Short Note

Clarification of the Paper "An Enhanced Understanding of Silver Halide Tabular-Grain Growth"

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Introduction

A paper describing a study that used two independent techniques to obtain information on the growth of high-aspect-ratio Ag(I)Br tabular grains was published by Maskasky in 1987 (referred to here as JEM).[1] This paper has been misinterpreted in discussions of a new tabular-grain growth model that involves the presence of {100} edge faces.[2,3] I regret any lack of clarity in the conclusions of JEM. In JEM, abrupt shape changes were observed in ~10% of the final tabular-grain population of emulsions made using oxidized gelatin,[4] high bromide ion concentration (0.072 M in KBr), and high temperature (80°C). These abrupt shape changes usually resulted in triangular grains. The percentage of grains found to have undergone an abrupt shape change showed a good correlation with the relative number of grains found to contain an odd number (probably 3) of parallel twin planes. The study concluded that the abrupt shape changes were caused by additional parallel twinning. The study did not conclude that most triangularly shaped tabular grains in all emulsions contain an odd number of parallel twin planes. (In this aspect, JEM is in agreement with Ref. 3.) As pointed out in JEM, the smaller triangularly shaped grains (~5 μm) that appeared as cores in many of the larger grains (~15 μm) of Emulsion F seem to contain two parallel twins. The relative population of doubly and triply twinned triangularly shaped tabular grains would assuredly depend on the emulsion growth conditions. This study did conclude that under conditions favoring good lateral growth, most triply parallel twinned tabular grains grow rapidly into triangles, but not all triangles need be triply twinned. (Note that very few triply twinned hexagons were found.)

The suggestion was made that during banding, iodide poisoning of the alternate "six" edges of a triangular or hexagonal tabular grain, not an additional twin, caused the abrupt shape changes found in JEM.[2] Such alternate edge selectivity seems rather improbable. This poisoning suggestion is also inconsistent with much of the data presented in JEM. It is inconsistent with the observation that the orientation of triangular surface deposits showed that of the two different alternate edge types of a triply parallel twinned grain, only one type grows rapidly.

In JEM, the iodide was added during four short intervals of ~1 min, to the emulsion at the stirrer head as a 0.001 M KI solution concurrent with the ~1 M $AgNO_3$ and KBr solutions. The iodide content of the band itself could be composed of no more than 0.1% iodide, and the total iodide content of the emulsion was only 33 ppm. Good agreement was found in the percentage of triangular grains having an odd number of parallel twins as measured by the pyramid growth technique (84.4%) and as predicted based on the luminescent banding patterns (82.4%). The good agreement between these two independent techniques, performed on the same emulsion, strongly suggests that the abrupt shape change was caused by an additional parallel twin. Note that relaxing the definition of a triangle more toward a hexagonal shape, as was suggested,[2] would merely equally increase the relative number of doubly twinned grains counted for both techniques because most hexagons were doubly twinned. This definition change would not alter the conclusion.

I have recently repeated the preparations of Emulsion C (AgBr tabular-grain control emulsion) and Emulsion F (iodide-banded AgBr tabular-grain emulsion) of JEM. These two polydisperse emulsions were sized using automated techniques that measure more than a thousand grains. (Both the average size and average thickness are area weighted.) Emulsion C Repeat had an average size of 10.2 × 0.12 μm, an aspect ratio of 85, and a decade ratio of 3.5. Emulsion F Repeat had an average size of 10.4 × 0.12 μm, an aspect ratio of 87, and a decade ratio of 3.4.* These two emulsions were further compared for differences in the populations of variously shaped triangular and hexagonal tabular grains. Approximately 500 tabular grains from each emulsion having threefold (or pseudo sixfold) symmetry and greater than 1.0 μm in diameter were measured, using automated equipment. Low magnification electron micrograph images of the emulsion were computer analyzed and classified into 10 shape classes having a long-edge-to-short-edge ratio of 10:X, where X is $> 0 \le 1$, $> 1 \le 2$, ... , $> 9 \le 10$. The data are shown in Fig. 1. The images were then manually examined and the numbers corrected for some obvious computer counting mistakes, such as counting trapezoids, counting severely ripened spherical grains as hexagons, and not counting grains that were touching another grain but were otherwise easily classified. This data correction resulted in a further improvement in the already good match between these two emulsions. For example, the triangular grain class having $X \le 1$, was 20.5% of the total sample population for Emulsion C Repeat and 20.5% for Emulsion F Repeat. Within experimental error, these two emulsions have the same average size, polydispersity, and grain-shape distribution. The similarity of these two emulsions is inconsistent with the suggestion of iodide poisoning of the edge structure of the iodide-banded emulsion.[2]

Further research is needed to understand tabular-grain growth. There is, at present, no completely satisfactory model. I hope that this paper serves to clarify JEM.

Original manuscript received September 22, 1995.

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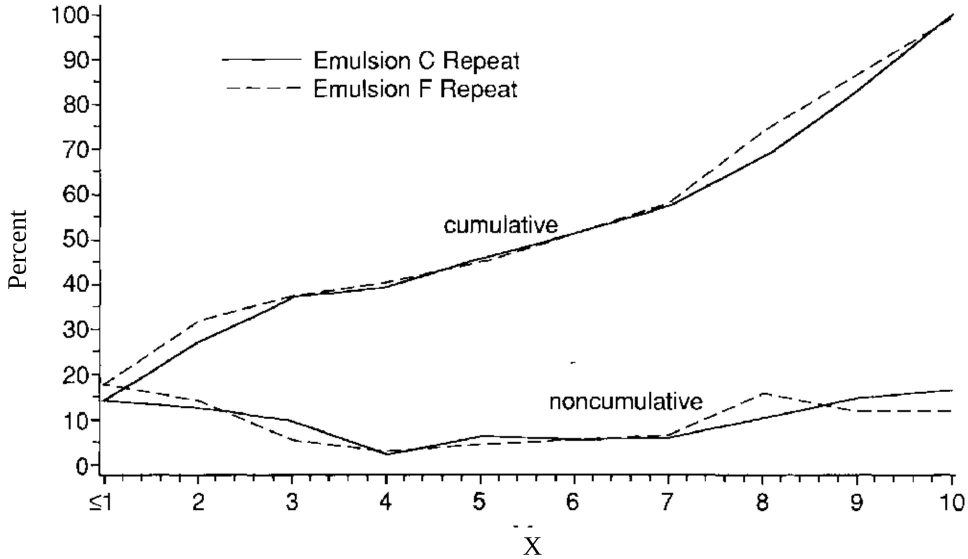
* Note that reliable thickness data were difficult to obtain on extremely polydisperse (in thickness) emulsions using the technique available at the time of JEM, manual counting of grains by Jamin-Lebedeff interference microscopy. (This technique yielded



Shape (long-edge-to-short-edge ratio 10:X)

Figure 1. Shape analysis results (prior to manual correction) of the triangular and hexagonal grain population of remakes of the pure bromide Emulsion C (average size 10.2 × 0.12 μm, decade ratio 3.5) and the iodide-banded Emulsion F (average size 10.4 × 0.12 μm, decade ratio 3.4) of JEM.[1] Within experimental error, these two emulsions have the same average size, polydispersity, and grain-shape distribution. There is no evidence for iodide poisoning of the edge structure of the iodide-banded emulsion.

a number-weighted average thickness.) Useful thickness data were not obtained for Emulsion F. In JEM, little significance was placed on average grain thickness data for these extremely polydisperse (both in thickness and in diameter) emulsions, except to show that the emulsions of this study were of very high aspect ratios and to mention the substantial thickness increase of a 10% iodide AgIBr emulsion. In a later publication, Emulsion F was used as the starting emulsion to grow edge shelves used to determine the separation between the parallel twin planes.[5] Because the subject of the study was edge shelf growth on the larger, easily measured tabular grains, the low mass grains were separated and discarded. The average thickness cited in this paper (0.23 μm ± 0.16) is after separation and is not the average thickness of Emulsion F of JEM. The separation procedure was unintentionally left out of this paper.[5] It is as follows: To 0.04 M of emulsion (35 g) was added 85 g of distilled water. The mixture was allowed to settle by gravity at 20°C for 2 h. The settled grains were resuspended in a 2% gelatin solution. This process was repeated three more times and the resuspended grains combined to give a total weight of 100 g. The resulting emulsion contained 0.11 mole silver, which is ~70% of the starting silver.

References

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4. J. Maskasky, *J. Imaging Sci.* **33**: 10 (1989); J. Maskasky, *ibid.* **33**: 13 (1989).
5. J. Maskasky, *J. Imaging Sci.* **31**: 93 (1987).