Anisotropic Growth and Screw Dislocation of {100} Tabular Grains

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Abstract

The dislocation structure of silver chloride tabular grains with {100} faces on the surface (hereinafter referred as "{100} tabular grain ") was observed by low-temperature transmission electron microscopy (LTEM) to study the mechanism of the anisotropic growth of the grains.

The results of the observation were as follows.

1. A nucleus trace was observed at one of the corners of almost every {100} tabular grain.
2. Two dislocations extended from a nucleus trace to the <31n> directions.
3. {100} tabular grains grew anisotropically from the nucleus only in two directions, to which the above-stated two dislocations extended.
4. The dislocations in {100} tabular grains were identified as "screw dislocation".

In this work, it was evidenced that the anisotropic growth of {100} tabular grains were caused by screw dislocations.

Introduction

In 1974, Mignot described {100} tabular AgBr grains and proposed a mechanism for their anisotropic growth[1], according to which screw dislocations would play an important role. However, he could show a little experimental data to prove his proposal. Brust and House described {100} tabular AgCl grains, although they did not discuss the mechanism for their anisotropic growth[2]. Therefore it is very important to clarify the growth mechanism of {100} tabular grains for further understanding of these grains.

Experimental

1. Precipitation of {100} tabular grains

Silver chloride nuclei were at first formed in an aqueous gelatin solution. Subsequently, a KBr solution was added to the solution to generate dislocations on the nuclei for their anisotropic growth. After ripening, double-jet addition of $AgNO_3$ and NaCl solutions were carried out to grow tabular grains.

The specimen for TEM observation was sampled at various stages during the precipitation of tabular grains, and were observed by TEM after treated by the procedure described in 3-1).

2. Incorporation of I^- in {100} tabular grains as traces of their growth

An aqueous solution of KI (0.006 mole/mole adding AgX) was added to a reaction vessel to incorporate iodide ions in the {100} tabular grains as traces for their growth without any detectable change in their anisotropic growth.

The sample were treated according to the procedure described in 3-1) and were observed by TEM.

3. LTEM

1). Precipitation of samples

The emulsions sampled at various stages during the precipitation of AgX grains were added to a methanolic solution of phenylmercaptotetrazole(0.001 to 0.01mole /mole AgX) to retard further growth of the grains. Each emulsion was centrifuged in order to separate the AgX grains from its gelatin phase. After the decantation of the solution, pure water was added anew to the grains to from their suspension. A drop of the suspension was put on a thin carbon film on a copper grids for TEM observation.

2). Observation of AgX grain

The specimen thus prepared was observed by an electron microscope JEX-2000 FKII manufactured by JEOL LTD., at an acceleration voltage of 200kV and at magnifications from 5,000 to 50,000 by use of a cooling holder, 626-0300 Cryostation manufactured by Gatan Co., to keep samples at -120°C.

4. Determination of Burgers vector by using a TEM

The specimen was prepared by the procedure described in 3-1). An electron microscope (JEOL, JEM-4000EX) was used, and operated at accelerating voltage of 400kV with a cooling holder, to keep the specimen at -120°C. The Burgers vector represents the displacement vector in dislocation.

Dislocations can be observed by TEM, since they introduce crystal distortion along them. However, dislocations are not observed in a TEM image under the condition that the image is composed of only the reflected beams from the planes that are free from the distortion .

For example, an edge dislocation-E or a screw dislocations-S in Fig.1 introduces distortion in the crystal, thus making it observable in a TEM image at almost all the direction. However , the Face-A or Face-B is not distorted by an edge dislocation-E or a screw dislocation-S respectively, and these dislocations are not therefore observable in a TEM image, which is composed of the reflected beams from these faces.

The Burgers vector was determined by using this feature, according to which the image of dislocations disappears depending on the reflection condition. Namely, we titled the specimen to the angle at which the image of dislocations was expected to disappear and looked at the change in the image.

5. Relationship between Burgers vector and direction of dislocations.

-Identification of a kind of dislocations-

Fig.1 shows a scheme of dislocation. In general, the relationship between Burgers vector and the direction of a dislocation is parallel with each other in the case of a screw dislocations, and is perpendicular to each other in the case of an edge dislocations.

On the basis of the above-stated knowledge , the identification of a kind of dislocation was made by examining the geometric relationship between Burgers vector and the direction of dislocation.

Results and Discussions

1. Observation of dislocations

Photo.1 shows a TEM image of AgCl {100} tabular grains sampled during their precipitation process. Many {100} tabular grains contained two dislocations.

The high magnification TEM images of grains having two dislocations are shown in Photo.2. The nucleus which formed at the nucleation stage of the grain was recognized at the crossing point of the two dislocations in each grain.

The microanalysis by means of TEM EDAX revealed the existence of bromide at the nucleus. Since bromide ions were added just after the formation of the nuclei, the place at which bromide was detected in a grain indicated the nucleus, from which the grain grew.

2. Relationship between directions of dislocation and of anisotropic growth of a grain

TEM image of AgCl tabular grains in which the growth traces were introduced are shown in Photo.3. Almost all the {100} tabular grains grew only in two directions, which agree with the directions of two dislocations. The morphology of the grains to which KI solutions were added for the incorporation of the growth traces was almost the same as that of the grain without KI, as judged from their thickness and equivalent circular diameter. It is therefore considered that the anisotropy was unchanged with the addition of KI solutions.

3. Identification of a kind of dislocations in {100} tabular grains

TEM images of {100} tabular grains taken under various reflected conditions are shown in Photo.4. The image of dislocations was still observable in the TEM image of the grains under the condition where the edge dislocations should be invisible, whereas it was not observable in the image under that condition where the screw dislocations should be invisible. When the grains were tilted to the angle at which the Burgers vector was parallel with the direction of dislocations, the image of the dislocations was almost disappeared. It was therefore concluded that the dislocations in {100} tabular grains were "screw dislocations".

In this work, it was proposed from the above-stated observations that the anisotropic growth of {100} tabular grains was caused by screw dislocations.

References

1.A.Mignot *J.Crystl.Growth* **2 3** :207 (1974)
2.T.B.Brust and G.L.House IS&T 49th Annual Conference 32 (1996)

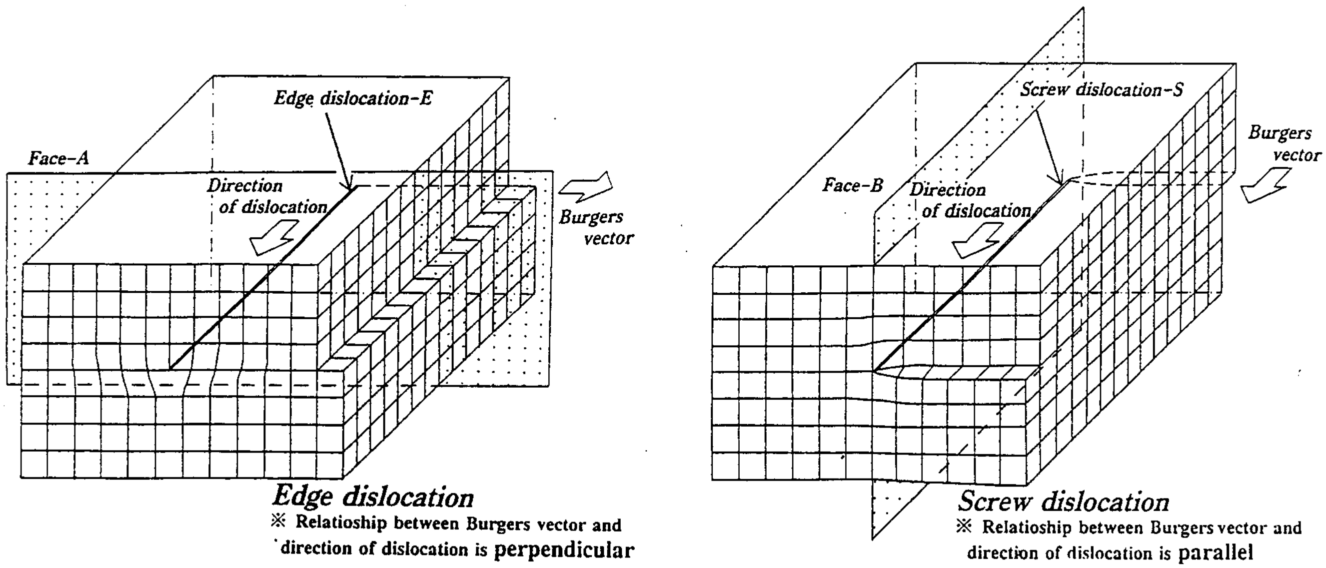


Fig.1 Scheme of dislocations.

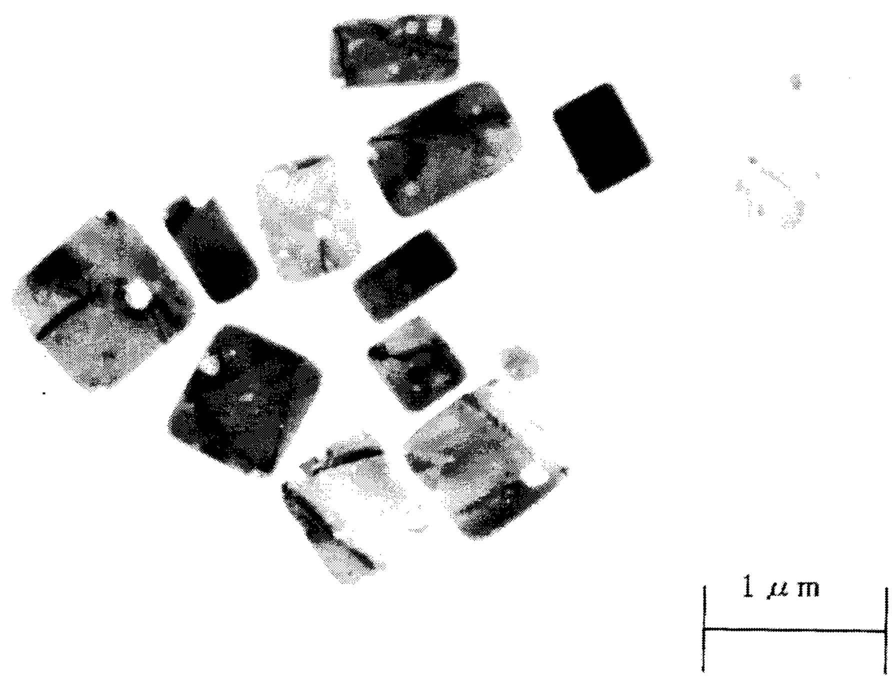


Photo.1 A TEM image of AgCl {100} tabular grains. An electron microscope (JEX–2000 FKII) with a cooling holder to keep the specimen at –120 ℃ was operated at accelerating voltage of 200kV.

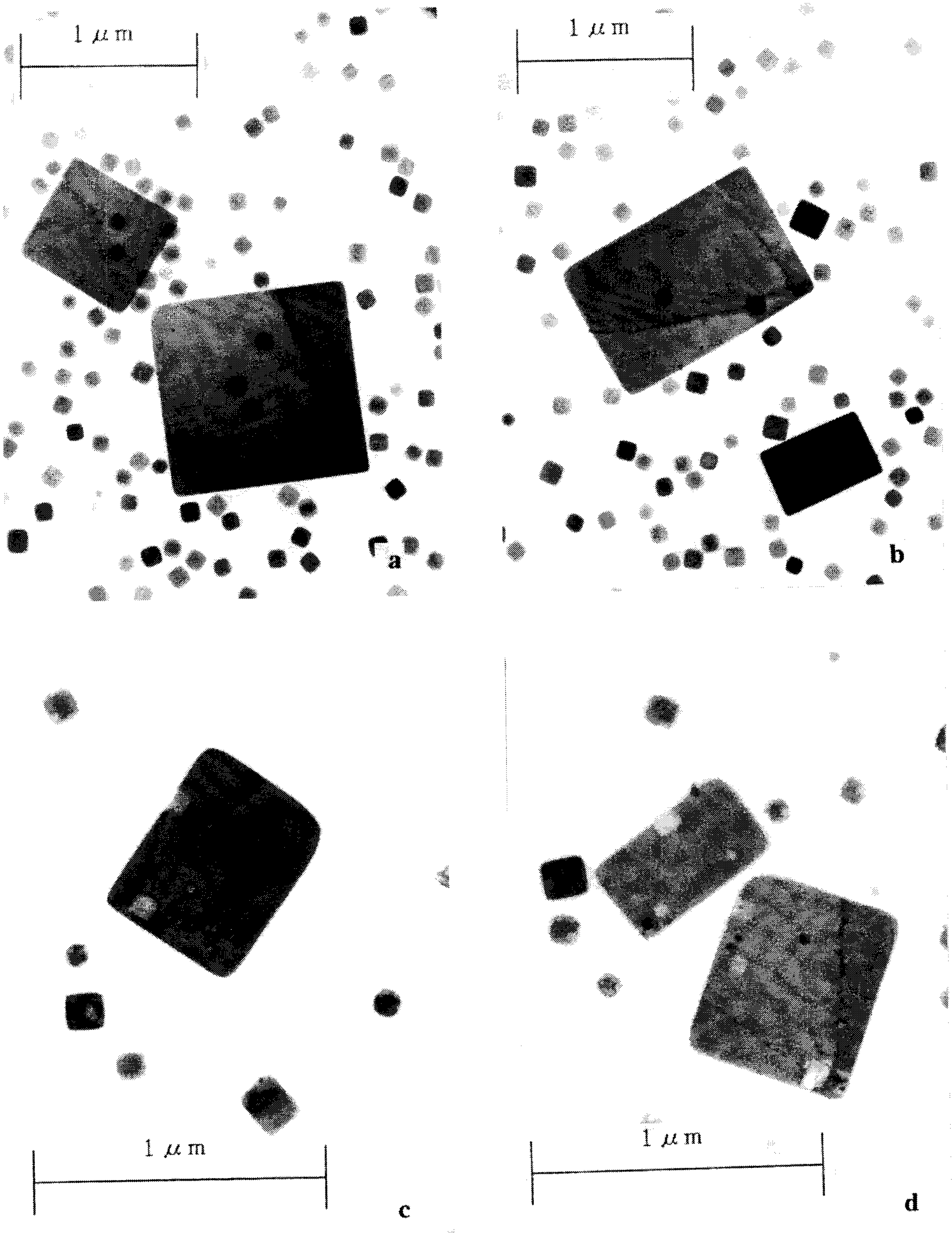


Photo.2 **High magnification TEM images of grains having two dislocations and nucleus which formed at the nucleation stage. An electron microscope (JEX-2000FKII) with cooling holder to keep the specimen at -120 ℃ was operated at accelerating voltage of 200kV.**

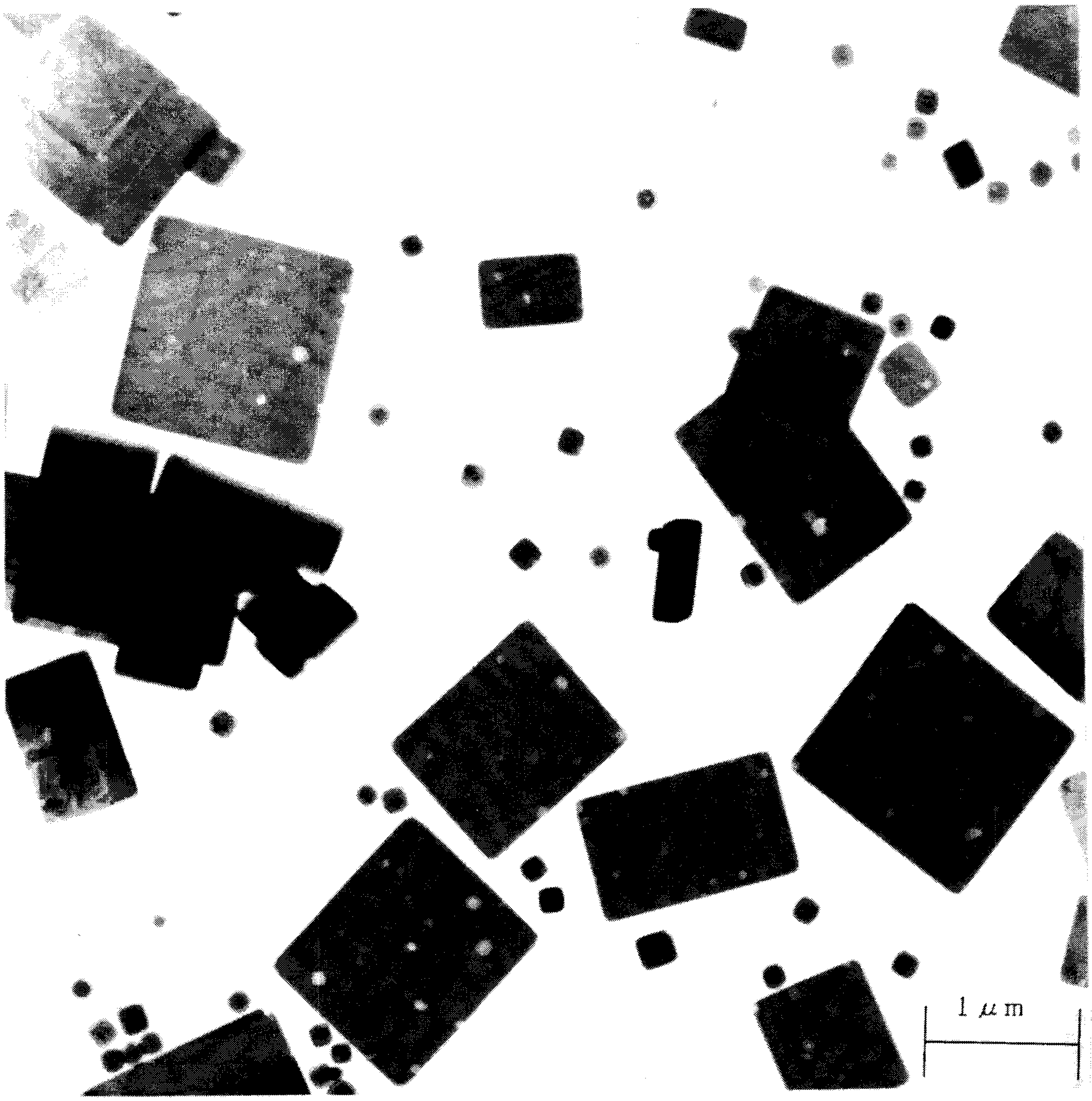
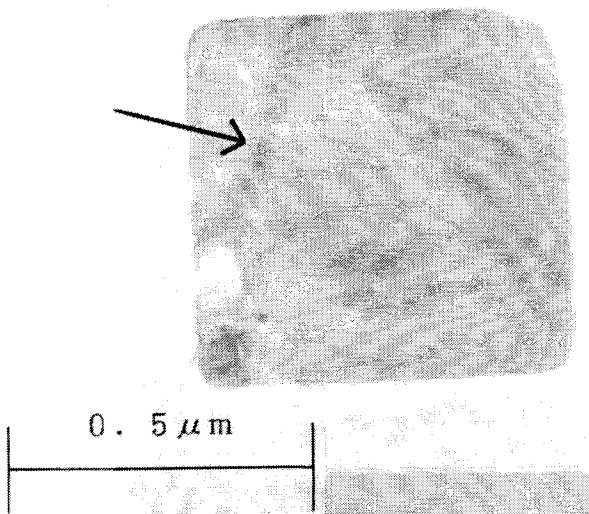
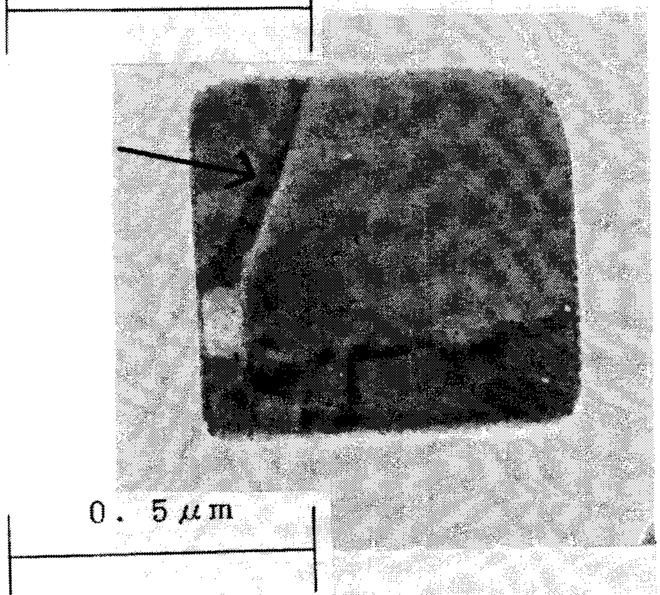


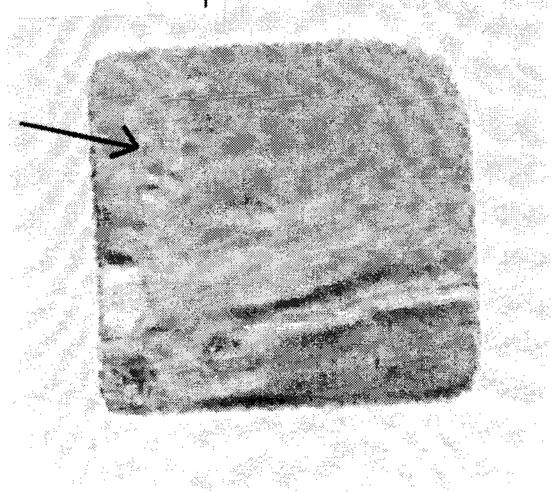
Photo.3 **A TEM image of AgCl tabular grains in which the growth traces were introduced. An electron microscope (JEX-2000 FKII) with a cooling holder to keep the specimen at −120 ℃ was operated at accelerating voltage of 200kV.**



(Photo.4–1)



(Photo.4–2)



(Photo.4–3)

Photo.4 **TEM images of {100} tabular grains taken under reflected conditions where the edge and screw dislocaions should be visible (Photo.4–1), the edge dislocations should be invisible (Photo.4–2), and the screw dislocations should be invisible (Photo.4–3). An electron microscope (JEM–4000 EX) with cooling holder to keep the specimen at –120 ℃ was operated at accelerating voltage of 400kV.**