Partial Grain Development of an Epitaxially Sensitized Tabular Grain Emulsion

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Abstract

Polydisperse grain size distributions are commonly observed in many photographic emulsions. This complicates use of bulk analytical measurements to relate latent image detection and amplification to the developing grain fraction and the extent of development per grain. To properly account for grain size dispersity effects for an epitaxially sensitized tabular grain emulsion, the size distribution of the developing grains was determined as a function of exposure. Sensitometric measurements were compared to the microscopically determined developing grain fraction and extent of development. Results suggest a development mechanism involving exposure dependent amplification.

Introduction

The goal of this work was to understand more about the photographic response of an epitaxially sensitized tabular grain emulsion by measuring the developing grain fraction and extent of development per grain. In principle, the silver scale from a coating developed in conditions favoring whole grain development could be used to measure the developed grain fraction for a monodispersed emulsion. House et al.[1] studied amplification effects in monodisperse AgBr octahedral emulsions using similar bulk analytical methods. However, for a polydisperse emulsion, results are skewed by the volume weighted size distribution of the emulsion grains. Although these bulk analytical approaches are free from sample preparation problems, this method will not provide the grain-to-grain development center distributions obtained from grain counting techniques. Recently, Black et al.[2] used analytical electron microscopy and microanalysis techniques to analyze the extent of development for individual emulsion grains. These techniques are useful to understand the photographic response curve.

Since its inception in 1984,[3] the technology for epitaxial deposition and sensitization of tabular grains has continued to evolve.[4] Preparation of these emulsions generally involves using sensitizing dye to direct epitaxial deposits of varying composition to the corners of host emulsion grains, and can lead to improved photographic performance. Maskasky has shown by arrested development using black-and-white developers that development initiates at the corners of epitaxially sensitized grains.[5] This paper describes the characterization of the exposure dependent development properties of a tabular grain emulsion with mixed halide epitaxial depositions in a color forming single layer. To properly account for grain size dispersity effects, the size distribution of the developing grains was determined. Sensitometric measurements were compared to the microscopically determined developing grain fraction and extent of development.

Experimental

A mixed halide epitaxial phase was directed to the corners of a uniform 3% iodide emulsion by adsorption of a combination of green sensitizing dyes. The resulting emulsion was optimally chemically sensitized. This emulsion was coated in a hardened single-layer format containing 0.75 g/m^2 of silver, 1 g/m^2 of a cyan dye forming coupler, and 3.2 g/m^2 of gelatin with an overcoat also containing 3.2 g/m^2 of gelatin. Minus blue exposures were made through a 0-3 neutral density step tablet using a tungsten source with Daylight V and Kodak Wratten 9 filtration. To produce samples of partially developed grains, a coating was processed for 3'15" in process C-41 developer, immersed in a stop bath, washed, and dried. The partially developed silver halide grains were removed from the gelatin binder by soaking a portion of the coating in a dilute Takamine solution. Using scanning electron micrographs of the partially developed grains, emulsion grains were divided into categories according to the number of development sites observed per emulsion grain. The size distribution of the emulsion grains in each category was measured using image analysis. The percentage of the coated silver that developed in the C-41 process was measured by atomic absorption from a coating that had been developed and fixed. A third coating was processed in the standard C-41 process for sensitometric measurement.

Results and Discussion

The H&D curve for the coating processed in the standard C-41 process is shown in Figure 1. The arrows indicate the exposures used for microscopic analysis, corresponding to D-min, toe, mid-scale and D-max exposures.

Figure 2 shows typical examples of partially developed and undeveloped grains, which received the toe exposure. About 57 percent of the sampled grains exhibited development centers. These development centers appear to initiate at the grain corners. For this exposure level, development was generally confined to a single corner of the developing grain. Figure 3 shows the number weighted size distribution for emulsion grains in the step that received the toe exposure. Non-developing grains were separated from the developing grains during the grain sizing procedure, and are shown as separate distributions in Figure 3. The average size of the non-developing grains is slightly smaller than for the developing grains.

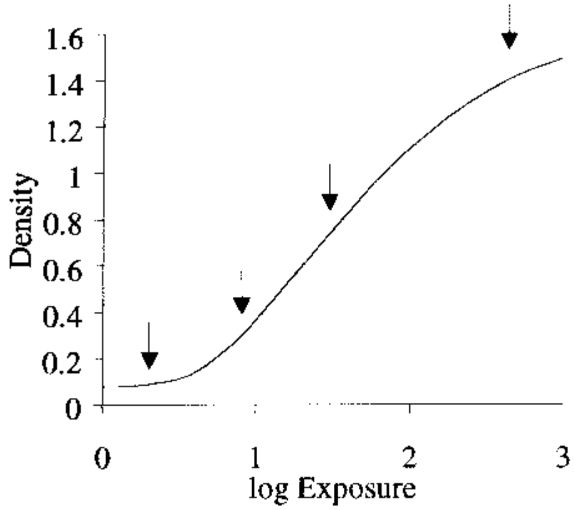

Figure 1. H&D curve for silver halide emulsion coating processed in C-41 process. Arrows indicate the exposures used for microscopic characterization of the partially developed grains. (a) D-min , (b) Toe, (c) mid-scale, and (d) D-max.

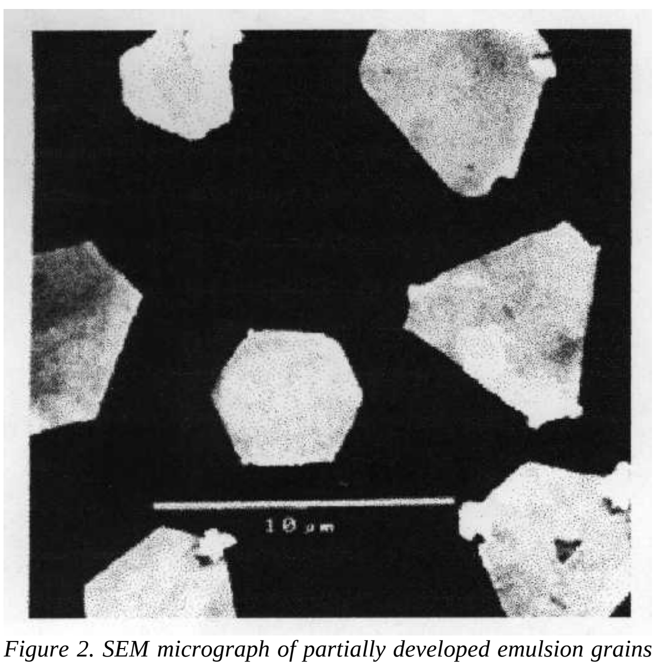

Figure 2. SEM micrograph of partially developed emulsion grains removed from a toe exposed step. The grains were developed in the C-41 developer for 3'15", immersed in a stop bath and wash prior to removal from the coating.

An SEM micrograph of partially developed grains that received a mid-scale exposure is shown in Figure 4. The additional exposure has resulted in multiple development sites per grain. Comparison of the development centers in Figures 2 and 4 show the size of the development sites appear to be independent of exposure. With the mid-scale exposure, 90 percent of the sampled emulsion grains exhibited development centers. There was a clear shift in the development center distribution, particularly for the largest grains. Figure 5 shows that 40 percent of the emulsion grains had two, three, or four development sites. The number weighted average size of grains with a single development center was ca. 0.96 um3, progressing to 1.5, 1.8 and 1.9 um^3 for emulsion grains with 2, 3, or 4 development centers per grain, respectively. The non-imaging grain fraction comprised only 3 percent of the coated silver.

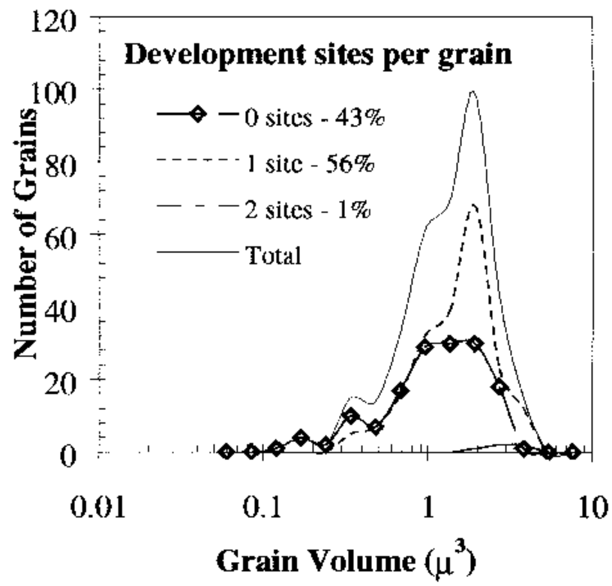

Figure 3. Number weighted development center distribution for partially developed grains removed from a toe exposed step.

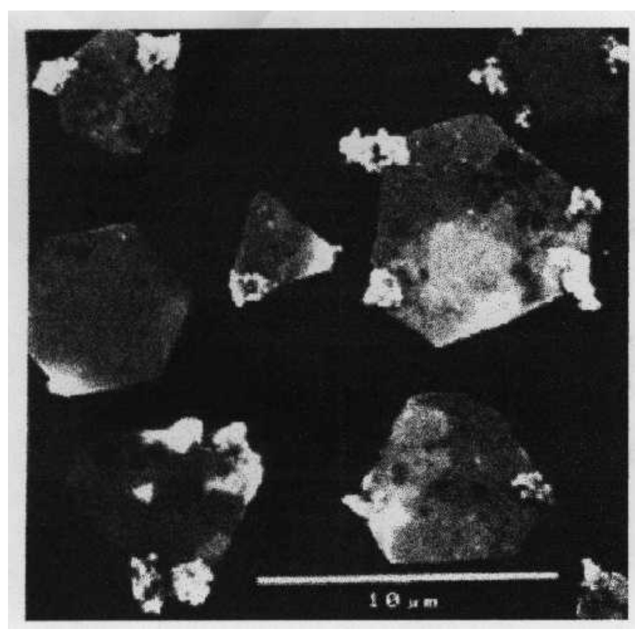

Figure 4. SEM micrograph of partially developed grains removed from a mid-scale exposed step. The grains were developed in the process C-41 developer for 3'15", immersed in a stop bath and wash prior to removal from the coating. The size of the development centers appears similar to development sites in the toe exposed grains.

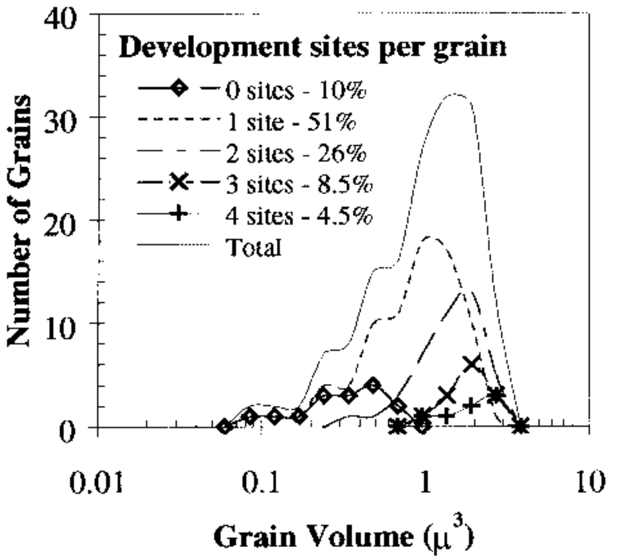

Figure 5. Number weighted development site distribution for partially developed grains removed from a mid-scale exposed step.

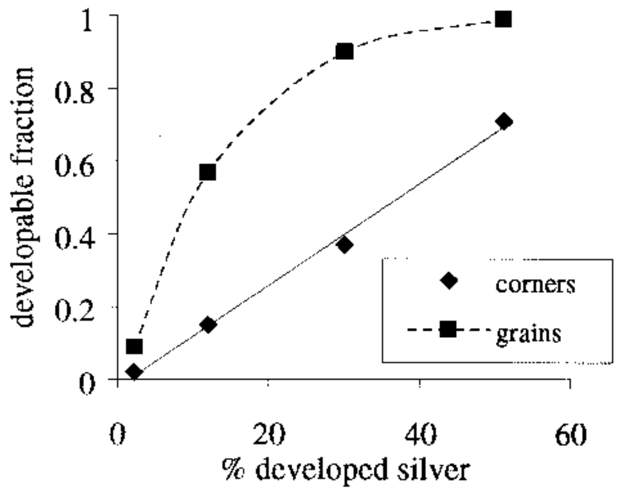

Figure 6. Developable grain fraction and developable corner fraction plotted vs the percent of the coated silver, which developed at 3'15" in the C-41 developer.

The SEM micrographs suggested that the development centers were of similar size throughout the exposure range, but that the number of imaging corners per grain increased with exposure. Benefits from this type of development have been described by Van Metter and others.[6] To quantify these observations, the amount of silver developed was measured in a C-41 processed coating, omitting the bleach step. Figure 6 shows the developable grain fraction and developable corner fraction obtained from grain counting plotted as a function of the percent of the coated silver that developed in the C-41 process. The amount of silver developed was proportional to the developable fraction of imaging corners. These results also suggest that for those grains with multiple development sites, amplification increases with exposure.

References

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