A METHOD OF PRODUCTION SYNTHETIC MICA CRYSTAL IN USING SEED CRYSTALS

NOBUTOSHI DAIMON, YOJI ITO and MINORU HIRAO

Department of Applied Chemistry

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Many research works on the production of synthetic mica crystals have been reported until recently.[1] In all these works batches of fluor-mica composition were melted either by external heating or by passing of electric current directly through the melt. When the melt was cooled down, it crystallized into aggregates of interlocked crystals orientated in a random way. Mica crystals must be separated from the aggregates by crushing or by hand-splitting. Most of mica crystals thus obtained were mostly small fragments and only a small portion of crystals were large enough for commertial uses.

Such factors as an inadequate control of the cooling rate, changes of the temperature gradient in the furnace and the decomposition of the melt were the causes of the formation of large number of small crystals. Even a superheating of the melt caused a remarkable supercooling and it resulted in the formation of large number of nuclei, once the crystallization started.[2] The melt containing many nuclei solidified into an aggregate of small crystals of random orientation. As a measure to prevent the spontaneous formation of many crystal nuclei, the use of seed crystals has been tried in our laboratory. The Kyropoulos technique using seed crystals assembled in parallel with each other was tested, but the result was not satisfactory to grow larger crystals, because of the decomposition of the melt during the drawing up of the seed crystals.[1i] Tsujimura and Noda tried to grow a seed crystal in the melt, but they failed because of technical difficulties.[1i] However the Stockbarger technique was proved to be excellent for the growth of "mica booklet", when seed crystals were originally placed in a closed crucible.

In this paper the technique and the results of the experiments are briefly given.

The vertical section of the furnace assemblage is shown in Fig. 1. A platinum crucible of rectangular form was placed in a Schamotte sagger (4), the lower part of which was made a column. The Schamotte column was mounted on a metal tube (3). The metal tube was attached to a float (2) which was placed in a water tank (1) and was vertically movable with the change of water level. The water was flown out through a syphone (6). The descending rate of the crucible was regulated by the rate of flowing out of water through a capillary (7) attached at the outer end of the syphone. The crucible (8) having a sectional area of 1 cm by 1 cm and a depth of 5 cm was made of a platinum foil of 0.03 mm in thickness. Sheet crystals (9) of 1 cm in width of synthetic fluor-phlogopite which should become the seed crystals were placed on the bottom of the crucible, their cleavage planes being parallel to a side wall of the crucible, as shown in Fig. 2. As the raw material, powdered synthetic fluor-phlogopite crystals, a mixture of chemicals

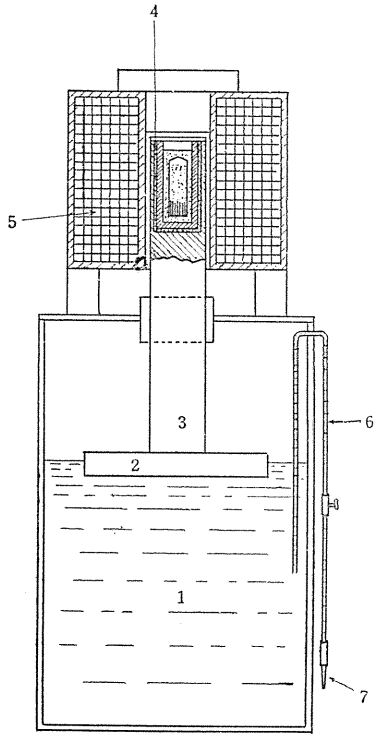

FIG. 1. The vertical section of the appratus.

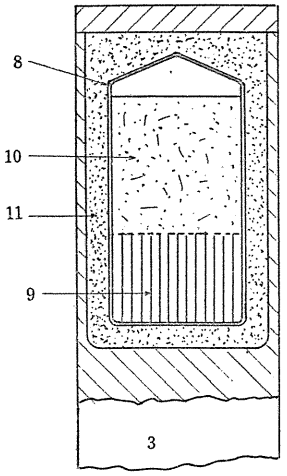

FIG. 2. The vertical section of the melting crucible.

or glass whose composition corresponded to that of fluor-phlogopite or molten blocks of fluor-phlogopite crystals were packed on the top of the upper end of the seed crystal layer. After the raw material (10) was charged, the upper end of the platinum crucible was sealed up and it was placed in the sagger. The space between the crucible and the wall of the sagger was filled with fused alumina powder (11). The crucible assemblage was inserted in a cylindrical electric furnace (5) to a position which was ajustable by the level of water in the tank. The part of the furnace which was at a level 5 mm above the upper edge of the seed crystal layer was heated up to 1,400° C in order to melt the raw material and the upper part of the seed crystal layer. This procedure needed a temperature gradient of 30-60° C/cm around the border of the seed crystal layer and the raw material. After the crucible was maintained at that position for about 30 minutes, the water in the tank was flown out through the syphone in order to descent the crucible through the furnace at the rate of 0.4-0.7 mm/hr which corresponded to the cooling rate of 1-4° C/hr. After the crucible was descended a distance of about 40 mm, the furnace was cooled down in order to take out the crucible from the furnace. The temperature gradient across the crucible at the beginning of the descendance is shown in Fig. 3. As the crucible was moved downward and the melt passed the melting temperature level in the furnace, then the unmelted portion of the seed crystals began to grow without any supercooling, thus preventing the spon-

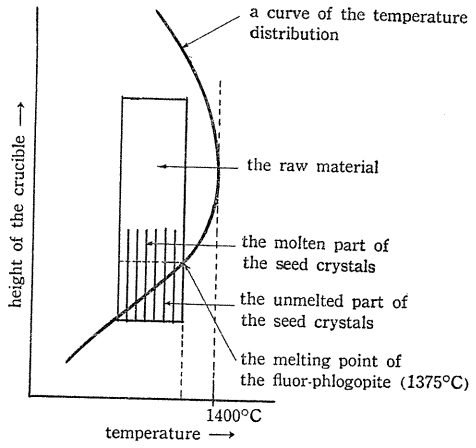

FIG. 3

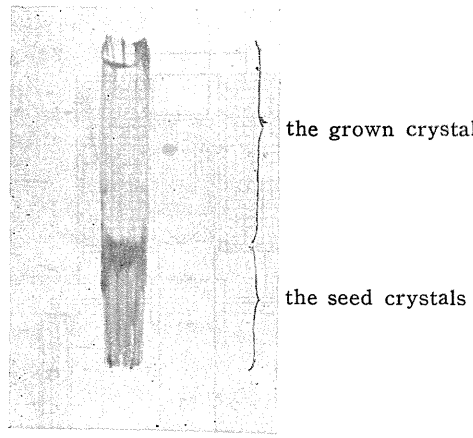

FIG. 4

FIG. 3. The temperature distribution in the crucible.

FIG. 4. A photograph of a grown crystal-booklet, shown in the direction parallel to the cleavage plane.

taneous formation of any superfluous nuclei and keeping the original orientation of the seed crystals. The seed crystals grew up to the top of the charge, and the resulted crystallized mass was a so-called "mica bookled" having cleavage planes parallel to each other. Thus the technique of production of "mica-booklet" has been established using seed crystals which were originally placed in a closed crucible.

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