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# A new method for fracturing mineral particles for cross-sectional FESEM analysis

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Abstract. — Cross-sectional samples of mineral particles observed in a field emission scanning electron microscope (FESEM) can provide information on their shape, dimensions, and crystal growth that is difficult to obtain with other preparation methods and analytical techniques. A novel approach for the preparation and measurement of mineral particles in cross-section was investigated in this work. The unique sample preparation method involves vacuum embedding the mineral particles in epoxy and mechanically fracturing the mineral particles across their lengths. The morphology and characteristics of the particle cross-sections can be directly observed in a FESEM. The dimensions of the particle cross-sections can then be measured from the FESEM images off-line with a commercial software package. The procedure was used to characterize a chrysotile standard reference material, South African crocidolite and an amosite standard reference material. The major and minor cross-section chord lengths were recorded for 25 particles for each of the three samples. The chrysotile sample had average major and minor cross-section chord lengths of 0.06 μm and 0.05 μm, respectively. The South African crocidolite sample had average major and minor cross-section chord lengths of 0.09 μm and 0.07 μm, respectively. The amosite sample had average major and minor cross-section chord lengths of 0.23 μm and 0.15 μm, respectively. The chrysotile sample had the smallest cross-sectional chord measurements, while the amosite had the largest cross-sectional chord measurements. The average chord cross-section ratios of the major to minor chord lengths were 1.1 for chrysotile, 1.4 for crocidolite and 1.7 for amosite. The chord cross-section ratios illustrate that chrysotile has a nearly circular cross-section in comparison to crocidolite and amosite.

Riassunto. — L'osservazione tramite microscopia elettronica in campo di emissione (FESEM) di campioni di particelle minerali tagliate trasversalmente all'allungamento può fornire informazioni sulla forma, dimensione e crescita dei cristalli, difficoltose da ottenere invece con altri metodi di preparazione e altre tecniche analitiche. In questo lavoro è stato indagato un nuovo approccio per la preparazione e per la misura delle particelle minerali in sezioni trasversali. Questo metodo esclusivo di preparazione del campione prevede un inglobamento delle particelle minerali in resina epossidica sotto vuoto e una fratturazione meccanica delle stesse, trasversalmente alla loro lunghezza. Tramite immagini FESEM è possibile osservare direttamente la morfologia e i caratteri delle particelle minerali tagliate in modo trasversale, mentre le loro

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dimensioni (spessori) possono essere misurate con un pacchetto software commerciale. La procedura è stata utilizzata per caratterizzare diversi materiali: uno standard di crisotilo, una crocidolite del Sud Africa e uno standard di amosite. Per 25 particelle di ognuno dei tre campioni sono state raccolte le misure delle lunghezze maggiori e minori delle sezioni trasversali. La media delle lunghezze maggiori e minori delle sezioni risultarono essere rispettivamente di: 0,06 μm e 0,05 μm per il crisotilo, 0,09 μm e 0,07 μm per la crocidolite del Sud Africa e 0,23 μm e 0,15 μm per il campione di amosite standard. Relativamente alle sezioni trasversali misurate, il campione di crisotilo risultò avere le misure più piccole, mentre l'amosite quelle più grandi. I rapporti medi delle lunghezze maggiori e minori risultarono di 1,1 per il crisotilo, 1,4 per la crocidolite e 1,7 per l'amosite. Tali rapporti evidenziarono che il crisotilo ha una sezione quasi circolare rispetto a quella della crocidolite e dell'amosite.

Key words: *cross-section, scanning electron microscopy, SEM, field emission scanning electron microscopy, FESEM, amosite, chrysotile, crocidolite, fracture*

## Introduction

The purpose of this study was to: 1) develop a reliable sample preparation procedure for cross-section mineral particle characterization and measurements; 2) identify a procedure to quantitatively measure the mineral particle cross-sections; and 3) to prepare, characterize and measure the cross-sections of three commonly used asbestos minerals formally used in manufactured products. Different sample preparation techniques for embedding the mineral particles in epoxy were developed and tested. Vacuum impregnation of the mineral particles in epoxy proved to be the most suitable for embedding and producing clean fractures of the particles. The morphology and characteristics of the particle cross-sections were directly observed in a field emission scanning electron microscope (FESEM). The dimensions of the particle cross-sections were measured from the secondary electron (SE) FESEM images off-line using AnalySIS© Version 3.2 software.

In previous studies, the native ends of groups of fibers and individual crystals have been observed in the optical microscope, polarized light microscope (PLM) and the scanning electron microscope (SEM) (King, 1994; Bandli and Gunter, 2001; Gunter *et al.*, 2007; Addison, 2008). Native ends of individual airborne particles have also been characterized in the FESEM. The airborne particulate was collected on mixed cellulose ester (MCE) filters. The MCE filters were carbon coated and dissolved in acetone, embedding the particles in the carbon film. The carbon film replica was then mounted on a 400 mesh locater transmission electron microscope (TEM) grid. Depending on the orientation of the particles on the prepared carbon replica, the native ends of various particles were imaged and characterized (Strohmeier *et al.*, 2007; Harris *et al.*, 2007).

Other procedures have been used to study the cross-sections of mineral particles. Cross-sectional samples of synthetic glass fibers have been analyzed in the SEM. The glass fibers were cured in an epoxy resin, cut completely through perpendicular to the fiber axis, and polished (Talbot *et al.*, 2000). Cross-sectional samples of synthetic tremolite crystals, chrysotile asbestos, and anthophyllite asbestos have been prepared for TEM. The synthetic tremolite crystals were dispersed in acetone and mounted on holey-carbon substrates. In order to expose the cross-sections of the tremolite, portions of the sample were embedded in epoxy resin and ion-milled (Ahn *et al.*, 1991). The chrysotile fibers were embedded in methyl methacrylate and cross-sectioned with an ultramicrotome with a diamond knife (Yada, 1967). Thin specimens of anthophyllite asbestos were prepared by argon-ion milling pieces of a petrographic thin section cut normal to the fibers (Veblen, 1980). However, this is the first study that involves vacuum impregnation of mineral fibers in epoxy and mechanical fracturing across the length of the fibers in order to view and measure freshly prepared cross-sections in a FESEM.

## Studied Materials

Three asbestos samples were analyzed in this study, including the National Institute of Standards and Technology (NIST) chrysotile 1866 Standard Reference Material (SRM), ore grade South African crocidolite, and NIST amosite 1866 SRM. Chrysotile belongs to the serpentine mineral group, while crocidolite and amosite belong to the amphibole mineral group. These samples were chosen because they represent the three major types of commercial asbestos formally used in the United States (Virta, 2006).

## Methods and Techniques

### *Cross-Sectional Preparation of Mineral Particles*

Multiple techniques for embedding and fracturing the mineral particles were developed and tested. Originally, the samples were prepared in a Lucite® mount and cured for eight minutes in a Buehler SimpliMet® 3000 Automatic Mounting Press at 360 °F and 4200 psi. However, when the mineral particles were fractured, significant pull out of the particles was occasionally observed. Vacuum impregnation of the mineral particles in epoxy proved to be more suitable for embedding and producing clean fractures of the particles. The sample preparation method includes two steps: 1) vacuum impregnation of the mineral particles in epoxy; and 2) mechanical fracture of the mineral particles across their lengths. In order to vacuum embed the mineral particles, a 1.5 inch phenolic ring was placed in a plastic cup. The ring was half filled with Buehler Epoxicure™ Resin and cured overnight at room temperature. After the epoxy resin cured for approximately 24 hours, a small sample of mineral particles was placed in the previously created epoxy ring and covered with additional Epoxicure™ resin. The assembly was placed in a Buehler Equipment I Vacuum Impregnator and evacuated to 15 inches of mercury. After the pressure reached ~$10^{-2}$ Torr, the vacuum was released and evacuated again for two additional cycles during the next ten minutes. This procedure removes air from the particle sample and allows the epoxy to flow around and surround the particles. The embedded sample was then allowed to cure for an additional 8 hours prior to mechanical fracture, as shown in Fig. 1a.

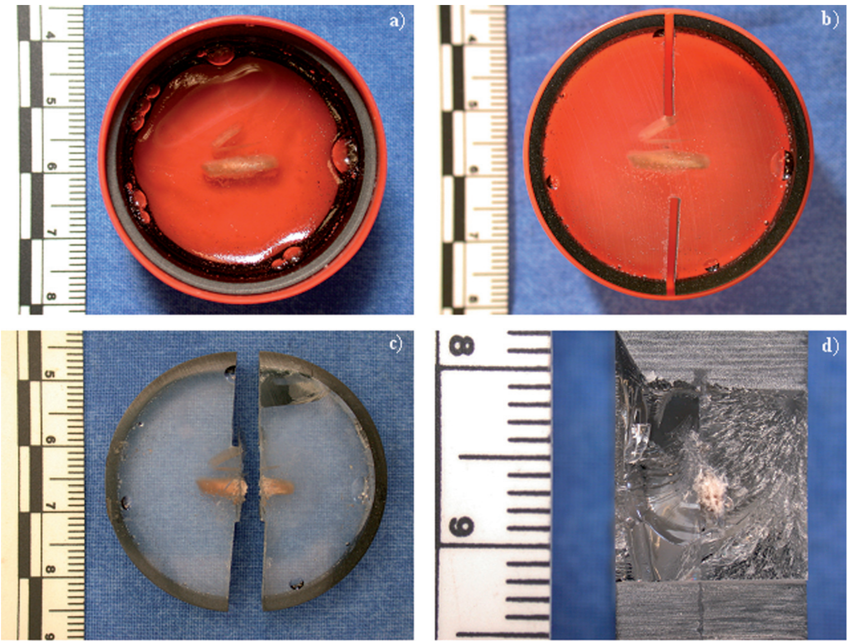


Fig. 1 – Optical images of the a) mineral particles vacuum impregnated in the phenolic ring mount, b) sample mount after a band saw was used to cut two notches on either side of the particles, c) sample after being fractured across the length of the particles, and d) cross-sectional view of the particles in the sample mount. The ruler with millimeter units is shown for scale.

In order to mechanically fracture the sample, a band saw was used to cut notches perpendicular to the particle bundle length on each side of the sample, as shown in Fig. 1b. The sample was placed in a vice with the band saw cuts parallel to the top of the vice. The top half of the sample was grasped with pliers and fractured into two pieces, creating two samples with exposed cross-sections of the mineral particles, as shown in Fig. 1c and 1d. It is believed that any resulting deformation of the mineral particles is negligible because of the method of embedding. Prior to analyzing the cross-sectional samples in the FESEM, the samples were platinum coated for 1.5 minutes at 20 mA.

*FESEM Analysis*

The fully prepared cross-sectional samples were analyzed in an FEI Sirion 400 FESEM instrument operated in the "ultrahigh resolution" mode with a Through-the-Lens Detector (TLD) and an accelerating voltage of 3 kV, a beam current of 200 pA, and a working distance of approximately 4.0 mm. SE FESEM images of the mineral particle cross-sections were digitally recorded for subsequent off-line cross-section measurements. Fig. 2a and 2b are SE FESEM images of the chrysotile cross-sectional sample. Fig. 2c and 2d are SE FESEM images of the crocidolite cross-

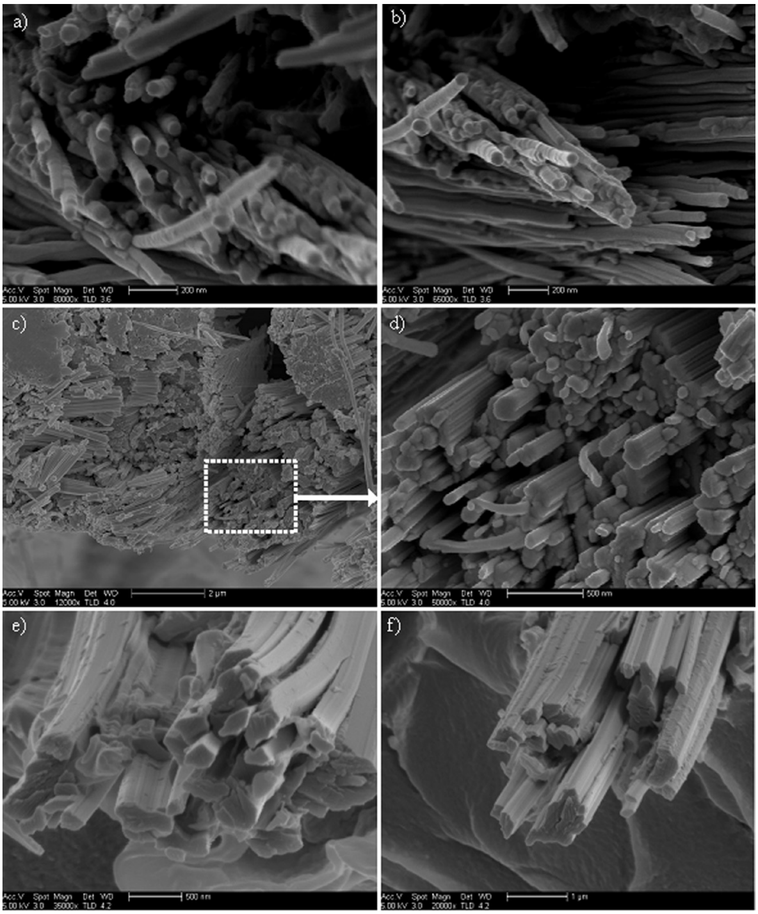


Fig. 2 – Secondary electron FESEM images of chrysotile cross-sections a) and b), crocidolite cross-sections c) and d), and amosite cross-sections e) and f). Scale bars are shown for each image.

sectional sample. Fig. 2e and 2f are SE FESEM images of the amosite cross-sectional sample.

*Particle Cross-Section Measurements*

Dimensions of the particle cross-sections were measured off-line from the digital SE FESEM images using AnalySIS© Version 3.2 software. The AnalySIS© software allows manual chord length measurements to be made directly on the SE FESEM particle cross-sectional images. Fig. 3 illustrates major and minor chord measurement overlays on the chrysotile cross-section SE FESEM image. As each chord is drawn on the SE FESEM image using AnalySIS©, the software overlays and sequentially identifies the chord on the image and simultaneously records the chord length measurement in an associated Microsoft® Office Excel file. Major and minor chord lengths were recorded and chord cross-section ratios (major chord length/minor chord length) were calculated for 25 particles for each sample, as shown in Table 1.

## Results and Discussion

The SE FESEM images of the chrysotile, crocidolite, and amosite particles shown in Fig. 2 reveal cross-sections with a wide variety of shapes, surfaces, and growth directions. The chrysotile particles appear to have circular cross sections, while the crocidolite and amosite particles have cross-sections ranging from circular to rectangular in cross section, which is consistent with previous studies (Steel and Wylie, 1981). In addition, all samples display particles exhibiting curvature. The measurements of the particle cross-sections produced by the AnalySIS© Version 3.2 software show a range of sizes depending on the sample under study. The major and minor chord length

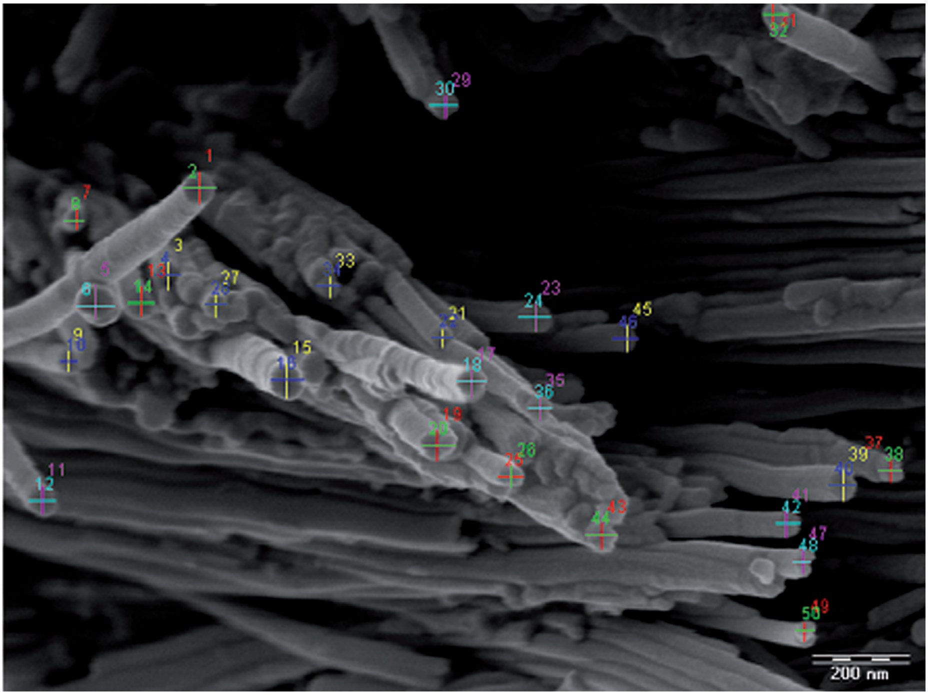


Fig. 3 – A secondary electron FESEM image of the NIST chrysotile sample illustrating major and minor chords positioned on particle cross-sections. As each chord is drawn on the AnalySIS© software overlay, the number of the drawn chord is displayed on the image. The numeric values of the chord lengths are recorded to a separate Excel file.

measurements as well as average, maximum, minimum and standard deviation calculations for 25 particles for each of the chrysotile, crocidolite and amosite samples are shown in Table 1.

TABLE 1 — *Fiber cross-section measurements for chrysotile 1866, South African crocidolite, and amosite 1866 made using AnalySIS© software*

| | Chrysotile 1866 | | | South African Crocidolite | | | Amosite 1866 | | |
|---|---|---|---|---|---|---|---|---|---|
| Particle Count | Major Chord | Minor Chord | Chord Ratio | Major Chord | Minor Chord | Chord Ratio | Major Chord | Minor Chord | Chord Ratio |
| 1 | 0.07 | 0.07 | 1.0 | 0.17 | 0.16 | 1.1 | 0.39 | 0.14 | 2.8 |
| 2 | 0.06 | 0.05 | 1.2 | 0.12 | 0.11 | 1.1 | 0.27 | 0.31 | 0.9 |
| 3 | 0.08 | 0.07 | 1.1 | 0.07 | 0.06 | 1.2 | 0.22 | 0.12 | 1.8 |
| 4 | 0.04 | 0.04 | 1.0 | 0.08 | 0.08 | 1.0 | 0.36 | 0.17 | 2.1 |
| 5 | 0.04 | 0.03 | 1.3 | 0.09 | 0.08 | 1.1 | 0.29 | 0.23 | 1.3 |
| 6 | 0.06 | 0.05 | 1.2 | 0.09 | 0.08 | 1.1 | 0.20 | 0.13 | 1.5 |
| 7 | 0.06 | 0.05 | 1.2 | 0.08 | 0.07 | 1.1 | 0.22 | 0.17 | 1.3 |
| 8 | 0.07 | 0.07 | 1.0 | 0.08 | 0.07 | 1.1 | 0.37 | 0.23 | 1.6 |
| 9 | 0.06 | 0.05 | 1.2 | 0.05 | 0.04 | 1.3 | 0.25 | 0.11 | 2.3 |
| 10 | 0.07 | 0.06 | 1.2 | 0.09 | 0.07 | 1.3 | 0.16 | 0.14 | 1.1 |
| 11 | 0.04 | 0.04 | 1.0 | 0.13 | 0.10 | 1.3 | 0.10 | 0.09 | 1.1 |
| 12 | 0.07 | 0.06 | 1.2 | 0.10 | 0.08 | 1.3 | 0.14 | 0.14 | 1.0 |
| 13 | 0.05 | 0.04 | 1.3 | 0.09 | 0.07 | 1.3 | 0.21 | 0.11 | 1.9 |
| 14 | 0.05 | 0.04 | 1.3 | 0.07 | 0.06 | 1.2 | 0.21 | 0.21 | 1.0 |
| 15 | 0.06 | 0.04 | 1.5 | 0.09 | 0.07 | 1.3 | 0.22 | 0.15 | 1.5 |
| 16 | 0.05 | 0.05 | 1.0 | 0.09 | 0.07 | 1.3 | 0.23 | 0.14 | 1.6 |
| 17 | 0.05 | 0.04 | 1.3 | 0.08 | 0.06 | 1.3 | 0.14 | 0.14 | 1.0 |
| 18 | 0.05 | 0.05 | 1.0 | 0.10 | 0.07 | 1.4 | 0.15 | 0.08 | 1.9 |
| 19 | 0.05 | 0.05 | 1.0 | 0.08 | 0.05 | 1.6 | 0.20 | 0.11 | 1.8 |
| 20 | 0.06 | 0.06 | 1.0 | 0.10 | 0.07 | 1.4 | 0.21 | 0.10 | 2.1 |
| 21 | 0.05 | 0.05 | 1.0 | 0.09 | 0.05 | 1.8 | 0.30 | 0.13 | 2.3 |
| 22 | 0.07 | 0.05 | 1.4 | 0.07 | 0.04 | 1.8 | 0.14 | 0.08 | 1.8 |
| 23 | 0.05 | 0.05 | 1.0 | 0.11 | 0.06 | 1.8 | 0.39 | 0.16 | 2.4 |
| 24 | 0.05 | 0.04 | 1.3 | 0.08 | 0.04 | 2.0 | 0.18 | 0.10 | 1.8 |
| 25 | 0.04 | 0.04 | 1.0 | 0.14 | 0.07 | 2.0 | 0.30 | 0.16 | 1.9 |
| Average | 0.06 | 0.05 | 1.1 | 0.09 | 0.07 | 1.4 | 0.23 | 0.15 | 1.7 |
| Maximum | 0.08 | 0.07 | 1.5 | 0.17 | 0.16 | 2.0 | 0.39 | 0.31 | 2.8 |
| Minimum | 0.04 | 0.03 | 1.0 | 0.05 | 0.04 | 1.0 | 0.10 | 0.08 | 0.9 |
| Std Dev. | 0.01 | 0.01 | 0.1 | 0.02 | 0.02 | 0.3 | 0.08 | 0.05 | 0.5 |

The data shown in Table 1 reveal increasing particle cross-section dimensions from chrysotile through crocidolite to amosite. The chrysotile sample had average major and minor cross-section chord lengths of 0.06 µm and 0.05 µm, respectively. The crocidolite sample had average major and minor cross-section chord lengths of 0.09 µm and 0.07 µm, respectively. The amosite sample had average major and minor cross-section chord lengths of 0.23 µm and 0.15 µm, respectively. The amphibole particles have a larger cross-section and tend to be more variable in width, as seen in previous studies (Steel and Wylie, 1981). Cross-section particle measurements from Table 1 are graphically displayed in Fig. 4. The plot illustrates the major and minor cross-section chord lengths in microns for the chrysotile, crocidolite, and amosite samples.

Fig. 5 is a plot of the major/minor chord cross-section ratios as a function of the average major chord length. The theoretical chord cross-section ratio for a particle with a circular cross-section is 1, while the theoretical chord cross-section ratio formed by {110} amphibole cleavage is 1.77. The data demonstrates that chrysotile has a nearly circular average chord cross-section ratio of 1.1; by contrast, both crocidolite and amosite have non-circular average chord cross-section ratios of 1.4 and 1.7 respectively, in which the major chord dimensions are significantly greater than the minor chord dimensions. In the case of amosite, the average chord cross-section ratio is close to that formed by the theoretical amphibole {110} cleavage faces.

## Conclusions

The novel sample preparation method described has proven to be consistent and repeatable in providing suitable cross-sectional mineral particle samples for FESEM analysis. Observing cross-sections of mineral particles in the FESEM provides information on the shape, dimensions, and crystal growth that is difficult to obtain with other preparation methods and analytical techniques.


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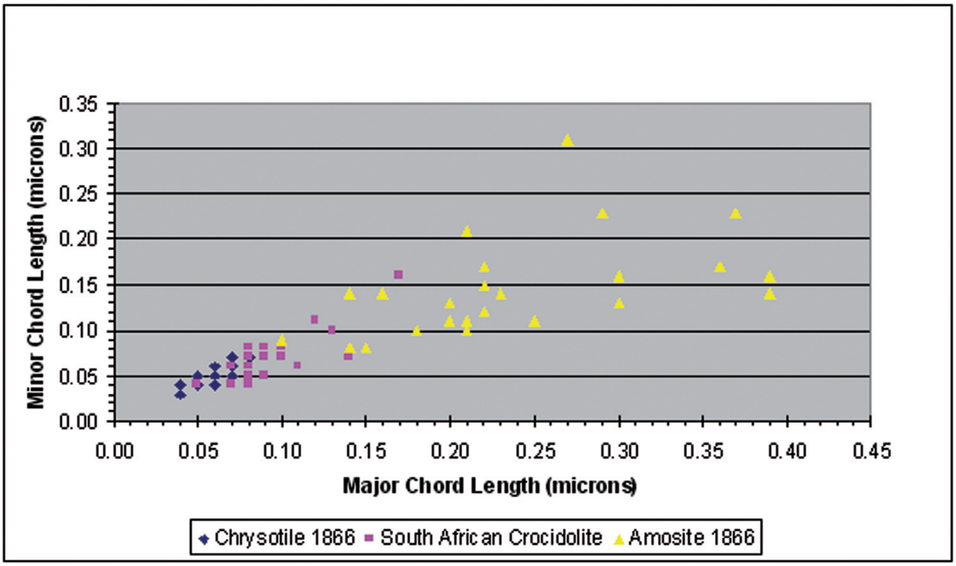


Fig. 4 – Asbestos particle cross-section chord measurements for chrysotile 1866, South African crocidolite, and amosite 1866.

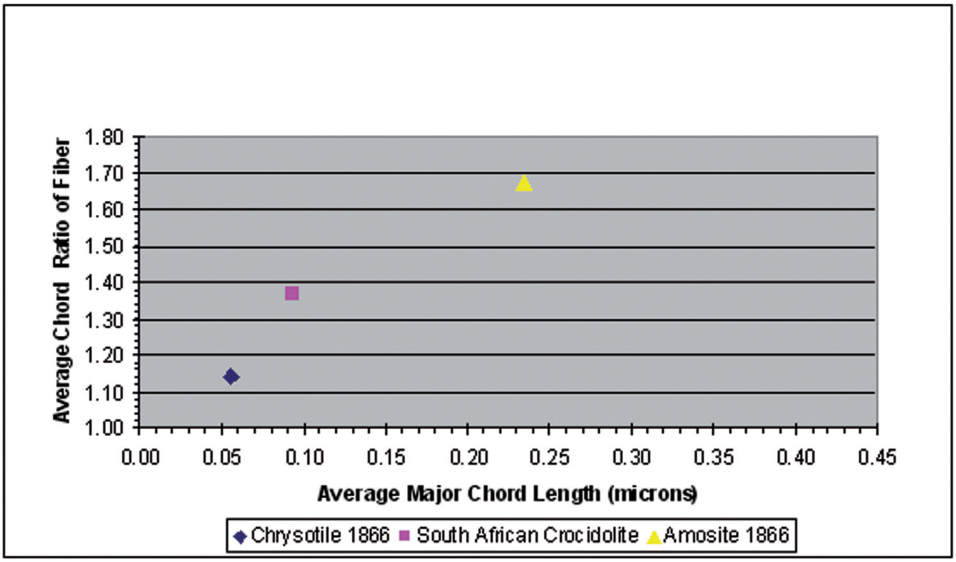


Fig. 5 – Plot of average chord ratio (major chord/minor chord) versus average major chord length for chrysotile 1866, South African crocidolite, and amosite 1866.

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