Studies of thermal resistance of heated flogopite

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Abstract. Research on thermal stability of mineralogical types of micas deals with materials with high thermal stability. At high temperatures, as a result of water release, mica loses its transparency, swells, stratifies and becomes mechanically unstable. Due to these changes, the properties of mica worsen. Phlogopites can vary in their quality. Therefore, when phlogopite is used in materials and devices operating at very high temperatures, it is necessary to use the type of mica whose thermal resistance meets the operating conditions.

1. Introduction

A characteristic of heated mica is swelling caused by water release. The process of swelling decreases the strength and worsens mica properties. Therefore, the temperature of swelling determines possible applications of mica and can indicate its thermal stability. Observations were made to study mica after heating. For these tests, mica samples of 10x25 mm in size and seven samples of phlogopite were heated to 1200 degrees. At the highest temperature, the sample was kept for 30 minutes. After cooling, the samples were compared with a standard (mica that has not been heated) by color, surface, transparency and shape [1]. P. Hidnert and J. Dixon [2] studied the effect of heat treatment of mica on its thickness, transparency and color. Five mica samples were made in the form of disks with a diameter of about 16 mm, an area of 2 cm² and a thickness of 0.09 to 1.5 mm. The samples were heated in an electric furnace up to 600, 700, 800, 900 and 1000 degrees. After cooling to room temperature, the thickness of mica was measured and its transparency and color were determined. The thickness of some samples, measured without pressure, was 8 to 20 times the initial thickness. Based on the results, it can be assumed that with an increase in the temperature from 600 to 1000 degrees, the shrinkage of expanded and cooled mica increases. Such an increase in shrinkage in phlogopites is more pronounced at 800-1000 degrees. In addition to changes in thickness, the transparency and color changed as well. The samples were classified into transparent, translucent and opaque. When the swelling level increased, mica became less transparent. The color of each sample, which had no metallic luster, was determined by the accepted color determination method. [3], [4]. Observations show that the swelling level manifests itself in different temperature ranges for different types of mica.

2. Materials and methods

Measurement of the thickness of heated mica determines the residual swelling, which gives only a rough idea of the swelling level at heating temperatures [5]. The swelling meter consists of a quartz tube into which a quartz baffle is fused. The tube is placed vertically in an electric oven with a thermocouple hole in a close contact with the quartz tube. The latter is not rigidly connected to the...
The mica sample is placed on a quartz tube baffle and covered with another smaller quartz tube. The rise of the quartz tube, caused by the swelling process, causes the lever to lift. Its end marks the degree of swelling. By creating a certain pressure by advancing the counterweight or applying a load to the lever, it is possible to determine the swelling drop curve during cooling, the residual swelling, and the force of mica when it swells. [6]

3. Research results and analysis

For different mineralogical types of mica, the changes caused by the process of heating is different. To determine the nature of changes, mica samples were heated at 500-1200 degrees. Samples of dark Slyudyanka phlogopite 15 and 18 were used. Each sample was a rectangular plate, 40x15 mm in size and 0.1 mm in thickness. It was placed in a porcelain boat in the electric tube furnace, where there was a junction near the sample platinum-platinum rhodium thermocouple. The temperature rise was 150 degrees per hour. The process of cooling began after holding each sample for 30 minutes at one of the above temperatures. One of the samples was not heated, but served as a reference. External changes in mica are presented in table 1.

| Heating temperature, degrees | Description of phlogopite samples  | Phlogopite deposit |
|-----------------------------|-----------------------------------|--------------------|
|                             | Sample 15                         | Sample 18          |
| 20                          | Partial transparency. Air inclusions in the form of foggy spots. Dark color | Partial transparency. Air inclusions in the form of foggy spots. Dark color | Slyudyanka deposit |
| 500                         | A slight increase in the number of hazy spots | Weak swelling in reflected light. Silver gray color. Transparency in small areas | Slyudyanka deposit |
| 550                         | A slight increase in hazy spots | Weak swelling in reflected light. Silver gray color. Transparency in small areas | Slyudyanka deposit |
| 600                         | A small increase in hazy spots | Weak swelling in reflected light. Silver gray color. Transparency in small areas | Slyudyanka deposit |
| 650                         | Small air inclusions. Reduced transparency in small areas | Increased swelling. | Slyudyanka deposit |
| 700                         | Noticeable air inclusions. A slight decrease in transparency | Swelling is more pronounced. The surface is golden in reflected light | Slyudyanka deposit |
| Temperature | Swelling Description | Color and Transparency Changes |
|-------------|----------------------|-------------------------------|
| 750         | Barely noticeable    | Slyudyanka deposit            |
| 800         | Slightly swollen     | Slyudyanka deposit            |
| 850-900     | Slightly swollen     | Slyudyanka deposit            |
| 950         | Fragility, Golden color | Slyudyanka deposit          |
| 1000        | A decrease in carcass strength | Slyudyanka deposit |
| 1100        | A greater decrease in transparency and an increase in fragility | Slyudyanka deposit |
| 1200        | Full opacity. Light brown color. Strong fragility. | Slyudyanka deposit |

4. Discussion

The study can characterize the thermal stability of mica samples and the nature of residual external changes, while changes occurring in mica are unclear. In sample 15, significant external changes were observed after calcination at 950-1000 degrees. [7] Samples of phlogopite 18 turned out to be strongly swollen and lost transparency after heating up to 500 degrees. By thermal stability, this mica is inferior to phlogopite 15. The phlogopite mica samples were examined after heating. The samples 10x25 mm in size were heated up to 600 degrees and then up to 1200. At the highest temperature, the sample was held for 30 minutes. The time for raising the temperature up to 600 degrees was 35 minutes, and up to 1200 degrees - 1.5 hours. After cooling, the samples were compared with the reference one by color, surface, transparency, and shape. Mica heated to 600 degrees did not change. Mica heated to the higher temperature underwent significant external changes. With an increase in temperature up to 1000 degrees, the shrinkage of expanded and cooled mica increases to varying degrees for different types of mica. This increase is pronounced within 800-1000 degrees [8], [9]

5. Conclusion

The accuracy of readings at a small thickness of samples decreases due to smaller deflections of the instrument lever. For more accurate results, it is necessary to use samples with a greater total thickness. However, in order to facilitate the preparation of samples, it is possible to reduce their thickness to 2 mm, at which similar results can be obtained. [10] Tests of mica pillars composed of plates of a different thickness, but with the same total thickness of 2 mm showed that the swelling level decreases with an increasing plate thickness. When testing, it is advisable to use thinner sheets with a thickness of 0.025 mm, usually obtained in the production of chipped mica [11]. The studies carried out at different temperature rise rates have shown that a change in speed within 5 to 25 degrees
per minute does not have a large effect on the change in the swelling values [12]. The measurement of phlogopite sample swelling showed that the swelling intensity at 850 degrees was 180-190% in case of heating at 5 and 25 degrees per minute. Residual swelling was equal to 10%. For all subsequent tests, samples of 20x20 mm in size were used. They were made of 0.025 mm mica sheets with a total thickness of 2 mm. [13] They were heated at a temperature which was 5–100 degrees higher than the temperature of maximum swelling [14].

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