

Semi-Hollow Silver Halide Crystals Containing High-Iodide Content

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Introduction

According to R.L Daubendiek(1), if special core/ shell precipitation is carried out at 35°C, Silver Iodide crystals formed at pAg 6.5 will be consisted of four hexagonal bipyramid, the four bases of which join to form a common tetrahedron with truncated corners. Each end face of the four truncated hexagonal bipyramids is a (0001) crystal face of β -silver iodide. The crystal lattice of (0001) crystal face of β -silver iodide is similar to that of (111) crystal face of silver bromide.

On each end face of the four truncated bipyramids occurs the nucleation of plate silver iodobromide crystal and succeeding crystal growth, as Ag^+ , Br^- and solvent of silver Iodide is added to the vessel containing dispersing medium, distilled water and above-mentioned silver Iodide seeds.

If epitaxial $\text{Ag}(\text{I},\text{Br})$ crystals are tabular, hollow silver halide grains will form into being(2). In this manner, we are trying to wrap up the residues of silver iodide seeds in the semi-hollow silver iodobromide crystals, before these seeds are completely dissolved.

The structure and composition of these crystals are determined by electronmicroscopy, energy-dispersive analysis and ultramicrotomy.

Experimental

Several kinds of silver iodide seeds were prepared by the Double jet method. These seeds have above-mentioned morphological configuration but different length of truncated hexagonal bipyramids.

Controlling PAg Value, temperature and quantity of solvent of silver Iodide, tabular silver iodobromide crystals containing high Iodide epitaxially occurred on (0001) crystal faces of silver Iodide seeds and finally formed the crystals containing more silver iodide. The microanalytical work was carried out using a JEM 2000FX electron microscope in conjunction with STEM unit and EDS(WOYAGE-3150, energy dispersive X-ray spectrometer).

The ultrathin sections of final crystals were coated by carbon and platinum (20A), observed and analyzed by TEM and EDX.

Result and Discussion

(1) The dynamical process of the formation of silver iodide seeds.

In several U.S. patents authors describe the preparation of above-mentioned silver iodide seed(3)(4). In our process of precipitation some conditions are changed for the purpose of the correction of silver iodide seed. In the process of precipitation the samples of silver iodide seeds were taken from vessel every other five minutes. These samples were treated with trypsin solution and water repeatedly. After the treatment of samples specimens for TEM observation were prepared.

The growth process of silver iodide seeds are shown in Fig.1. In this process, PAg value and temperature are important conditions. In certain steps of precipitation the increase of temperature can promote the formation of truncated hexagonal bipyramid. The soluble power is not the only important factor although the rise of temperature causes the increase of the solubility of silver iodide. We are trying to use the solvent of silver iodide instead of the rise of temperature in the process of precipitation, but we could not get the above-mentioned morphological configuration.

As shown in Fig.1, this kind of silver iodide seed has above-mentioned morphological configuration but the common tetrahedron from four base of four hexagonal bipyramids have original form no more and another four truncated ends are transformed into short and thick ends.

(2) Semi-hollow silver halide crystal containing high iodide content

As mentioned in the introduction, if the conditions of the epitaxial growth is appropriate, the nucleation of plate silver iodobromide crystal and following growth will occur on the end face of each of the four truncated bipyramids of silver iodide seed. In the previous work(2), four truncated hexagonal bipyramids of this silver iodide seed are longer and thinner. While the edges of the adjacent epitaxial crystal meet to form new silver halide grains, the formed grains will be big and hollow.

We changed the conditions of precipitation and get desirable silver iodide seeds. The four ends of these seeds join to form a common body which was not in the original

form and the other four ends are truncated nearly in the middle of bipyramid(Fig.2).

In this case the edges of the four growing plate silver iodobromide crystals on four end faces of each silver iodide seed meet more easily and close the crevice between the plate crystals more earlier (Fig.3)(Fig.4)

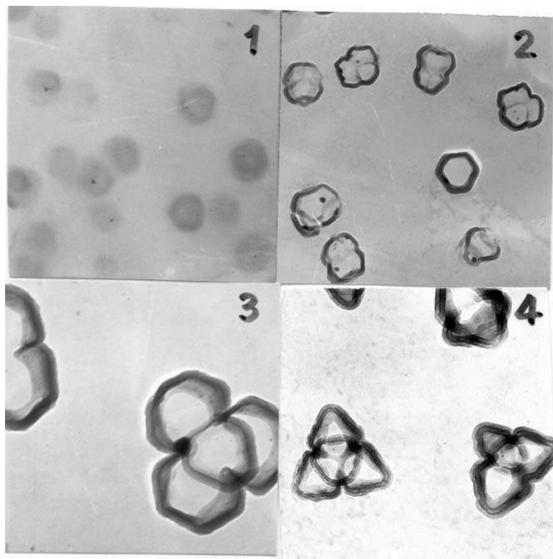


Fig.1 the growth process of silver iodide seed



Fig.3 epitaxial plate crystals

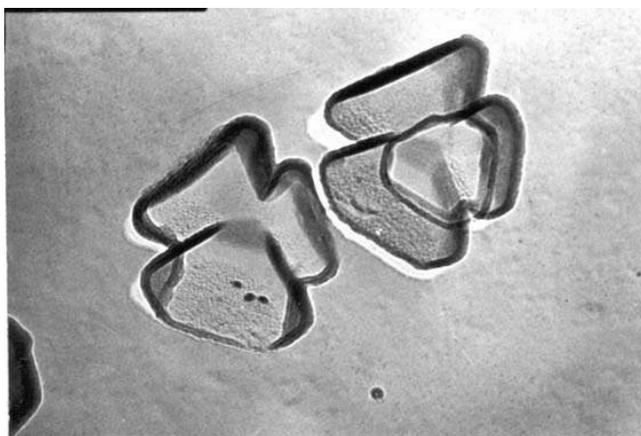


Fig.2 short truncated bipyramid

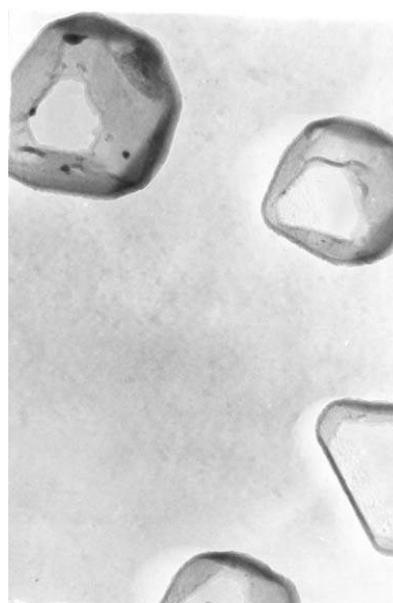


Fig.4 semi-hollow grains

In this manner we are trying to wrap up the residues of silver iodide seeds in semi-hollow silver iodobromide crystals before these seeds are completely dissolved. Ultimately silver bromide is precipitated on the surface of semi-hollow iodobromide crystal.

Fig.5 shows the result of micro-area analysis by STEM/EDX. In the center of semi-hollow iodobromide crystal the content of iodide is 22.4 mole%, the content of iodide in the area between the center and the edge of the same grain is 16.4mole%, that near the edge is 4.2mole%.

The ultrathin sections of the coating of such emulsion were coated by carbon and platinum. After selective shorter solution for silver halide, ultrathin sections were observed by TEM.

Fig. 6 shows the result of ultrathin section. The black area of the grain in Fig.6 was considered the residue of silver halide seeds. The result of component analysis of the black area is uncertain because the ultrathin section is too thin for determination accurately.

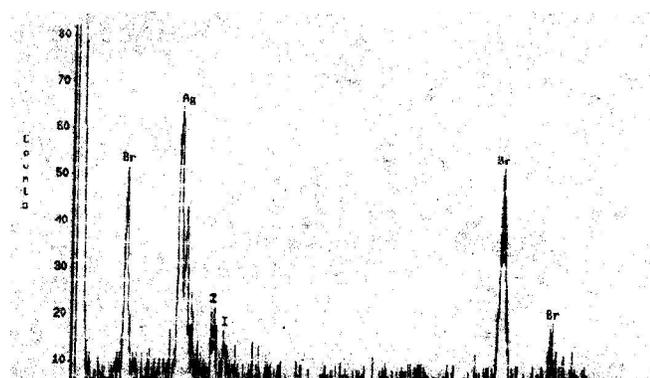


Fig. 5 the result of micro-area analysis

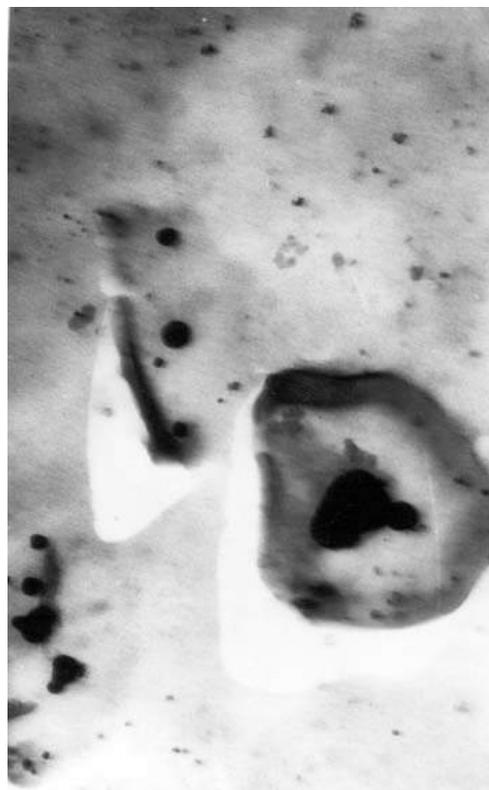


Fig. 6 the electronmicroscopic photograph of ultrathin section

-This work was supported by the National Natural Science Foundation of China

Reference

- 1.Daubendiek R.L.,
Paper from the International Congress of Photographic
Science, 140-143(1978)
- 2.Irving M.E.ect.
U.S. Patent 4,916,052(1990)
- 3.Irving M.E.
U.S.Patent 4,927,745(1990)
- 4.Jones C.G.etc.
U.S.Patent 5,240,825(1993)qasNM