

Preparation of Indicator Minerals for Electron Microprobe Analysis

Indicator minerals are used in the exploration for a variety of mineral deposits as each mineral type can uniquely identify a particular type of deposit or rock lithology. Generally, indicator minerals have a relatively high density, are silt to sand sized and are physically and chemically resistant (Averill 2001; McClenaghan 2005). These properties help to isolate the indicators in mineral concentrates, created through a variety of size, gravity and magnetic separation techniques so they can be selected with relative ease using a binocular microscope. Examples include scheelite and wolframite as indicators for tungsten deposits, cinnabar for gold or mercury deposits and cassiterite as an indicator for tin deposits (McClenaghan 2005).

Section preparation can be applied to any type of indicator mineral or to any type of sample that is limited in the number of grains for analysis or in grain size. For example, kimberlite indicator minerals are invaluable tools for diamond exploration. As few as five Cr-pyrope grains in a single 10 kg till sample may provide sufficient evidence that further sampling in a region is warranted as they indicate the presence of a kimberlite (McClenaghan et al. 2000; Averill 2001). However, it is the mineral chemistry of that grain that is most revealing, as it can provide information about the upper mantle environment where this mineral formed and the potential for association with a diamond-bearing host (Fipke et al., 1995; Schulze, 2005; McClenaghan and Kjarsgaard, 2007).

Mineral chemistry data obtained by electron microprobe analysis of selected indicator minerals are used to classify indicator minerals and to determine the

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diamond prospectivity of an area (McClenaghan & Kjarsgaard 2007). Pyrope garnets are sorted according to their composition; G10 garnets contain less Ca than G9 garnets, with G10 garnets being generally the target in diamond exploration (Fipke et al. 1995; Averill, 2001; Schulze 2005). This garnet classification scheme is not always possible solely using information from a binocular microscope examination. As well, minerals such as crustal garnet and eclogitic garnet are difficult to distinguish optically and can only be distinguished from each other with confidence using electron microprobe analysis. The Fe content of the minerals, determined by electron microprobe analysis, is used to classify these garnets, with crustal garnets typically containing more Fe (Schulze 2005). Similarly, kimberlitic ilmenite is distinguished chemically from crustal ilmenite as kimberlitic ilmenite will contain a minimum of 4% MgO (McClenaghan & Kjarsgaard 2007).

In generating high quality microanalytical data using the electron microprobe, a flat highly polished surface on each mineral grain is a key pre-requisite. Surface roughness impacts data quality in two ways. Firstly, the geometry of x-ray measurement in the wavelength dispersive x-ray spectrometer requires the sample surface, the refracting crystal and the detector to lie on a single plane known as the Rowland circle. Departures from this plane cause defocusing of the characteristic x-rays at the detector resulting in a loss of precision. This loss is less of a concern where the spectrometers are mounted at an inclined plane to the specimen holder, but most electron microprobes are equipped with vertically mounted spectrometers. The second problem caused by surface roughness is that of variable absorption in the x-ray path to the spectrometer. This problem has more of an impact on lower energy x-rays which are characteristic of light elements such as Na, Mg, Al, and Si and also results in a loss of precision. To ensure greater precision and accuracy of microprobe data, the grains must be properly prepared in polished sections with a mirror like finish. The most common problems encountered in preparing indicator minerals for microprobe analysis is the variable hardness among indicator minerals and occasionally, the indicator minerals being quite small. The following section illustrates some ways of overcoming these problems.

How are indicator minerals prepared for electron microprobe analysis?

Two types of sections are commonly used to mount indicator minerals for electron microprobe analysis: 1) bird's eye polished sections and 2) polished grain mounts. Bird's eye polished sections, so-named for their appearance, are used for very fine indicator minerals (<0.25 mm grains). Polished grain mounts are the more common method for mounting coarse indicator minerals (>0.25 mm grains). Typically sections can be 25 mm or 30 mm in diameter and grains are exposed across the entire surface of the section, however polished sections of any diameter or thickness can

be produced to accommodate any microprobe.

- 1) A bird's eye section is used if there is at least 0.5 g of sample. For example, in mineralogical studies of gold ore, the gold is separated from the gangue using a series of gravity separation techniques (Zhou et al. 2004). This procedure will result in a very small, often fine-grained, concentrate. Bird's eye polished sections are often produced from these concentrates for mineralogical examination. When used for indicator mineral analysis, each bird's eye polished section will only contain one indicator mineral species from one sample. The section is made from a 25 mm diameter resin plug. A small diameter, shallow indent is created in the centre of the plug using a lathe (Fig. 1). This indent can be of any diameter, and will depend on the

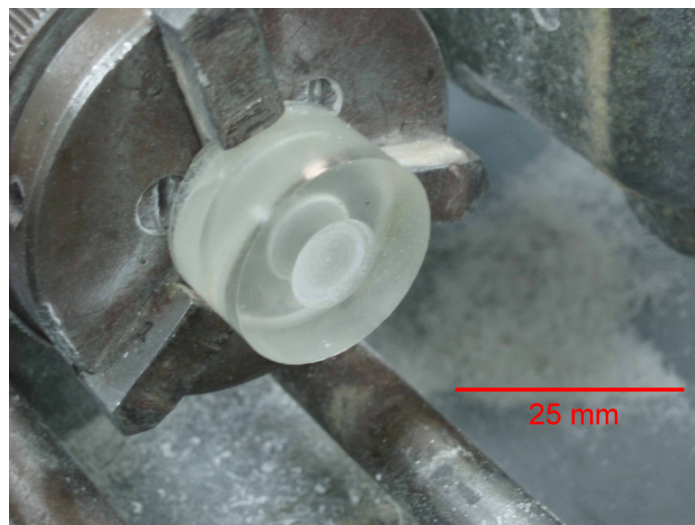


Figure 1: A 25 mm diameter resin plug is used for preparation of a bird's eye polished section. A lathe is used to make a small diameter divot in the surface of the plug.

amount of material available for mounting. The indicator mineral grains are placed in this divot and a small amount of resin is added. The sample is mixed with resin in the divot using a toothpick and the section is placed in a pressure vessel to cure. Once hardened, the section is prepared for polishing, beginning with light grinding to level out the surface and expose the grains. After grinding, the sections are finished on a polishing machine where progressively finer diamond pastes are used to ensure a mirror finish, free of scratches (Fig. 2). In preparing a bird's eye polished section, some indicator mineral grains will be lost during grinding and polishing and some grains will not be exposed at the surface for electron microprobe analysis.

- 2) The more common specimen mounts used for indicator minerals are polished grain mounts. This method is used for grains >0.25 mm in size. Different types of indicator minerals from many different samples are placed on the same mount, making organization during mounting essential. For kimberlite indicator minerals, oxides are separated from silicates as different analytical routines are utilized by the microprobe software for

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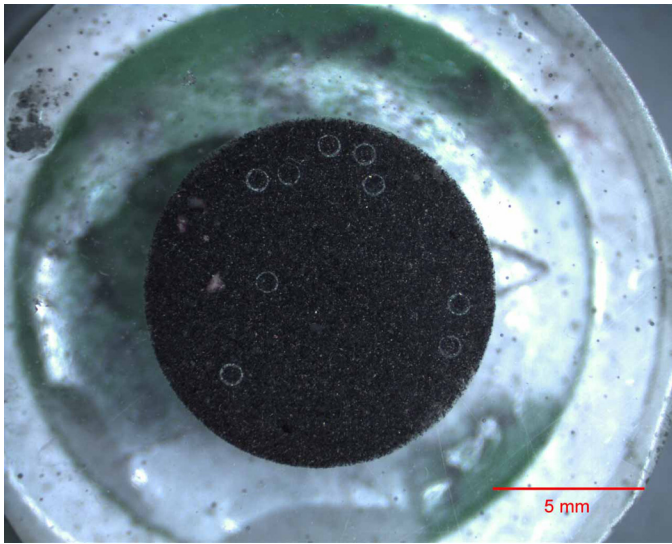


Figure 2: Completed bird's eye polished section prepared from a heavy mineral concentrate. Grains of interest have been scribed with a circle.

these mineral groups. Grains are separated by hardness as well as by size, with a separate mount for 0.25 to 0.5 mm, 0.5 mm to 1.0 mm and 1.0 to 2.0 mm sized grains. This separation helps prevent grain loss due to plucking during the final grinding and polishing process. Following indicator mineral selection, recovered grains are placed in vials or on grain cards. Each grain is manually placed on a glass slide, covered with transparent double-sided tape, and their location is recorded on a spreadsheet for easy reference. The slide is placed over a fine grid to ensure the grains are arranged in straight lines (Fig. 3). Binary grains can be specifically arranged so that both phases will be exposed

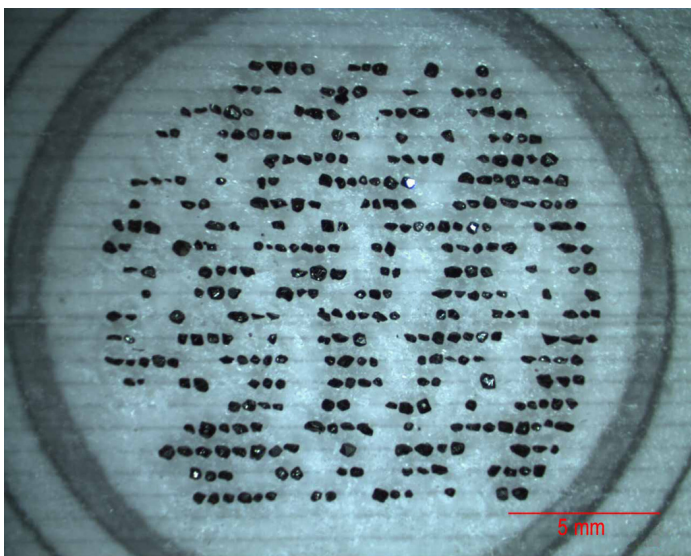


Figure 3: For a polished grain mount, grains are initially arranged right to left on a glass slide. The completed section will be the mirror image, with grains running from left to right.

for electron microprobe analysis. Grains are placed in each row moving from right to left, keeping in mind that the tape represents the surface of the polished section. The finished section will be the mirror image, with grains running from left to right. A significant space is left between grains from different samples. When all grains have been mounted on the slide, a 25 mm diameter mold is placed on the slide. Resin is poured into the mold; low viscosity resin is used to ensure all grains are completely covered. The section is placed in a pressure vessel to cure. When the resin has hardened, the mold is removed and the glass slide is heated so the resin mount can be removed. The surface of the section is examined for holes. Pits must be backfilled before grinding to prevent grains from being plucked from the section. Minerals in the mount are carefully exposed by grinding prior to polishing. Similar to bird's eye polished sections, a polishing machine is used with progressively finer polishing compounds to produce a smooth surface (Fig. 4). The section is checked often during grinding and polishing using a reflected light microscope to prevent grain loss. The under side of the grain mount can also be polished so the general morphology of the minerals can be observed.

Why is careful section preparation important?

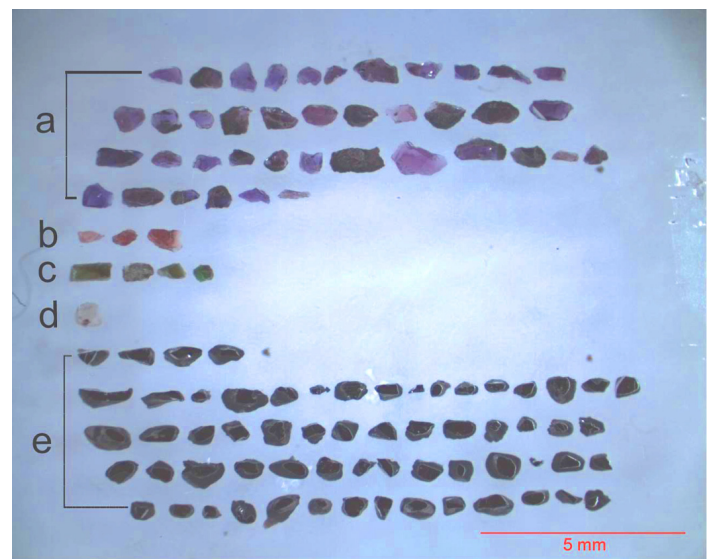


Figure 4: Completed 25 mm diameter polished grain mount. Labeled grains are: a. pyrope garnet; b. eclogitic garnet; c. chrome diopside; d. olivine; e. oxides.

A great deal of effort and expense goes into an indicator mineral sampling program and while section preparation may seem a very small part of the process, it is nonetheless very important, as the loss or misidentification of a few grains could mean that considerable funds will have been wasted and anomalies missed. Organization is essential to the successful preparation of indicator minerals for electron microprobe analysis and it is not a task that should be completed by someone who lacks this skill. Care must be taken at all steps in the preparation process to ensure grains are not mislabeled or lost during polishing or the entire

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exploration program may be set back.

Improper section preparation may also yield inaccurate microprobe results which may in turn lead to erroneous exploration decisions. For example, in diamond exploration, thermobarometric data calculated from inaccurate electron microprobe data might indicate a favorable geotherm for diamond preservation, plotting just inside the diamond stability field when in reality, the grains are not. Further exploration including drilling and microdiamond analysis may be undertaken when, in fact, accurate data may not have warranted additional work. Conversely, properties may be relinquished because electron microprobe analysis may incorrectly suggest that the mineral chemistry of grains recovered from the mineral concentrates is not sufficiently indicative of diamond content. Both situations are possible if the electron microprobe data are obtained from the surface of a poorly prepared grain mount. Similarly, the loss or misidentification of a single indicator mineral during section preparation can have very detrimental effects.

While section preparation is a simple step in part of a complex exploration program, it must be carried out properly and consistently over time to ensure reliable geochemical data are obtained from each indicator mineral.

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