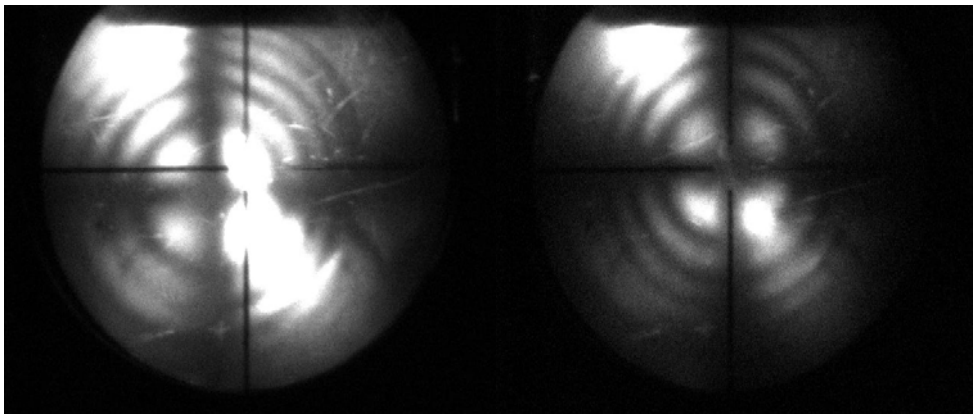

Figure 4 Misalignment (left) and aligned (right), black and white CCD camera images.

After the optical figure is aligned to the reference fiducial, the transfer dop is introduced. Figure 5 illustrates the kinematic-magnetic vee-block for holding the transfer dop.

Figure 5 Transfer dop mounted in vee-block (left), magnetic base (middle), and kinematic seat for repeatable locating (right). The dop shown is approximately 50 mm in length and 6.3 mm in diameter.

Figure 6 illustrates how the transfer dop is next brought to the specimen. Frame “a” shows the transfer dop loaded on the kinematic mount. In frame “b” FL3/PF2 have been removed and the immersion cell lowered. FL3/PF2 are removable without using fasteners, and

repeatably located using mechanical reference surfaces. The immersion cell is covered, and a vessel is introduced to collect solvent used to rinse the methylene iodide from the specimen (not shown). Adhesive is applied to the end of the transfer dop and the lower stage is moved to carefully position the adhesive in contact with the specimen (frames "c" and "d"). The upper stages have fine adjustments to allow for translating the transfer dop in the horizontal and vertical directions so that the bond may occur at a satisfactory location on the specimen. After the adhesive is cured, the transfer dop vee-block may be opened (frame "e") and the lower stage retracted, leaving the transfer dop attached to the specimen.

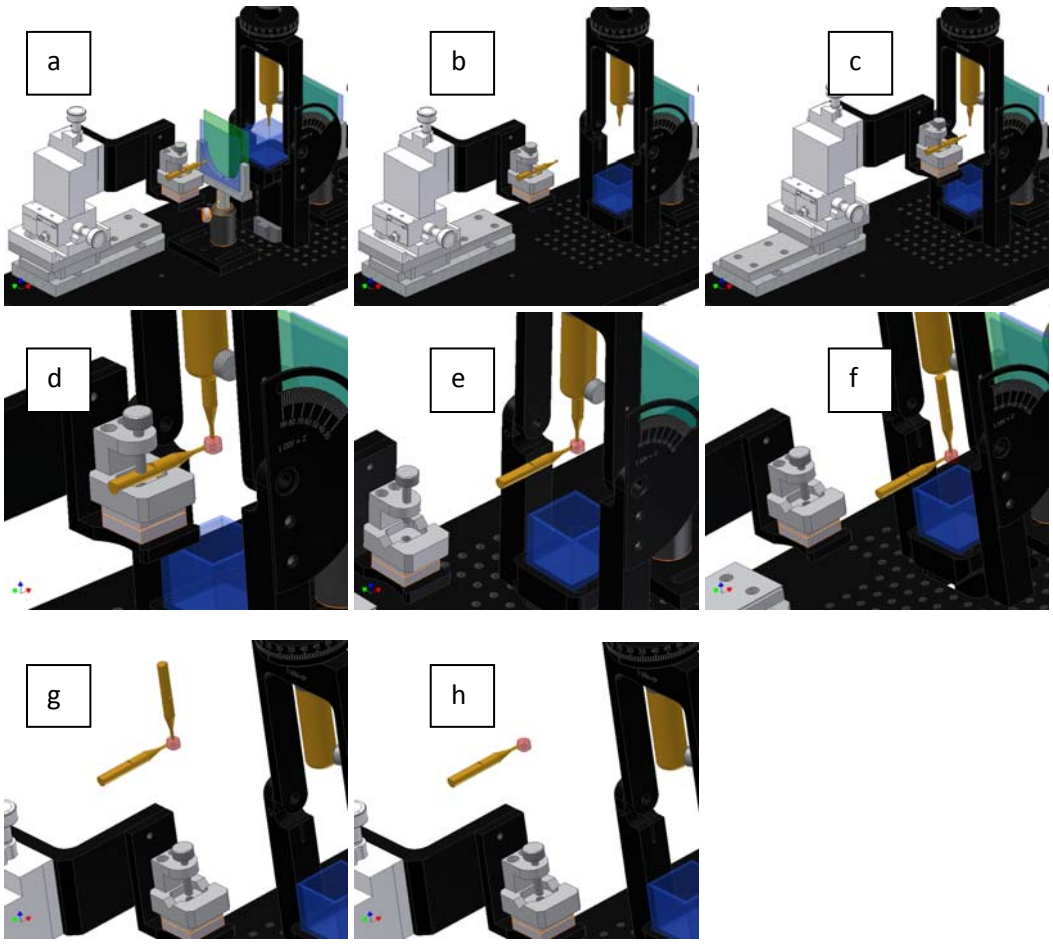

Figure 6 Attachment of the transfer dop to the specimen.

The initial dop may now be removed from the spindle (frame "f"), yielding two dops attached to the specimen (frame "g"). Finally the initial dop is removed, leaving the aligned transfer dop attached to the specimen (frame "h"). The specimen is then ground and polished.

Results

X-ray diffraction data (Shen, 2008) shown in Figure 7 illustrates the performance produced by the tool, using an earlier and less sophisticated transfer mechanism and without laser alignment of the transfer dop mount. The optical components were laser aligned. The X-Y-Z

mechanics, magnetic kinematic dop vee-block, and alignment laser are recent improvements believed to improve the performance and usability of the tool.

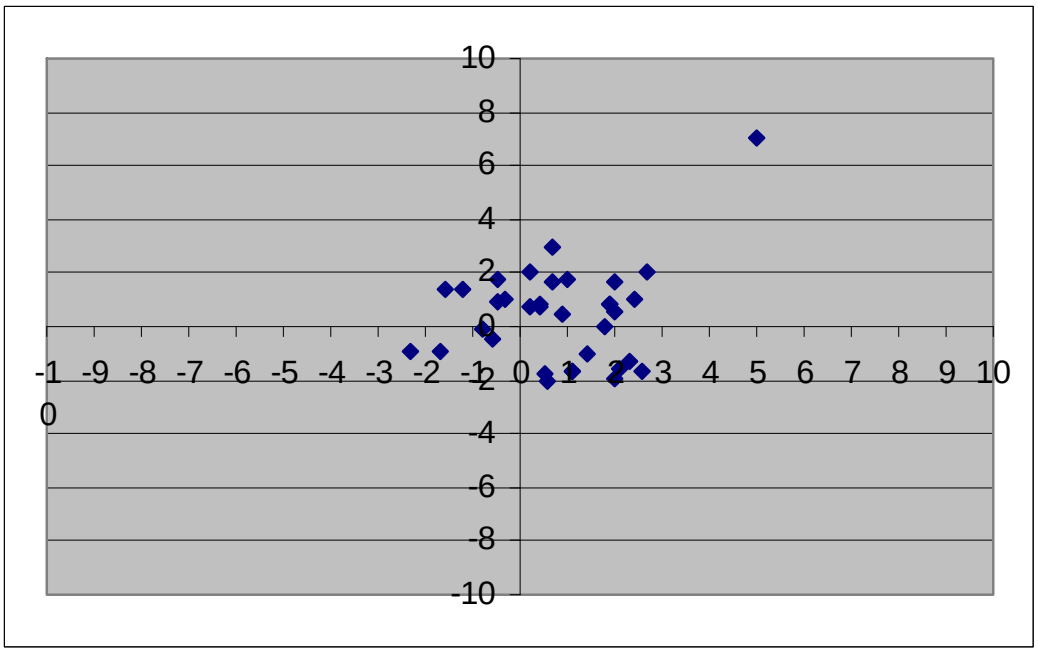

Figure 7 C-axis error from polished face in degrees.

The data indicates an average error in the horizontal direction of .8° and a standard deviation of 1.5°, while in the vertical direction the error is .4° with a standard deviation of 1.8°. Since this data was produced, the precision X-Y-Z stages were introduced, as well as characterization of the dop grinding and polishing device. The data shows one data point at 5°, 7°, most likely caused by moving the sample before the adhesive was fully set, or possibly bumping it with the introduction of the transfer dop.

The tool is able to easily find the c-axis on thin samples. Figure 8 illustrates the tool performance with no sample, a sample of approximately 10 mm thickness, and a sample approximately 300 micrometers in thickness.

The optical figure produced when no sample is loaded is caused by residual strain in the optical components. This was not found to interfere with finding the optical axis of samples.

Heat and forces generated during the grinding and polishing process can sometime cause the specimen to separate from the dop. This can be improved by first performing additional sample preparation by pre-grinding the sample roughly perpendicular to the c-axis prior to mounting. Fast curing UV adhesives or waxes are another option, but waxes may be difficult to keep fluid during the dop attachment process.

Dops without samples were polished and measured using laser retroreflection. In one case, the polishing head required approximately 0.6° degrees of correction to reduce the error introduced by the polishing machine.

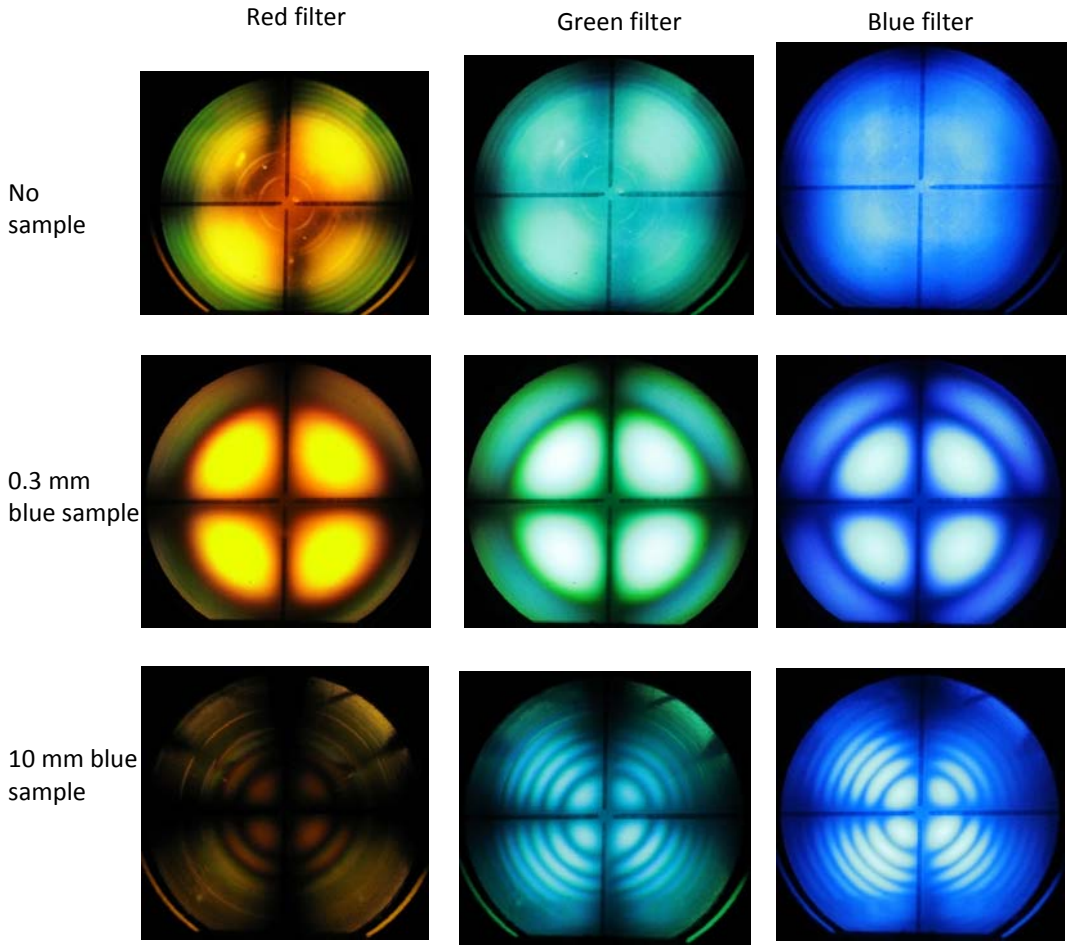

Figure 8 Operator view of the C-Axls Tool for different thicknesses and filter selections.

Heavily fractured rough may require thinning to produce a reliable optical figure useable enough for finding the c-axis. Twinned material produces a figure comprised of the superposition of the tilted axes, and may be noted in the specimen tracking data as a point of reference.

Conclusion

A precision tool for mechanically orienting and attaching a dop aligned to the c-axis of corundum samples has successfully been developed and introduced at several GIA laboratories. The tool is in use and producing aligned corundum samples as part of GIA's country of origin database.

Acknowledgements

Dr. John Emmett, this tool is based on Dr. Emmett's own tool which he developed and graciously loaned to GIA for the early stages of this work. GIA's Dr. Andy Shen and Cornell University for acquiring the x-ray diffraction data.

References

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Shen, A., (2008). T. Thomas, GIA Carlsbad Research, using Cornell University x-ray diffraction equipment,