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Terrestrial development of the experiments on the fullerite C_{60} crystal growth in microgravity

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Microgravity creates favorable conditions to reduce dislocations and grain boundaries density in growing crystals due to absence of close contact with the ampoule walls and absence of plastic deformation of the crystal under its own weight. For improvement of the fullerite C_{60} crystal growth technology before the scheduled space experiments on the ISS the growing of the high purity grade fullerite C_{60} crystals with the sufficiently high structural perfection were carried out on the Earth from the C_{60} vapor in sealed quartz ampoules (pre-evacuated to the pressure of 10^{-3} Pa) at temperatures in the evaporation zone ranging from 560 – 610 °C with a temperature gradient between the evaporation and deposition zones of 3 – 10 K/cm within 72 h. The grown single crystals had a size of $\sim 5 \times 5 \times 5$ mm and habitus corresponding to the fcc lattice. IR spectroscopy shows the high purity fullerite C_{60}.

Keywords: fullerite, C_{60}, crystal growth, sublimation, microgravity, IR spectroscopy.

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1. Introduction

To investigate the electron–electron correlation and polaron effects caused by the small conduction band (width of 0.05 eV) the perfect fullerite C_{60} crystals with sufficiently large overall dimensions (about several millimeters) are required. The continuous growth of fullerite C_{60} crystals by physical vapor transport has been described by the various authors [1–3]. The fullerite C_{60} crystals grown in the first experiments in microgravity aboard the FOTON-M3 spacecraft [4] had more perfect crystal structure in comparison with the crystals grown on the Earth: microgravity creates favorable conditions to reduce the dislocations density and grain boundaries in the growing crystals due to absence of close contact with the ampoule walls and absence of plastic deformation of the crystal under its own weight.

2. Experimental

For further improvement of the technology before the scheduled space experiments on the ISS, the growing of the high purity grade fullerite C_{60} crystals with the sufficiently high structural perfection were performed on the Earth from the C_{60} vapor in sealed quartz ampoules (pre-evacuated to the pressure of 10-3 Pa) at temperatures in the evaporation zone ranging from 560 – 610 °C with the temperature gradient between the evaporation and deposition zones of 3 – 10 K/cm within 72 h. The preparation of initial samples for analogous growth experiments was studied in detail in the previous work [5] and in our case, includes a low-temperature treatment of the fullerene C_{60} powder under dynamic vacuum of 10^{-3} Pa at the temperature of 350 – 400 °C within 3 h, then this powder was sublimated under dynamic vacuum of 10^{-3} Pa at 600 – 650 °C within 8 h. From this sublimated powder the primary fullerite C_{60} crystals were grown under a static vacuum of 10^{-3} Pa in sealed quartz ampoules at the temperature 610 °C with the temperature gradient between the evaporation and deposition zones 5 K/cm within 72 h. Then the regrowth of the primary deposit was run at the same conditions, yielding the high purity material. After these purification procedures, the resulting material was charged into quartz ampoules evacuated to a pressure of 10^{-3} Pa, hermetically sealed and placed into the multi-zone growth furnace for crystal growth. Three types of the gradients used in the experiments are shown in Fig. 1. The polycrystalline deposits of C_{60} were obtained in the runs #1 and #2. The fullerite C_{60} single crystal was grown in the run #3.

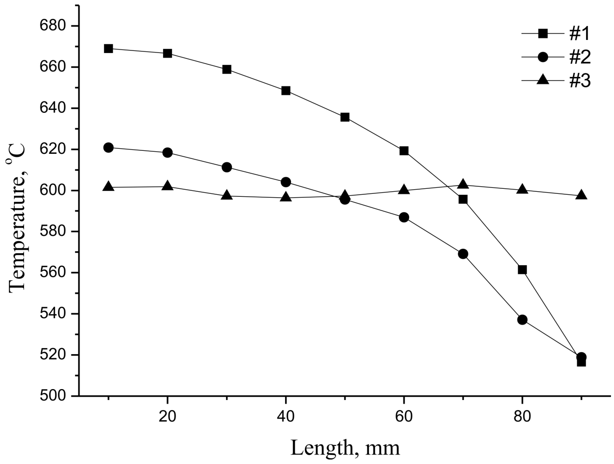

FIG. 1. Three types of the gradients of the multi-zone growth furnace (runs #1, #2, #3)

3. Results and discussion

The single crystals grown in the run #3 have the size of about $5 \times 5 \times 5$ mm and habitus corresponding to the fcc phase of C_{60} (see Fig. 2). The presence of twins was revealed using the optical microscopy and the single-crystal diffractometer Oxford Diffraction Gemini-R. The twins interface corresponds to the $\{111\}$ fcc conjunction plane.

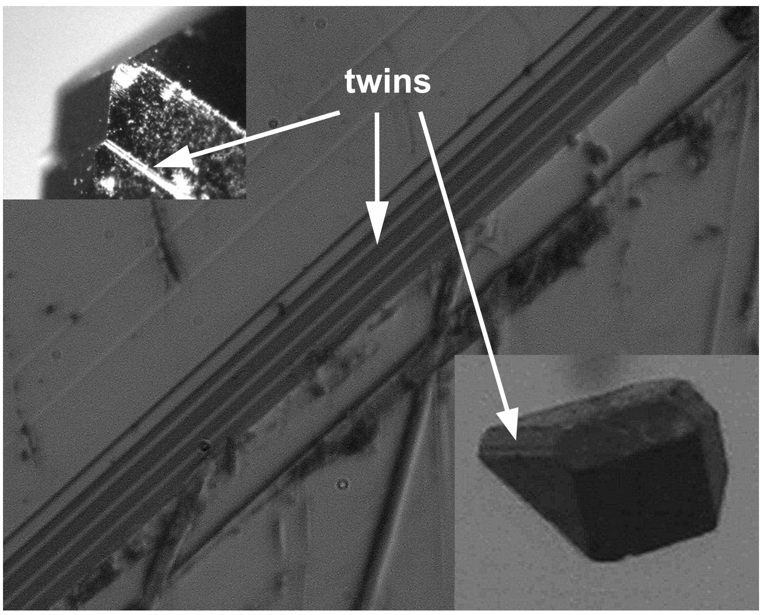

FIG. 2. Optical image of the twins (arrows point “rungs” – the twins interface)

IR transmission spectrum of the C_{60} powder produced from the fullerite of run #3 is represented in Fig. 3. Strong scattering on shorter waves is explained by the KBr surface roughness. These data show the high purity of fullerite C_{60} after the overall technology chain.

4. Conclusions

The heating rate and the temperature distribution in the furnace must be carefully controlled because they have strong influence on the nucleation and growth rate and hence on the perfection of the grown crystals. Optimizing these parameters, the large C_{60} single crystals of good quality with size more than 5 mm can be grown reproducibly.

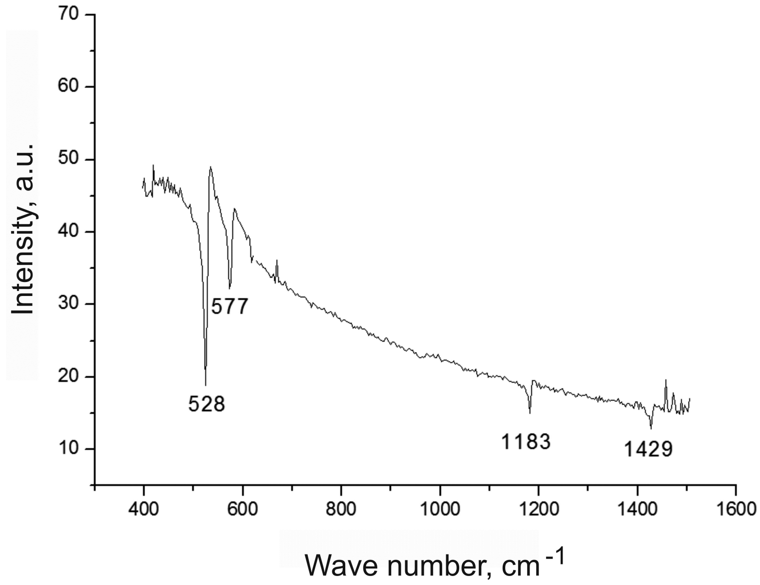

FIG. 3. IR spectrum measured for fullerite C_{60} single crystal from the run #3

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