

X-RAY DIFFRACTION STUDIES OF SOME MICA SPECIES OF INDIA

BY N. S. NAMPOOTHIRY* AND R. V. G. SUNDARA RAO

(*Department of Physics, Indian Institute of Science, Bangalore-3*)

Received January 23, 1956

ABSTRACT

A number of specimens of different species of Indian mica, including Muscovite, Lepidolite, Biotite, Phlogopite and Mahadevite have been studied by X-ray powder technique, and interplanar spacings and relative intensities of the various patterns have been reported.

1. INTRODUCTION

Study of mica species by X-ray methods mostly using powder techniques had been made by a number of workers, Jackson and West (1931), Nagelschmidt (1943, 1937), Maegdefrau and Ubrich Hofmann (1937), Van Dijke Beatty (1949) and others. Most of the data are recorded in the A.S.T.M. Index. Ramaseshan (1945), while investigating a species of mica of the 'Bison hill range' (Eastern Ghats), established a new species of mica, Mahadevite, largely on the basis of chemical composition and study of some of its optical properties. It is considered worthwhile to investigate the latter along with other species of mica available in various parts of India by X-ray diffraction methods for making a comparative study of the same. For this purpose, therefore, a variety of species including Muscovite, Phlogopite, Biotite, Lepidolite and Mahadevite with different specimens obtained from different sources have been studied and the results are given in this paper.

2. EXPERIMENTAL PROCEDURE

The specimens have been investigated using X-ray diffraction powder technique. Unicam 9 cm. (diameter) powder camera has been used and it was previously calibrated with NaCl powder picture. The mica specimen was ground to very fine powder till the powder lines showed no spotty structure. The powder was mounted on a very fine glass fibre in a base of collodium. Exposure times, tube current and voltage have been kept identical for all the specimens so that comparison could be made under similar conditions.

Since in general the lines were rather broad, the middle point of each line was taken for the purpose of measuring the interplanar distances. The distance between the powder lines was measured by using a pair of dividers and accurate

* Short-time worker from the University of Travancore.

glass scale. The sodium chloride powder pattern used for calibration was very sharp and the measured interplanar distances agreed well within $\pm 0.5\%$ on the average with the standard spacings given for the substance. Since the mica patterns were rather broad and diffuse in general, the accuracy of measurements of the interplanar spacings is lower than in the case of sodium chloride pattern and the maximum error may be $\pm 1\%$. Of course, the measurements in the high-angle regions are theoretically more accurate but this was in part offset by the fact that at high angles, the lines were in general weak and diffuse. $\text{Cu K}\alpha$ radiation was used throughout.

Visual method of estimating the relative intensities of the powder lines was used in the investigation. For this purpose, calibrated intensity scale of sodium chloride pattern with the known intensity ratios was used. Using the same specimen of sodium chloride mounting, four powder pictures under identical tube conditions were taken with exposure times 30, 20, 15, 10 minutes respectively and developed under identical conditions. The four pictures provided a sufficiently accurate intensity scale for measuring the intensities of the mica patterns.

Three different specimens of Muscovite, two of Phlogopite, two of Lepidolite, one of Biotite and two of Mahadevite have been studied. Besides, two other specimens whose identity was not exactly known were also studied. The spacings and the relative intensities of the lines are given in the following tables. Mention must be made, however, that the strongest lines corresponding to the reflections from the cleavage planes are more or less saturated in all the patterns and so estimation of their intensities would necessarily be very approximate. As had been suggested by Van Dijke Beatty (1949) it would probably be much better if in tabulating the relative intensities in mica the two intense lines were omitted.

3. RESULTS

TABLE I

Muscovite

| <i>Specimen (1)</i> | | <i>Specimen (2)</i> | | <i>Specimen (3)</i> | |
|--------------------------|-----------|--------------------------|-----------|--------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 4.33 | 60 | 2.784 | 13 | 4.215 | 47 |
| 3.78 | 2.8 | 2.548 | 120 | 3.27 | 47 |
| 3.27 | 13 | 2.352 | 13 | 2.95 | 16 |
| 2.95 | 8.7 | 2.212 | 13 | 2.784 | 16 |
| 2.784 | 8.7 | 2.121 | 20 | 2.548 | 80 |
| 2.548 | 80 | 1.985 | 20 | 2.385 | 5 |
| 2.385 | 8.7 | 1.638 | 24 | 2.212 | 9 |
| 2.212 | 8.7 | 1.504 | 47 | 2.121 | 12.5 |
| 2.121 | 9 | 1.349 | 7 | 1.961 | 16 |
| 1.961 | 8.7 | 1.300 | 7 | 1.638 | 15 |
| 1.638 | 11 | 1.246 | 6 | 1.504 | 16 |
| 1.504 | 16 | | | 1.349 | 4.5 |
| 1.349 | 4.5 | | | 1.298 | 5 |
| 1.298 | 4.4 | | | 1.246 | 4.4 |
| 1.246 | 4.5 | | | | |

TABLE II
Phlogopite

| Specimen (1) | | Specimen (2) | |
|--------------------------|-----------|--------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 4.33 | 6 | 3.27 | 70 |
| 3.24 | 80 | 2.591 | 70 |
| 2.574 | 70 | 2.386 | 24 |
| 2.386 | 24 | 2.245 | 6.5 |
| 2.245 | 5 | 2.152 | 35 |
| 2.152 | 24 | 1.985 | 24 |
| 1.985 | 24 | 1.671 | 24 |
| 1.671 | 18 | 1.525 | 47 |
| 1.525 | 24 | 1.37 | 9.5 |
| 1.370 | 7 | 1.319 | 6 |
| 1.319 | 5 | 1.134 | 4.5 |
| 1.309 | 5 | 1.003 | 2.2 |
| 1.263 | 5 | 0.994 | 2.2 |
| 1.134 | 2.2 | 0.976 | 2.2 |
| 1.003 | 2.2 | 0.914 | 2.2 |
| 0.994 | 2.2 | 0.890 | 2.2 |
| 0.976 | 2.2 | | |
| 0.928 | 1.8 | | |
| 0.890 | | | |

TABLE III

Lepidolite

| <i>Specimen (1)</i> | | <i>Specimen (2)</i> | | | |
|--------------------------|-----------|--------------------------|-----------|--------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 3.27 | 27 | 4.33 | 35 | 1.985 | 16 |
| 2.548 | 7 | 3.27 | 35 | 1.671 | 16 |
| 2.121 | 2.2 | 2.95 | 5 | 1.504 | 19 |
| 1.985 | 4.4 | 2.784 | 6.2 | 1.349 | 6 |
| 1.824 | 1.8 | 2.548 | 60 | 1.290 | 6.2 |
| 1.638 | 1.8 | 2.213 | 5 | 1.246 | 5 |
| 1.490 | 1.8 | 2.121 | 13 | | |

TABLE IV

Biotite

| <i>Specimen (1)</i> | | <i>Specimen (2)</i> | |
|--------------------------|-----------|--------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 4.33 | 2.2 | 1.525 | 35 |
| 3.27 | 40 | 1.37 | 7 |
| 2.591 | 40 | 1.319 | 6 |
| 2.55 | 0.9 | 1.134 | 1.8 |
| 2.386 | 16 | 1.096 | 1.8 |
| 2.245 | 5 | 1.008 | 2.2 |
| 2.152 | 18 | 0.994 | 2.2 |
| 1.985 | 16 | 0.976 | 1.8 |
| 1.868 | 0.9 | 0.914 | 4.4 |
| 1.671 | 19 | 0.892 | 4.4 |

TABLE V
Mahadevite

| <i>Specimen (1)</i> | | <i>Specimen (2)</i> | |
|--------------------------|-----------|--------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 4.33 | 5 | 4.33 | 4.4 |
| 3.27 | 80 | 3.27 | 24 |
| 2.836 | 2.8 | 2.591 | 35 |
| 2.591 | 120 | 2.464 | 1.8 |
| 2.464 | 3.4 | 2.386 | 6.5 |
| 2.386 | 16 | 2.245 | 4.4 |
| 2.245 | 8.7 | 2.152 | 16 |
| 2.152 | 47 | 1.985 | 15 |
| 1.985 | 47 | 1.671 | 16 |
| 1.671 | 47 | 1.525 | 20 |
| 1.531 | 70 | 1.36 | 6 |
| 1.36 | 18 | 1.319 | 4.5 |
| 1.329 | 6.5 | 1.134 | 1.8 |
| 1.3095 | 6.5 | 1.096 | 1.8 |
| 1.221 | 2.5 | 0.994 | 1.8 |
| 1.134 | 6.5 | 0.974 | 0.9 |
| 1.067 | 2.8 | 0.914 | 0.9 |
| 1.008 | 6.5 | 0.890 | 0.9 |
| 0.978 | 7.0 | | |
| 0.917 | 5.0 | | |
| 0.892 | 4.5 | | |
| 0.854 | 2.8 | | |
| 0.841 | 2.8 | | |
| 0.824 | 0.9 | | |

Two specimens whose identity is not exactly known were also studied. One of them was of uniform bluish green colour with the usual vitreous lustre and a specimen of about 1 mm. thick is completely opaque. The results of the two are given in Table VI.

TABLE VI

| <i>Bluish green variety</i> | | <i>Unidentified specimen</i> | |
|-----------------------------|-----------|------------------------------|-----------|
| Spacing in Å units | Intensity | Spacing in Å units | Intensity |
| 3.3 | 60 | 4.2 | 60 |
| 2.783 | 5.5 | 3.2 | 60 |
| 2.547 | 20 | 2.89 | 8 |
| 2.386 | 16 | 2.734 | 8 |
| 2.245 | 6.5 | 2.547 | 80 |
| 1.985 | 19 | 2.349 | 2.2 |
| 1.868 | 2.8 | 2.213 | 2.2 |
| 1.824 | 2.8 | 2.122 | 15 |
| 1.56 | 1.8 | 1.961 | 20 |
| 1.531 | 7 | 1.638 | 16 |
| 1.504 | 2.2 | 1.49 | 16 |
| 1.393 | 5 | 1.339 | 6.5 |
| 1.329 | 0.9 | 1.30 | 5 |
| 1.29 | 2.2 | 1.246 | 5 |
| 1.237 | 2.2 | | |

4. SOME OBSERVATIONS AND CONCLUSIONS

At the outset it might be mentioned that in all the mica patterns, the background intensity is very large and variable from specimen to specimen. This makes the estimation of intensities unusually difficult. Examination of the various tables shows that there are significant variations in the occurrence of lines from specimen to specimen; and there are variations in spacings or intensities or both in all the species and even in different specimens of the same species. Our observations are in conformity with those made by Van Dijke Beatty and others. This,

however, is probably to be expected in view of the fact that the composition of mica is extremely variable both qualitatively and quantitatively.

However, if we confine ourselves to the three strong powder lines corresponding to the approximate spacings 4.33, 3.27, 2.59 Å and their relative intensities, there does seem to be some indication to distinguish between muscovite, phlogopite, biotite and mahadevite, muscovite having a relatively very intense 4.33 Å line. But a larger number of specimens of each species must be studied before we know the precise import of these relative intensities as the latter change from specimen to specimen.

This leads one to the conclusion that although X-ray powder technique is a powerful method in identification analysis, it proves to be a rather difficult one in the case of micas.

5. ACKNOWLEDGEMENTS

The authors in conclusion thank Professor R. S. Krishnan for suggesting the problem and for his kind interest throughout the work.

REFERENCES

1. Jackson, W. W. and West, J. "The Crystal Structure of Muscovite," *Zeit. Krist.*, 1931, **167**, 211.
2. Nagelschmidt, G. .. "Röntgenographische Untersuchungen an Tinon," *Ibid.*, 1934, **87**, 120.
3. ————— .. "X-ray investigation of clays, Part III," *Ibid.*, 1934, **97**, 514.
4. Maegdefrau and Hofmann "Glimmerartige Mineralien als Tonsubstanzen," *Ibid.*, 1937, **98**, 21.
5. Ramaseshan, S. .. "Mahadevite—A New Species of Mica," *Proc. Ind. Acad. Sci.*, 1945, **22 A**, 177.
6. Suzanne Van Dijke Beatty .. "X-ray spectrometer studies of mica powders," *Amer. Mineral.*, 1949, **34**, 74.