

# The Effect of Environment on Latent Image Formation and Stability

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## Abstract

This paper uses an unsensitized emulsion and two chemically sensitized emulsions to investigate the effect of oxygen and water vapor on latent image formation and stability. Storage of a latent image in a humidified environment will induce a speed loss in some emulsions. The unsensitized emulsion was the most sensitive to environment while the sulfur-plus-gold-sensitized emulsion was not. This is presumably due to the composition and size of the latent image. Maximum changes over time require the presence of both oxygen and water vapor. Extended development and gold latensification restored some of the speed loss observed with the unsensitized emulsion. The unrecovered speed loss is due to either latent-image centers being completely oxidized or being too small to respond to chemical latensification.

## I. Introduction

The effect of environment on photographic materials has been studied for over 120 years. Previous investigators have found that atmospheric components, such as water vapor and oxygen, can affect the photographic speed and developable density of a silver halide emulsion. Most investigators have used exposure times of tens to thousands of seconds to study the effect of environment on a multitude of emulsions. Extended exposure times are used to maximize the signal-to-noise ratio, but they can also cause a significant amount of low-intensity reciprocity failure (LIRF) in photographic materials.

Some work has been performed to examine the relationship between the environment and latent-image stability over time. Difficulties associated with this type of study have limited the scope of these investigations. Examples of these difficulties include pre- and post-exposure conditioning of the test emulsion and the handling of materials in a reproducible manner.

The purpose of this study was to investigate how oxygen and water vapor may affect photographic materi-

als using an exposure time that causes minimal LIRF. The effects of oxygen and water vapor were determined by observing latent-image stability and initial photographic speed in several environments. An unsensitized and two chemically sensitized emulsions were used to observe the effect of chemical sensitization on latent-image formation and stability.

## II. Previous Investigations

The following paragraphs outline several selected investigations relevant to this thesis. *Theory of the Photographic Process*, edited by T. H. James, is an additional reference for many of the topics discussed in this section.<sup>1</sup>

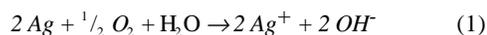
In 1872, Lt. Col. Wortley<sup>2</sup> wrote to the *British Journal of Photography* concerning photographic sensitivity. In his letter, Wortley states that he treated his photographic plates to "a couple of hours of strong heat" and that this increased the speed of his emulsion. He goes on to state that this increase in speed was lost after three days.

The editors of the *Journal* studied the photographic plates Wortley sent with his letter and noted that the colonel was reporting from Naples, Italy. The editors<sup>3</sup> suggested that he was observing the effect of relative humidity on his photographic plates. Naples, being a Mediterranean coastal city, has an elevated relative humidity because of the warm, sea air. Wortley's heat treatment removed atmospheric water from the plates and increased the photographic speed of the emulsion. After three days of reconditioning to the ambient climate of Naples, his plates had returned to their "ordinary degree of humidity" and the speed gain was lost.

James<sup>4</sup> discussed in detail the effects of pressure, moisture, and oxygen on latent-image formation. Pressure effects can be generated by using a dry gas or vacuum in emulsion studies. A dry environment will remove water from the gelatin matrix and cause it to contract. As the gelatin contracts, it can exert pressures up to 2000 kg/sq cm and will desensitize a silver halide grain. It is thought that this desensitization is caused by a deformation of the

silver halide grain, although the mechanism is not understood.<sup>5</sup> If the pressure is kept below ~1000 kg/sq cm, this desensitization is reversible. At pressures much above this level, desensitization is permanent.

Changing the moisture and oxygen content of an emulsion layer may affect latent-image formation and its stability. One possible mechanism<sup>4</sup> by which water and oxygen may affect the latent image is:



It is easy to assume from Reaction (1) that removing oxygen from an emulsion layer can increase the stability of the latent image. James notes, however, that not all emulsion layers are sensitive to oxygen. He reports that previous investigators are almost evenly split regarding the effect of oxygen on latent-image formation and its stability. It is important to note that these other investigators used a variety of emulsions that were chemically and/or spectrally sensitized

Water may have both a physical and a chemical role in latent-image formation and stability. One such chemical role is presented in Reaction (1) above. The physical role of water may be to swell the gelatin matrix. Swollen gelatin exerts almost no pressure on the surface of a silver halide grain, preventing any pressure-induced desensitization from occurring.<sup>4</sup> Swollen gelatin is also more permeable than dried gelatin. The more permeable the matrix is, the easier it is for oxygen and water vapor to reach the grain surface.

### III. Experimental

#### 3.1 Emulsion.

A 0.45  $\mu$ m AgBr octahedral emulsion was precipitated in gelatin. A sample of this emulsion was sulfur sensitized using 1.33 mg sodium thiosulfate/Ag mole. A second sample was sulfur-plus-gold sensitized using the same sodium thiosulfate level plus the addition of 2.00 mg potassium chloroaurate/Ag mole. These levels of chemical sensitizers are historically known to provide near optimum sensitization conditions for this emulsion.<sup>6</sup>

The unsensitized, sulfur-, and the sulfur-plus-gold-sensitized emulsions were coated on a clear acetate support at 100 mg Ag/sq ft with a hardened gelatin overcoat. The emulsion melts were adjusted to pH 5.5 and vAg 90 mV at 40°C before coating. Materials were cut into 12 inch by 35 mm film strips for ease of handling.

#### 3.2 Sensitometer.

Pre- and post-exposure conditioning of the film samples was done using an environmental sensitometer. This device is a modification of the stainless steel bell jar used by James, et al.<sup>4,7</sup> One modification is the incorporation of a 0 to 3.0 density, 15-step tablet into the exposure window. Eighteen film samples, in six groups of three, can be conditioned, exposed, and stored in a controlled environment until processing.

Exposure times of 0.001 and 1 s were used to investigate the photographic response of the test emulsions in a regime where there is minimal or no reciprocity failure. The light source for the 0.001 s exposure was an EG&G Mark VII sensitometer. The EG&G sensitometer was placed so that its light source was perpendicular to the exposure window of the environmental sensitometer. A water-cooled quartz-halogen lamp was used for the 1 s exposure.

#### 3.3 Environments.

Seven different environments were chosen for study at the 0.001 s exposure time. They were 0% RH vacuum, air, and nitrogen; 20% RH air and nitrogen; and 80% RH air and nitrogen. Environments were restricted to 0% RH vacuum, air, and nitrogen; 20% RH air and nitrogen for experiments using a 1 s exposure time.

Materials were conditioned in a test environment for 72 h before the start of an experiment. A vacuum of 0.05 torr was generated by a mechanical pump with a molecular sieve trap to minimize oil back streaming. Nitrogen was supplied using commercially available compressed gas cylinders. Air flow was provided by the building air supply. A preliminary study was done to compare the building air supply to a compressed air cylinder. No differences in sensitometry were found.

Relative humidity was controlled by bubbling the test gas through distilled water, cooling it to the desired wet bulb temperature using a water/glycol bath, and allowing the gas to equilibrate to room temperature before its introduction into the environmental sensitometer. Relative humidity was checked using a hand held temperature/relative humidity probe placed in the gas effluent of the sensitometer.

#### 3.4 Latent-Image Hold Times.

Six different times between exposing and processing were chosen to study latent-image stability. They were 72, 48, 24, 4, 1, and 0.5 h of delay between exposing and processing. It was not possible to generate latent-image hold times shorter than 0.5 h because of the time required to unseal the environmental sensitometer, remove the samples, and transport them to the processing laboratory.

An error of  $\pm 2$  s was allowed between the scheduled and actual exposure time during the last four hours of the latent-image hold time profile. An error of  $\pm 15$  min was allowed for the 24, 48, and 72 h exposure points.

#### 3.5 Processing.

Fresh, commercially available processing chemistry was used. Kodak D-19 Developer and Kodak Fixer were made within 72 h of scheduled processing. Film samples were developed for 6 min; immersed in a stop bath for 30 s; fixed for 5.5 min; and washed for 5 min. A 2 s burst of nitrogen was used every 7 s to agitate the processing chemistry. A Wing-Lynch temperature controller was used to maintain a 20°C processing temperature. After

washing, the samples were placed in a forced-air drying oven for 30 min.

Six min of processing in Kodak D-19 Developer appears to develop five-atom and larger silver centers.<sup>8,9</sup> After 48 min of development, approximately 10% of the three-atom silver centers will initiate development, whereas larger metal clusters will have a 100% probability of initiating development. A latent image incorporating gold will be developable after 12 min of processing if there are at least three metal atoms in the cluster.

The optimum method of processing would be to process all 18 samples together in the same tank of developer. This procedure would also require a controlled-temperature processing tank that was several times larger than what was available. The best alternative was to process each sensitization type individually.

It was not physically possible to place three sets of samples into three separate processing tanks simultaneously. To compensate for this, the processing of the unsensitized and sulfur-plus-gold-sensitized emulsion samples were offset by  $\pm 10$  s around the desired processing time. The sulfur-sensitized materials were processed at 0.5 h after the last exposure. The unsensitized emulsion samples were placed in the developer 10 s before the sulfur-sensitized emulsion samples. The sulfur-plus-gold-sensitized materials were placed in processing chemistry 10 s after sulfur-sensitized materials. Error in processing time was estimated to be  $\pm 1$  s.

### 3.6 Gold Latensification.

Gold latensification was performed on samples of the unsensitized emulsion. Materials were placed in a gold bath as described by James, Vanselow, and Quirk<sup>10</sup> for 10 min and then submerged for 5 min in a 1-g potassium bromide/L solution. Immediately following the potassium bromide bath, materials were processed using the procedure described in section 3.5.

### 3.7 Sensitometry.

Film densities were measured using an X-rite densitometer. These densities were transferred to a computer that calculated photographic speed data. The point used for comparison in this thesis is photographic speed at base fog plus 0.50 density units (speed at D-min + 0.50). This point is approximately mid-scale on the density-log relative exposure (D-log E) curves for all the materials examined.

## IV. Results

### 4.1. Changes in Photographic Speed Over 72 h of Latent-Image Hold Time.

The unsensitized emulsion had the greatest loss of latent image over the 72 h test period. Tables I and II indicate that a speed loss of 0.18 log E was observed in samples treated in a 20% RH air environment at both exposure times. The speed loss at 80% RH was not statis-

tically different from the 20% RH air treatment at the 0.001 s exposure time. A speed loss was observed in 0% RH air at a 1 s exposure time. The speed loss observed at the 0.001 s exposure in 0% RH air was not statistically significant. Treatment of materials in the vacuum and nitrogen environments resulted in a stable latent image. Changes in speed for the unsensitized emulsion are shown in Figures 3 and 4.

**Table I. Changes in Speed for the Unsensitized Emulsion at a 0.001 s Exposure.**

Delta Speed (log E) Relative to the 0.5 h Point.				
Hold Time (h)	Vac.	0% RH Air	20% RH Air	80% RH Air
0.5 h	0	0	0	0
1 h	0.02	0.03	-0.01	-0.02
4 h	0.02	0	-0.03	-0.03
24 h	0.04	-0.01	-0.12	-0.10
48 h	0.04	-0.04	-0.14	-0.12
72 h	0.05	-0.04	-0.18	-0.15

Delta Speed (log E) Relative to the 0.5 h Point.			
Hold Time (h)	0% RH N2	20% RH N2	80% RH N2
0.5 h	0	0	0
1 h	0	0	0.01
4 h	0.01	0.03	0.03
24 h	0.03	0.03	0.02
48 h	0.03	0.03	0.03
72 h	0.00	0.03	0.03

**Table II. Changes in Speed for the Unsensitized Emulsion at a 1 s Exposure.**

Delta Speed (log E) Relative to the 0.5 h Point.					
Hold Time (h)	Vac.	0% RH Air	20% RH Air	0% RH N2	20% RH N2
0.5 h	0	0	0	0	0
1 h	0.02	-0.02	0	0	0.03
4 h	0.02	-0.02	-0.05	0.02	0.04
24 h	0.02	-0.05	-0.12	0.02	0.05
48 h	0.05	-0.06	-0.15	0.03	0.06
72 h	0.03	-0.09	-0.18	0.03	0.05

With two exceptions, chemical sensitization resulted in a stable latent image for all environments studied. The first exception is for the sulfur-sensitized emulsion samples treated in humidified air. At a 0.001 s exposure time,

a speed loss of 0.06 log E was observed over the first 24 h of latent-image hold. There was no continued fading of the latent image after the 24 h point.

The second exception is a speed gain between the 0.5 h and the 1 h latent-image hold times for the sulfur-plus-gold-sensitized emulsion. This gain was seen at the 0.001 s exposure, but not at the 1 s exposure time. There are doubts whether this speed gain is real.

#### 4.2 Recovery of Subdevelopable Latent Images Through Changes in Minimum Developable Size.

Experiments were performed to attempt recovery of the lost latent image in the unsensitized emulsion after 72 h of latent image hold time. The development time was extended to detect smaller latent-image sites. Three sets of six unsensitized emulsion samples were treated in a 20% RH air environment and a 72 h latent-image-hold-time profile was performed. Development times of 6, 24, and 48 min were used. Results are in Table III.

**Table III. Delta Speeds Obtained During Extended Development for the Unsensitized Emulsion Over 72 h of Latent-Image Hold. 0.001 s Exposure Time.**

Delta Speed at D-min + 0.50 (log E) from 0.5 h Point			
Hold Time (h)	6 Min	24 Min	48 Min
0.5 h	0	0	0
1 h	-0.05	-0.02	0.02
4 h	-0.09	-0.04	-0.03
24 h	-0.13	-0.10	-0.06
48 h	-0.18	-0.11	-0.11
72 h	-0.20	-0.16	-0.13

A development time of 6 min resulted in a speed loss of 0.20 log E over the 72 h hold time profile. Twenty-four min of development reduced the speed loss to 0.16 log E. After 48 min of development, the speed loss was reduced to 0.13 log E. Extended development did recover some of the latent-image sites that had faded to below an  $Ag_5$  center.

Gold latensification was also used to attempt recovery of the sub-developable latent-image sites. Three sets of unsensitized emulsion samples were conditioned in a 20% RH air environment and a 72 h latent-image-hold-time profile was performed. One set was gold latensified using the procedure outlined in Section 3.6 of this thesis. A second set was submerged for 15 min in a solution containing 1 g potassium bromide/L. This pre-soaking treatment would account for any effect of gelatin swelling on development. The third set was kept in room conditions and was not pre-soaked before development. These materials were developed in Kodak D-19 Developer for 6 min.

Both the samples kept under room conditions and those presoaked in the bromide bath prior to development lost 0.18 log E over 72 h. Samples receiving gold latensi-

fication lost 0.08 log E of speed during the same time period. This indicates that at least some of the lost speed is due to latent-image sites degrading to a sub-developable size. It is not known how much of the residual speed loss is due to latent-image sites not made developable through gold latensification, or to latent-image sites being destroyed.

**Table IV. Changes in Speeds Obtained During Gold Latensification for the Unsensitized Emulsion Over 72 h of Latent-Image Hold. 0.001 s Exposure Time.**

Delta Speed at D-min + 0.50 (log E) from 0.5 h Point.			
Hold Time (h)	Gold Latensification	Bromide Bath	20% RH Air
0.5	0	0	0
1	0	-0.02	-0.01
4	-0.02	-0.04	-0.03
24	-0.05	-0.11	-0.11
48	-0.08	-0.15	-0.14
72	-0.08	-0.18	-0.18

## V. Discussion

### 5.1 Latent-Image Stability Over 72 h of Latent-Image Hold Time.

Unsensitized emulsion samples treated in either a dry or humidified nitrogen environment do not show a speed loss over 72 h of latent-image hold time whereas treatment in an air environment does induce a speed loss.

The lack of a speed loss in the nitrogen environments indicates that nitrogen does not affect the stability of a latent image. The data also indicates that water vapor in an inert atmosphere will not degrade a latent image over 72 h of latent-image hold. These statements are also valid for the chemically sensitized emulsions studied in this thesis.

A speed loss was observed after treatment in dry air using a 1 s exposure. There is a trend, not statistically proven, indicating that treatment in a 0% RH air environment and using a 0.001 s exposure will also result in a speed loss over time. Extending the length of the latent-image hold time would verify whether a speed loss actually occurs as a result of treatment in 0% RH air.

Observing a speed loss after treatment in a dry air environment does not mean that oxygen is the only material available to react with a latent image. Flowing 0% RH air over the emulsion will dry the gelatin. However, not all the water will be removed.<sup>11</sup> Some water will be trapped through hydrogen bonding with the gelatin or may be trapped on the grain surface. Water and oxygen can then degrade the latent image.

There are two possible reactions between oxygen and a latent image.



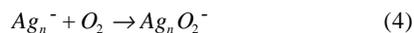


Reaction (3) is equivalent to Reaction (2), but appropriate for the acidic nature of the coating.

Water vapor may participate in the latent-image oxidation process as a reactant or as a transporter of protons. Evidence supporting the synergistic action of oxygen and water vapor is seen in the data for the unsensitized emulsion. A speed loss of 0.18 log E is observed after treatment in a 20% RH air environment for 72 h. Treatment in a humidified air environment is much more conducive to speed loss than treatment in a 0% RH air environment. The presence of water is important to obtain maximum speed loss.

A third possible way that water can participate in the loss of latent image is by physically swelling the gelatin. Swelling the gelatin will increase the permeability of the emulsion layer. This will facilitate the flow of reactants to a latent-image site. It is quite possible that water may participate in all three ways simultaneously.

Colton and Wiegand<sup>11</sup> have indicated that water is still present in a dried emulsion layer. The question can be asked whether oxygen can degrade a latent image without the presence of water. Lee and Ervin<sup>12</sup> found that it is possible for a negatively charged gas-phase silver cluster to react with oxygen.



This reaction may be a part of the reaction sequence shown in Reactions (2) and (3). The reaction rate is approximately ten times greater for an even-sized silver cluster than for an odd-sized silver cluster. Also, larger silver clusters react faster than smaller clusters.

Gas-phase gold clusters will react with oxygen in a fashion similar to that of gas-phase silver clusters. However, Lee and Ervin found that the oxidation rate for gold clusters is approximately ten times slower than the oxidation rate for silver clusters. This result would suggest that a gold-incorporated latent image would be less prone to oxidation than a silver-only latent image. The observations in this thesis for the sulfur-plus-gold-sensitized emulsions support this hypothesis.

The unsensitized emulsion was found to be more susceptible to latent-image fading in a humidified environment than the sulfur-sensitized emulsion. The observations made by Lee and Ervin above may explain this result. An unsensitized emulsion often forms only one latent image when exposed to light. Sulfur sensitization directs latent-image formation to multiple sites on the surface of the silver halide grain. Assuming that the two materials have received exposures that will result in comparable densities and that the latent images in question are larger than the minimum developable size, the single latent image on the unsensitized emulsion grain will be larger than the multiple latent-images formed on the sulfur-sensitized emulsion grain.<sup>13</sup> Computer simulations of latent-image formation performed by Hailstone<sup>14</sup> support this statement. As a result, the larger latent im-

age on the unsensitized emulsion grain may have a greater probability of being oxidized than the smaller, multiple latent images on the sulfur-sensitized emulsion grain.

Investigations performed both by Tani and by Matsubara and Levy may also support the hypothesis that both size and composition of a latent image can affect the potential for oxidation. Tani<sup>15</sup> investigated the redox potential of the latent image by submerging variously sensitized emulsion samples in a redox buffer for up to 65 h and then processing the materials. He found that the unsensitized and the sulfur-sensitized emulsions both lost similar amounts of latent image caused by treatment in the buffer. The sulfur-plus-gold-sensitized emulsion was stable. He attributes this to the high electronegative value of gold in the silver-gold centers.

Matsubara and Levy<sup>16</sup> performed similar experiments using a five minute treatment in the same type of redox buffer. They also found that the sulfur-plus-gold-sensitized emulsion was stable. However, Matsubara and Levy found that the unsensitized emulsion lost more latent image after treatment in the redox buffer than the sulfur-sensitized emulsion. They suggested that this observation may, in part, be due to silver sulfide being incorporated into the latent image of a sulfur-sensitized emulsion. The presence of silver sulfide in the latent image may change the oxidation characteristics of the latent image. This incorporation could explain why, at a 5-atom minimum developable size, a latent image in a sulfur-sensitized emulsion is more stable than a 5-atom latent image in an unsensitized emulsion.

Both investigations may be correct. Tani assumed that he had reached equilibrium after 65 h of treatment. Matsubara and Levy assumed that they were not in equilibrium. It is possible that the unsensitized emulsion has a higher initial rate of latent-image oxidation than the sulfur-sensitized emulsion. Once equilibrium is reached in the redox buffer, the unsensitized and sulfur-sensitized emulsions have similar loss of latent image. The difference in latent-image loss rates between the unsensitized and the sulfur-sensitized emulsions in humidified air may support Matsubara and Levy's observations. Extending the latent-image hold time for the unsensitized and sulfur-sensitized emulsions in a humidified air environment could indicate whether an equilibrium would actually be obtained.

After 72 h of latent-image hold, gold latensification and extended development restored some, but not all the speed loss observed in the unsensitized emulsion. Extending development time can develop smaller latent-image centers. After 48 min of development in D-19, the speed loss was reduced to 0.13 log E. This partial recovery of the speed loss is probably due to latent-image sites smaller than an Ag<sub>5</sub> being developed.

Gold latensification reduced the speed loss from 0.18 log E to 0.08 log E after 72 h of latent-image hold. This result indicates a portion of the observed fading is due to latent images becoming too small for conventional de-

velopment, but are still recoverable through gold latensification. Gold can combine with subdevelopable silver centers and form a developable silver-gold latent image.<sup>17</sup> It is not known what percentage of the remaining speed loss is due to silver centers that are not gold latensifiable, or to completely decayed latent-image centers.

## VI. Conclusions

1. At exposure times that cause little or no low-intensity reciprocity failure, vacuum treatment of an unsensitized or chemically sensitized emulsion can result in speeds significantly slower than those found in a humidified environment. This result is opposite what many investigators have seen when a long duration exposure has been used. The extended pre-conditioning technique used in this thesis may have removed additional oxygen and water vapor from the photographic materials. This procedure may have affected this speed difference between treatments in vacuum and humidified environments.

2. The unsensitized emulsion had a reduction in the speed difference between treatments in vacuum and humidified environments as the exposure time was increased. This reduction in the speed difference may be due to the onset of low-intensity reciprocity failure in the unsensitized emulsion. The sulfur- and sulfur-plus-gold-sensitized emulsions were not sensitive to this change in exposure time.

3. Storage of a latent image in a humidified air environment will induce a speed loss in some emulsions. The unsensitized emulsion lost significant speed over time whereas the chemically sensitized emulsions were relatively stable. Chemical sensitization can affect the ability of a latent image to be oxidized.

4. Humidified nitrogen environments did not induce a speed loss over time. This result indicates that water alone does not affect the stability of a latent image in the unsensitized, or chemically sensitized, emulsions used in this thesis.

5. Oxygen appears to be the primary oxidizing agent for latent-image decay. The presence of atmospheric water vapor can accelerate this decay process. Both the size of the latent image and the sensitization of the silver halide grain can influence the stability of a latent image over time. Latent images incorporating gold were stable over the 72 h of latent-image hold time. The latent image in an unsensitized emulsion grain is much more readily oxidized.

6. Speed losses in the unsensitized emulsion treated in humidified air are a result of latent-image sites being

made undevelopable. Gold latensification restored approximately 50% of the lost speed. The unrecoverable speed loss is due to latent-image centers being either completely oxidized or too small to respond to the chemical latensification procedures used.

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