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THE PREPARATION OF MICA CYLINDERS FOR MEASUREMENT OF THERMAL CONDUCTIVITY

A.G. MCDONALD AND V.V. MIRKOVICH

INDUSTRIAL MINERALS LABORATORY
CERAMIC SECTION

DECEMBER 1975

MINERALS RESEARCH PROGRAM

MINERAL SCIENCES LABORATORIES

CANMET REPORT 76-4

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Catalogue No. M38-9/76-4

Nº de catalogue M38-9/76-4

Price subject to change without notice

Prix sujet à changement sans avis préalable

Information Canada
Ottawa, 1975

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A.G. McDonald* and V.V. Mirkovich**

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SUMMARY

Measurements of thermal conductivity parallel with the basal {001} cleavage planes of the industrially important mica minerals have not been made in the past because of the difficulty of sample preparation. A method was devised to produce cylindrical mica specimens, 25.4 mm in diameter x 25.4 mm high, suitable for use in a comparative method apparatus for measurement of thermal conductivity. The mica cylinders can be prepared so that the planes of cleavage are either parallel or perpendicular to their longitudinal axis.

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*Technician, **Research Scientist, Mineral Sciences Laboratories, Canada Centre for Mineral and Energy Technology, Department of Energy, Mines and Resources, Ottawa, Ontario, Canada.

LA PREPARATION DE CYLINDRES EN MICA POUR MESURER LA CONDUCTIVITE THERMIQUE

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A.G. McDonald* et V.V. Mirkovich**

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RESUME

Autrefois, le mesurage de la conductivité thermique parallèle aux plans de clivage principaux {001} des minéraux de mica d'importance industrielle ne se faisait pas à cause de la difficulté de préparation des échantillons. Une méthode a été inventée afin de produire des spécimens cylindriques en mica, de 25.4 mm de diamètre par 25.4 mm de hauteur, appropriés pour leur utilisation dans un appareil de méthode comparative pour le mesurage de la conductivité thermique. Les cylindres en mica peuvent être préparés de sorte que les plans de clivage soient ou parallèles ou perpendiculaires à leur axe longitudinal.

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*Technicien, **Chercheur scientifique, Laboratoires des sciences minérales, Centre canadien de la technologie des minéraux et de l'énergie, Ministère de l'Energie, des Mines et des Ressources, Ottawa, Ontario, Canada.

INTRODUCTION

Because of their layered atomic structure (1,2), the relatively weak bonds holding the layers together, the high flexibility and elasticity of the folia, and the perfect basal {001} cleavage, micas can be split into extremely thin sheets. This characteristic, however, can be an obstacle in the measurement of some physical properties, as it is often required that the sample's geometrical configuration satisfies the measuring device. In thermal conductivity measurements, accurate dimensions of the specimen are of particular importance. The difficulty in preparing mica specimens in which heat would flow parallel with the schistosity of the mineral was undoubtedly the major reason that thermal conductivity values reported in the literature have been generally limited to measurements performed normal to the {001} cleavage, i.e., the flat surface of the micas (3,4,5,6,7). Horai (8) applied the needle-probe technique to a mixture of powdered mica and water in an attempt to measure an average thermal conductivity. The anisotropy of the thermal conductivity of micas proposed by Jannettaz (9,10) was studied by Goldsmid and Bowley (11). The latter indirectly obtained values for thermal conductivity of micas parallel to {001} by measuring thermal diffusivity. They found the difference in conductivities parallel and perpendicular to {001} to be one order of magnitude. Also, all thermal conductivity measurements were obtained for micas at or near room temperature except for the work by Griffiths, Powell and Hickman (6). Their measurements of thermal conductivity were made in the

100° to 600°C temperature range, but only in the direction across the cleavage planes.

As stated before, the preparation of specimens of suitable dimensions was the chief impediment to the determination of thermal conductivity parallel to the cleavage planes of mica. Not only do specimens have to conform with the dimensions of the apparatus in which the measurements are to be made but, also, their final dimensions must be sufficiently large to incorporate realistically the overall physical properties of the mica as it occurs naturally. A disc-shaped specimen of 25-mm diameter and 1-mm thickness, such as that used by Griffiths et al. in their cut bar apparatus, would be difficult if not impossible to prepare with the basal cleavage of the mica in any orientation other than parallel to the plane faces of the disc.

The apparatus used for measurements of thermal conductivity of mica (and for which the mica samples had to be prepared) was described previously (12). In essence, the measuring arrangement, schematically shown in Figure 1, consists of two cylindrical standards with a sample in between, all three placed on a heat stabilizer. The standards, the sample, and the heat stabilizer are of the same size. The column thus formed is held between the heat source and the heat sink and is centered within the heat guard. Heat guarding, achieved by matching the temperature of the heat guard to that of the sample column on the same level, is controlled through five heaters. These are wound on the outside of the guard cylinder, on levels corresponding to the heat source and the four cylinders.

The sample used for this apparatus, a 25.4-mm diameter by 25.4-mm high cylinder, is sufficiently large to integrate the contribution of individual mica layers toward its overall thermal conductivity. Furthermore, as the heat flux in this apparatus is unidirectional, the measurement of thermal conductivity of mica can be made, depending on the orientation of the structure of the specimen, either perpendicular to or parallel with the schistosity.

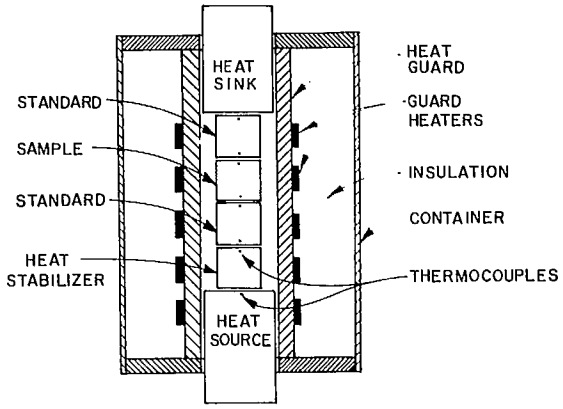

Figure 1. Schematic of the comparative method thermal conductivity apparatus.

The objective of this work was to devise a method of preparing specimens suitable for measurement of thermal conductivity as a function of temperature and direction of measurement. Cylindrical specimens from three types of mica, biotite, phlogopite and muscovite, were prepared with basal cleavages running parallel and perpendicular to the cylinder axis.

SPECIMEN PREPARATION

The mica used in this investigation was obtained from Canadian sources. Table 1 gives the location of the deposits and a description of the specimens.

It was rather difficult to obtain sound blocks of mica

of uniform quality and sufficient size to yield specimens of the required dimensions. However, the greatest impediment in the preparation of the specimens was the very nature of mica: the perfect and easy separation of the folia. To conform with the dimensions of the thermal conductivity apparatus the specimens had to be cut in the form of a cylinder 25.4 mm diameter and 25.4 mm high. Furthermore, as the direction of the heat flow in the thermal conductivity apparatus is only parallel with the axis of the cylindrical specimen, two specimens had to be prepared from each sample: one in which the axis of the cylinder was perpendicular to the planes of cleavage and the other where the axis was parallel with the planes of cleavage.

TABLE 1

Characterization of Micas Used for Specimen Preparation

Specimen	Geographical Origin	Source Rock	Density, g/cm^3	Colour	Transparency
Biotite	Silver Crater Deposit-Bancroft	Carbonatite mass	2.93	Black	Opaque
Phlogopite	Cantley Mica Mines, Cantley-Wakefield, P.Q.	Calcite-apatite vein	2.78	Glossy-brown	Opaque, thin folia translucent
Muscovite	Purdy Mica Mines, Eau Claire-Mattawa, Ont.	Pegmatite	2.73	Light gray	Translucent, thin folia transparent

Of the several methods tested for the preparation of 25.4-mm diameter mica cylinders, the closest to being successful was that in which a solid piece of mica, previously trimmed to a

suitable size, was cemented with an epoxy cement onto the centre of a horizontal disc. A circular diamond saw, perpendicularly oriented with respect to the surface of the rotating disc, was slowly lowered onto the rotating mica sample, cutting and grinding off the sides of the sample. The saw was positioned about 13 mm (0.5 in.) from the axis of the rotating mica sample so that the resulting cylinder was 25.4 mm (1 in.) in diameter. Unfortunately, as the height of the cylinder approached 25.4 mm, probably because of the vibration and stresses induced by cutting and grinding of the cirular saw, the specimens invariably suffered a certain amount of disintegration.

The problem of specimen preparation was finally solved by encapsulating the original mica sample in epoxy resin and core-drilling the cylindrical specimen. A typical chunk of mica, shown in Figure 2, was about 15 to 20 cm long, 10 to 12 cm wide and not less than 3.5 cm thick.

Figure 2. A sample of mica (biotite) before encapsulation in epoxy resin.

After careful visual examination of the striations for homogeneity of the layered structure, flaws, distortions or intergrowth of crystals, the sample was placed in an aluminum-foil-lined cardboard box, the bottom of which already had a cured layer of epoxy resin, 0.5 to 1 cm thick. The epoxy resin was then poured and allowed to harden in quantities sufficient to build only 1-cm thick layers. This gradual build-up was necessary for two reasons: (1) the curing of an epoxy resin is an exothermic process and the rate of curing depends on the temperature of the epoxy. Thus, if the evolved heat of reaction is not removed, it will raise the temperature of the resin, which in turn accelerates the rate of reaction, which then generates more heat and finally may cause the plastic to char and burn; (2) too-high temperature gradients in the mica sample could cause weakening of the layered structure of the mineral.

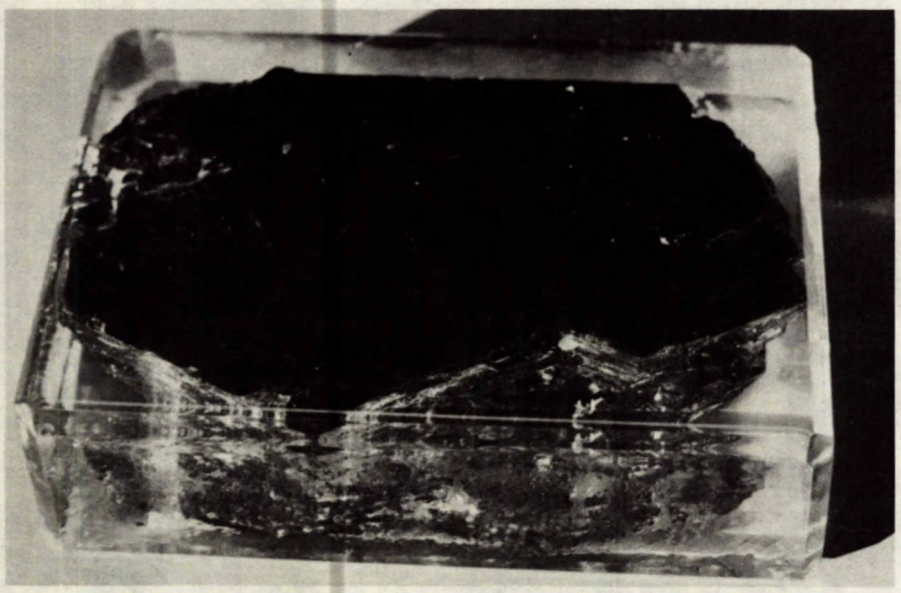

Figure 3. Mica sample from Figure 1 completely encapsulated in epoxy resin.

Epon* 815 and Epon 828, with 10-12% of diethylenetriamine as curing agent, were found to be satisfactory for encapsulating the samples.

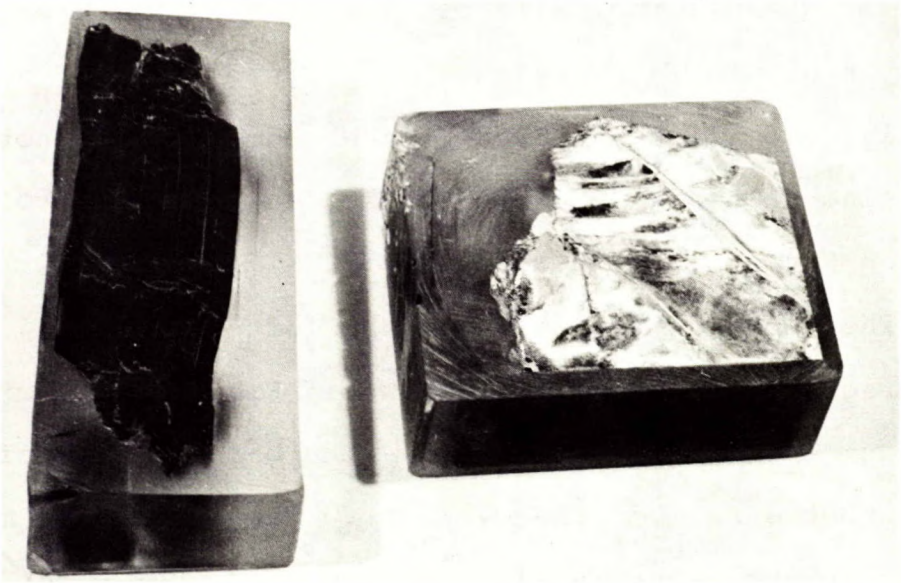

Figure 4. Segments of mica cut from larger samples which were originally completely encased in epoxy resin. The segment on the left (a biotite) is to be used for preparation of cylinders in which the basal cleavages will be parallel with the axis of the cylinder. The cylinders made from the segment on the right (a muscovite) will have the basal cleavages perpendicular to the axis.

After the last layer of epoxy resin had solidified, the mica sample thus completely enveloped by the plastic was removed from the cardboard box and the aluminum foil was peeled off the plastic.. The aluminum foil was used because it facilitated separation of the plastic and the cardboard and, when peeled off, it left the surface of the plastic sufficiently clear to allow viewing

*Epoxy resins manufactured by Shell Oil Company Limited, Chemical Division, Toronto, Ontario.

of the sample. The encapsulated specimen, shown in Figure 3, was once again visually examined for damage which might have occurred during the hardening of the plastic, and then cut, by means of a circular diamond saw, into 28-30-mm wide segments. Without the encasement of the original mica block in epoxy resin, preparation of coherent segments, such as shown in Figure 4, could not be successfully accomplished. Even if the segment would not break up while being sawn off, it would weaken sufficiently to disintegrate during any subsequent cutting.

The mica segment shown on the left in Figure 4 was cut from the original biotite sample shown in Figure 2. The specimen prepared from this segment would have the basal planes of cleavage parallel with the axis of the cylinder. The segment of muscovite mica, shown on the right in Figure 4, on the other hand, was used to prepare a mica cylinder with basal planes of cleavage perpendicular to the axis. The exposed sides of both segments were carefully ground plane-parallel on a diamond impregnated lap wheel so that the resulting thickness of each segment was 25.4 mm ± 0.01, insuring that the ground surfaces were either perpendicular or parallel to the cleavage planes. As the required thickness was approached, progressively finer diamond grit was used and finally the ground surfaces were polished to a fine lustre.

The polished surfaces normal to the cleavage planes were covered with 2 to 4 mm of epoxy resin, which served to prevent delamination of the specimen during subsequent core drilling. The segment was then ground square with respect to the basal planes so that it could be gripped in a vise with the laminations vertical.

The specimen, held in the vise, was submerged in water and a cylindrical specimen was drilled out using a diamond core drill with 25.4-mm inside diameter. Before removing the epoxy plastic from the flat surfaces (top and bottom) of the cylinder with an organic solvent (CH_2Cl_2), two 0.75-mm diameter, 12.7-mm deep thermocouple holes were drilled into the side of the cylinder with an ultrasonic drill. The holes, extending radially from the axis to the surface of the cylinder were 22.9 mm apart so that each hole was 1.3 mm from the end of the cylinder. The finished cylindrical specimen with mica schistosity parallel to the axis of the cylinder is shown in Figure 6.

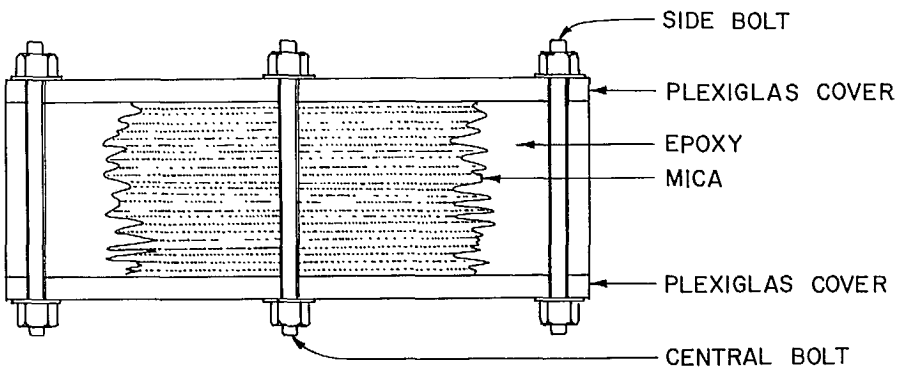

Figure 5. Schematic of the reinforcement arrangement of encapsulated mica sample for core-drilling perpendicular to the cleavage planes.

In the case where the drilling was done perpendicular to the cleavage planes, it was necessary to provide a different reinforcement to the exposed surfaces. As shown in Figure 5, a 2.5-mm diameter hole was drilled perpendicular to the cleavage planes and extending from the top to the bottom of the specimen.

Four additional holes, one in each corner of the segment, were drilled from top to bottom through the encasing epoxy plastic. By means of threaded rods within these holes, each with a washer and nut on either side, the specimen was firmly sandwiched between two 3-mm thick plexiglass plates. The pressure exerted on the sample by this arrangement could be maintained during the core drilling, thus preventing fracture of the specimen along its basal cleavages.

Figure 6. Cylindrical mica specimen (biotite) with schistosity parallel with the axis of the cylinder.

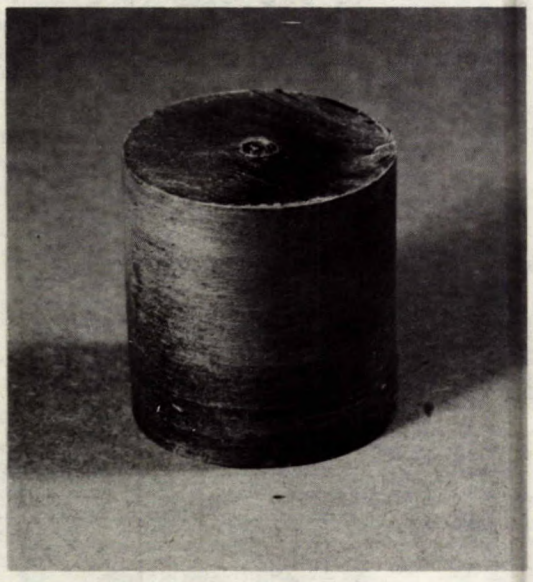

Figure 7. Cylindrical mica specimen (muscovite) with schistosity perpendicular to the axis of the cylinder.

As before, the core-drilling was done in water with the core drill centered about the threaded rod in the mica. Before removing the threaded rod and the plexiglass discs from the cylindrical specimen thus obtained, two thermocouple holes, 0.75 mm in diameter, were drilled in the same positions as in the other case, but to

a depth of only 6 mm. After removing the threaded rod from the 2.5-mm axial hole (which actually represents less than 1 per cent of the cross-sectional area of the cylinder and would therefore not significantly influence the measured thermal conductivity values) it was filled with finely ground mica obtained from the same specimen. Figure 7 shows the finished mica cylinder, 25.4 mm in diameter and 25.4 mm high, with cleavage planes perpendicular to the axis.

CONCLUSION

It has been demonstrated that cylindrical mica specimens, 25.4 mm in diameter and 25.4 mm high, suitable for measurement of thermal conductivity in a comparative method thermal conductivity apparatus, can be made from a single crystal of mica by the described method. The mica cylinders can be made with planes of cleavage either parallel with or perpendicular to the longitudinal axis of the cylinder.

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