

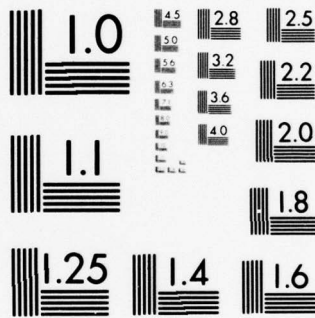
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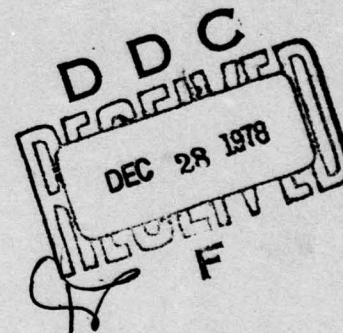
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CAMERA SPEED DRY SILVER FILM

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FEBRUARY 1978

TECHNICAL REPORT AFAL-TR-78-8
Final Report covers Period May 1974 — September 1977

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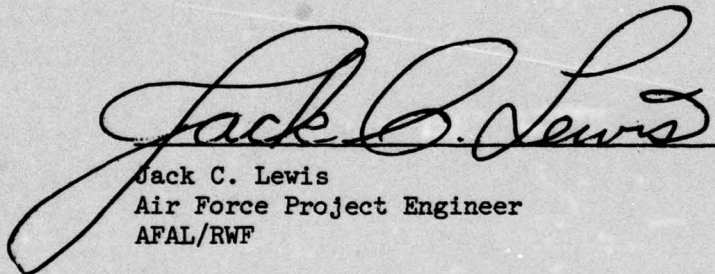
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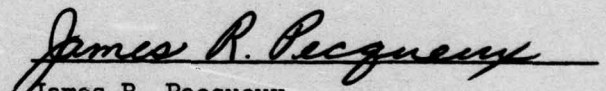
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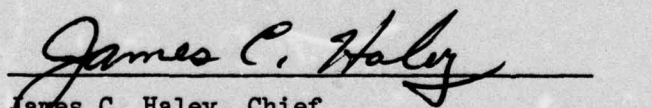
This report has been reviewed by the Information Office (OI) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.


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FOR THE COMMANDER


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The objective of this study was to determine the technical feasibility of formulating, coating, and testing experimental laboratory coatings of dry silver films with panchromatic photosensitivity of AEI=20, and capable of being thermally processed from exposed latent image form to usefully permanent visible silver image form within 30 seconds or less using temperatures less than 300F.

During this program, coating variants were made whereby the halidization of the silver behenates were modified, and mild organic reducing agents capable of promoting the thermographic reaction were found to be most useful. By adaptation of these variants to previous dry silver film formulations, films were produced experimentally which had aerial exposure indexes of 1.6 through 12.0

→ The dry silver films coated with high photosensitivity were found to have high base fog, and tended to have short, useful package shelf life as well as limited image permanence, and limited reproducibility.

Aspects of the experimental panchromatic sensitive dry silver films which require further work to provide a more useful film are higher photosensitivity with latent image stability and light stability.
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FOREWORD

This report describes work performed by the Imaging Products Project of Microfilm Products Division of Minnesota Mining and Manufacturing Company (3M), St. Paul, Minnesota, under USAF Contract No. F33615-74-C-1081, "Camera Speed Dry Silver Film AEI-20." The work was administered under the direction of the Air Force Avionics Laboratory, Reconnaissance and Weapon Delivery Division, Mr. Jack C. Lewis, AFAL/RWF-4, Project Engineer.

This final report covers laboratory work conducted from May, 1974 to September, 1977. Principal investigators for the contract were Mr. Robert Russell (Advanced Development Chemist) and Mr. Ken Metz (Chemist). Other 3M personnel that participated in this program were: Dr. Jack E. Reece (Research Specialist), Mr. Steve Gaudette (Technician), Mr. Charles Gomez (Chemist), Mr. Ross Serold (Supervisor), and Mr. David Weigel (Supervisor).

Special appreciation is given to Ms. Robin Atkins for her secretarial assistance in preparing this report.

The authors are indebted to all of these individuals for their valuable contributions and to Mr. Jack C. Lewis, Project Engineer from the USAF Avionics Laboratory, for his consultation, guidance, and assistance in testing material and report preparation.

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SECTION I

INTRODUCTION

The need exists for a photographic film that can be processed without the requirements for pure water, chemicals, fixed concentrations, and disposal of processing solutions. The properties of Dry Silver answer this need with their thermal development (Figure 1). The only requirement of Dry Silver is that a source of heat (standard temperatures below 300 F) be present.¹ Commercially available Dry Silver film is primarily used as a recording material. Many uses exist for a more sensitive Dry Silver film.

Prior to this contract the 3M Company had done extensive research on formulation of Dry Silver toward sufficient photosensitivity, image quality, processing reliability, and simplicity for use in commercial applications. The 3M Company was also completing a one year contract, Camera Speed Dry Silver Film Aerial Exposure Index = 5.² The AEI-5 contract was designed to test a Dry Silver state-of-the-art film in a specific system by the Air Force. The AEI-5 contract placed 3M camera speed Dry Silver film within the lower limits of recording film. The AEI=20 contract was designed to provide added work on increasing the sensitivity of camera speed Dry Silver film.

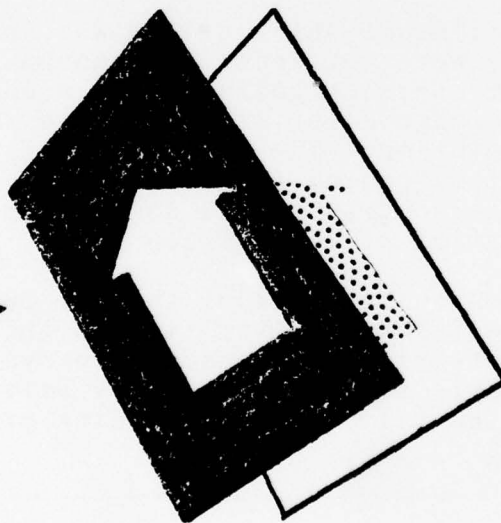
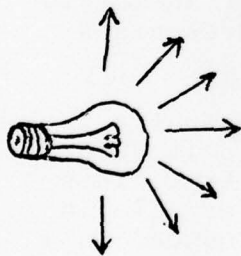
In the laboratory, films of AEI 1.6-12.0 were prepared. Although these films were not sensitive enough to meet contract goals, they did meet contract goals in the areas of spectral sensitivity, gamma, image color, log exposure range, film processing, and resolution. The emulsion variables of the faster laboratory films proved too critical to transfer to the pilot plant. The contract final sample represents a reproducible formulation at the AEI 1.6 level.

This report describes the results of the AEI=20 contract, which supplied a special Dry Silver film to the Air Force for evaluation in a tactical reconnaissance system. This includes the obtainable sensitivity of Dry Silver film in the laboratory and the film capable of being produced in a pilot plant coating.

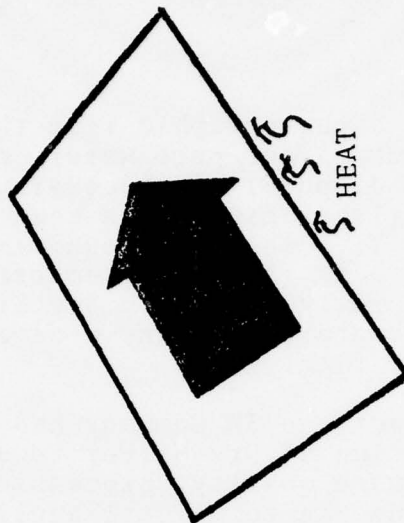
¹ Chemistry of Dry Silver in Appendix 1.

² Camera Speed Dry Silver Film Aerial Exposure Index = 5, Contract F33615-73-6-1273, is discussed in the contract's final report AFAL-TR-75-11.

LIGHT SOURCE



LATENT IMAGE
FORMATION



AMPLIFICATION OF
LATENT IMAGE

FIGURE 1 - Exposure and Processing of 3M Dry Silver

SECTION II

OBJECTIVES

The objective of the program was to provide the U.S. Air Force with a Dry Silver film whose sensitivity was within the sensitivity range of aerial film. The test application of the film was to be in a tactical reconnaissance system. Thus the objectives of the contract were such that the final film would be applicable to tactical reconnaissance systems. The desired characteristics were defined as:

1. Spectral Sensitivity: The films should have panchromatic sensitivity from 400 through 720 nanometers of the electromagnetic spectrum.
2. Photosensitivity: The films should have an Aerial Exposure Index of 20 as a goal. AEI is defined as $1/2 E$ where E is the exposure in meter candles seconds to a 5900 - 6100 Kelvin light source required to produce a density response of 0.6 slope of the measured bar gamma.

Photosensitivity Reciprocity Law Failure:

The films should have the Exposure Index of 20 as a goal at exposure times of 1/500, 1/1000, 1/2000, and 1/4000 second exposure times since these represent shutter speeds normally used in Tactical Reconnaissance applications. The films should be designed to have minimum reciprocity law failure response within this exposure time range.

3. Bar Gamma: The films should have a contrast response (gamma) from 1.2 to 1.6 as a goal, using thermal processing techniques designed for easy control, repeatability, and processing to a highly stable image form.
4. Image Color: The films should produce a visibly neutral black image when thermally processed to stable image form and viewed on a Richard's Photo Interpretation Light Table equipped with daylight type fluorescent tube lamps.
5. Useful Log Exposure Range: The films should have a 2.0 useful Log Exposure range as a goal.
6. Image Resolution: The films should record at least 200 cy/mm of a MIL-STD-150A high contrast (100 to 1 brightness range, 3.0 density difference) resolution test target pattern, as a goal.
7. Film Base: The films shall be coated on optically clear 3 mil thick polyester film base.

Objectives - continued

8. Film Processing: As a goal, the films should process to highly stable image form in 30 seconds or less time and at temperatures not exceeding 300F. The films should be designed to produce the required sensitometric, image definition, and image permanency characteristics, insofar as is possible, by a "to-completion" process whereby nearly all of the thermally releasable processing chemistry is consumed during the thermal processing, thereby resulting in a highly stable image.
9. Latent Image Stability: As a goal, the films should retain exposed latent images without sensitivity loss for a period of at least 24 hours from the time of exposure until the time of development.
10. Image Permanency: As a design goal, the films should have image permanency such that exposed and processed imagery will survive a seven day exposure on the Richard's Photo Interpretation Light Table surface with the fluorescent tube lamps operated at approximately 1000 foot lamberts. Minimum density should not increase by more than 0.10 after this seven day light table image aging test.
11. Dimensional Stability: The film should be of sufficient dimensional stability, so that shrinkage shall be less than 1/2 percent in either the widthwise or lengthwise dimension of the film when exposed to a Reseau Grille of known dimensions and after normal thermal image processing.
12. Useful Package Shelf Life: As a goal, the films should have good photographic shelf life in the original unopened package for a period of at least 12 months at storage temperatures below 50F.

The final sample was to incorporate these characteristics such that the Air Force could test the applicability of Dry Silver recording speed photosensitive film in actual Air Force photographic equipment.

SECTION III

PROGRESS

The photosensitive material which was prepared to meet the contract objectives is a negative working, panchromatic, photosensitive Dry Silver film which is developed by heat. This film is approximately five to 100 times more sensitive than commercial 3M Dry Silver films in the commercial film's spectral regions. Yet the contract film employs the same basic materials with refinements. In all Dry Silver films, the light sensitive component is silver halide which forms the latent image. The unique aspect of Dry Silver is the amplification of the image. The image is developed by a thermographic reaction which is catalyzed by the latent image. It is this reaction that gives 3M Dry Silver its distinct advantage, the elimination of wet or vapor processing.

In order to increase the sensitivity of Dry Silver, knowledge of both the photosensitive and the thermographic aspect of Dry Silver had to be advanced. It is the silver halide on which a latent image can be formed that actually determines the light sensitivity of the material. In order to increase this sensitivity, silver halide crystals which are more prone to photolytic reduction had to be prepared. The choice of halide ion (Cl^- , Br^- , I^-) and the associated cation have been found to be critical for the highest sensitivity. Similarly the environment in which the light sensitive crystal is formed is important. However, increased sensitivity must be transferable to the thermographic reaction. To do this, the light sensitive material had to remain in synergistic association with the image forming source. Mild organic reducing agents capable of promoting the thermographic reaction were found to be most useful.

The increased sensitivity necessary for camera speed Dry Silver film places stricter requirements on the materials that make up the Dry Silver emulsion and the manufacture of camera speed Dry Silver film. Thus in the course of the contract, a continuing effort was directed toward monitoring and improving the materials that are used in the Dry Silver formulation. The coating of the camera speed Dry Silver film was also examined. In both materials and coating ability, improvements were made. The more exact analysis of materials aided in the reproducibility of the camera speed formula. The increased coating of the pansensitive camera speed film enabled the pilot plant coating crew to familiarize themselves with coating in infrared lighting. Once accustomed to infrared coating, the coating crew was able to adapt coating and monitoring techniques to an IR light environment. These techniques enable the coating crew to observe and correct any coating defects while coating.

Progress -- continued

The requirement for dye sensitization in Dry Silver material is that in addition to sensitizing the silver halide grain, the dye must bleach during the development reaction. The 3M Company has extensive background knowledge in this area. The increased sensitivity and broad spectral sensitivity of the contract goal required further refinements in 3M Dry Silver dye technology.

Examples of the refinements in dye sensitizing are shown in Figure 2. The main objective in the area of dye sensitization was to eliminate any spectral insensitivities such as occurred in the previous camera speed Dry Silver film contract final sample at 600nm. The uniform spectral sensitivity from 400 to 700nm did not cause overall sensitivity to decrease and remained at the 2-3 ergs/cm² exposure level to produce a 1.0 density.

The AEI-5 contract material had been used successfully in a test flight of a tactical reconnaissance system. However, areas of heavy exposure tended to flare severely. To investigate methods of reducing flare accutance and antihalation systems were studied. The flare is produced in three areas of the film; scattered light in the emulsion, reflected light at the emulsion-base interface and the base-air interface (Figure 3). Four methods of reducing scattered or reflected light were used; an acutance dye in the emulsion layer to absorb scattered light in the emulsion, an antihalation underlayer (AHU) at the emulsion-base interface to absorb reflected light, an antihalation backside coating (AHB) at the air-base interface to absorb reflected light, and a dye base to absorb light. The acutance and antihalation systems improved resolution. With the acutance systems, sensitivity is reduced. The nature of this contract was to improve the sensitivity of Dry Silver. Therefore, the acutance and antihalation systems were shown to be feasible. With further work, these could be incorporated into a camera speed Dry Silver system. Commercial products have used these systems successfully.

During the course of the contract hundreds of emulsions were made to examine the effects of different independent variables. It is known that the physical and chemical environment during halidization have direct effect on sensitivity. By changing certain variables, a 1.4X increase in speed was observed (Figure 4). Since AEI by definition is a function of gamma and toe shape, the sensitivity of a film can be increased by extending the toe of the film. Manipulation of the toe shape, however, is not an appropriate way to achieve contract goals. With a given film, over-processing tends to extend the toe, as can be seen later in a discussion of gamma. A disadvantage of this method is that Dmin and Dmax are sacrificed. Another,

Progress - continued

more direct method is to change the chemistry of the film. Figure 5 shows a faster film, which was achieved by improving the thermographic sensitivity of the most light sensitive grains in the emulsion. In this case, gamma was changed significantly.

By properly formulating the Dry Silver emulsion, camera speed films of AEI 1.6-12.0 were obtained in the laboratory. These films are not sufficiently sensitive to meet contract goals. These films, which are negative working, panchromatic, and photosensitive are unique in that the latent image is amplified by the simple application of heat to give a usable photographic image with desired properties.

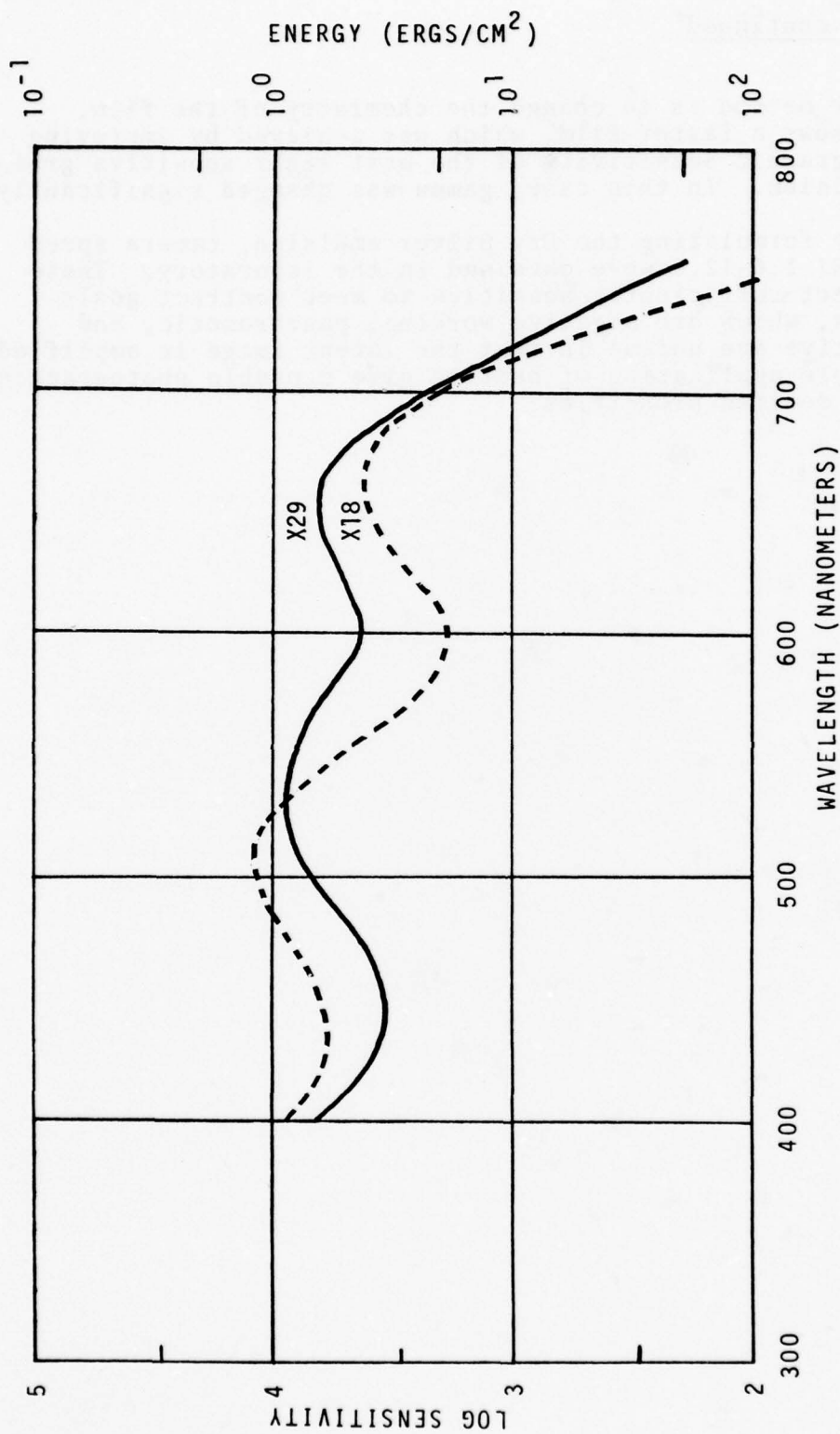


FIGURE 2 - Improved Dye Sensitization of Camera Speed Dry Silver Film

Sensitivity = reciprocal of the exposure at point of no reciprocity law failure in millijoules/cm² required to produce a gross density of 1.0. The curves represent X18 precontract sample, and X29 final contract dye sensitization.

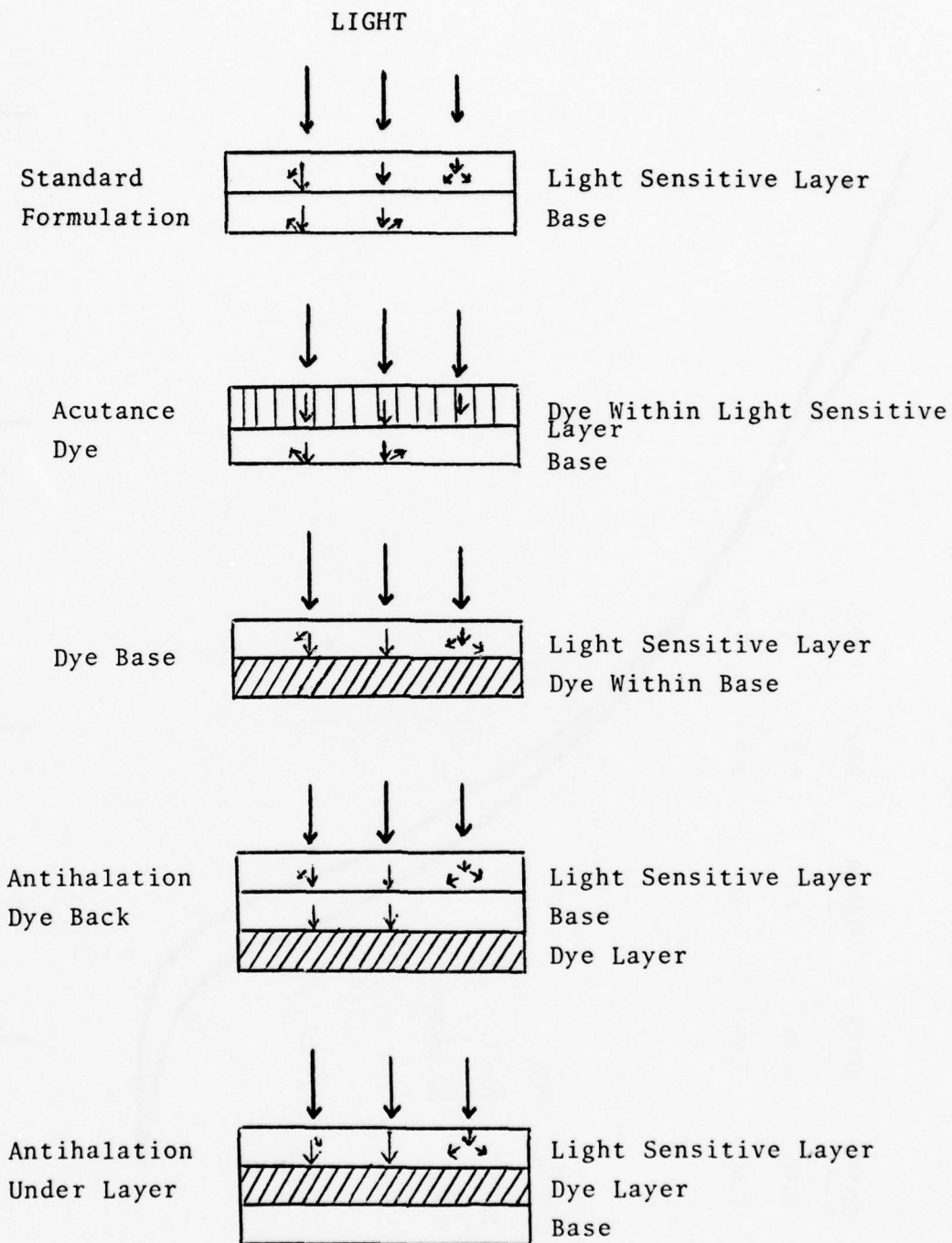


FIGURE 3 - Acutance and Antihalation Systems Used to Reduce Flare in Camera Speed Dry Silver Film

CAMERA SPEED DRY SILVER FILM

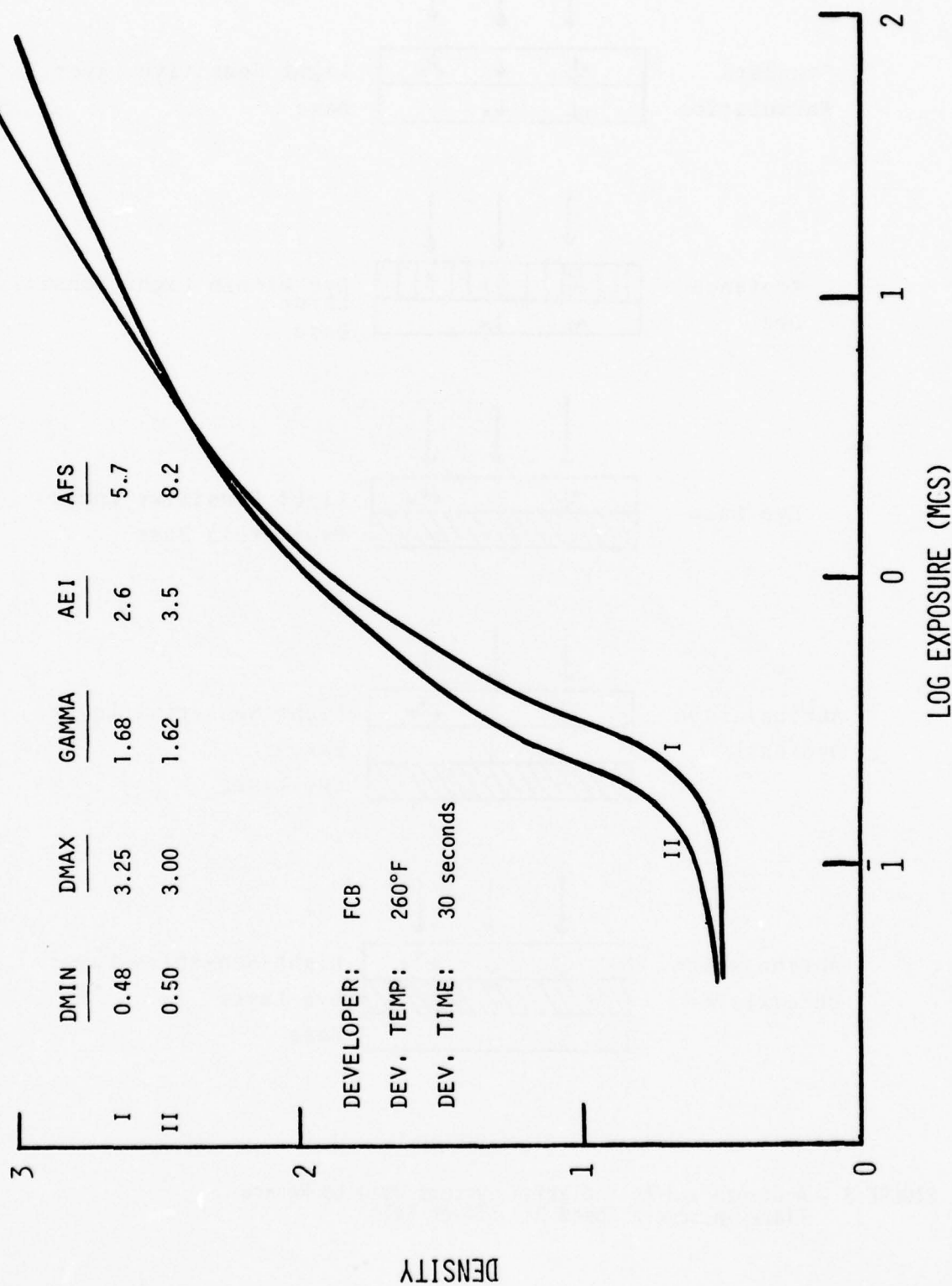


FIGURE 4 - Effect of Physical and Chemical Halidization Parameters on Sensitivity

CAMERA SPEED DRY SILVER FILM

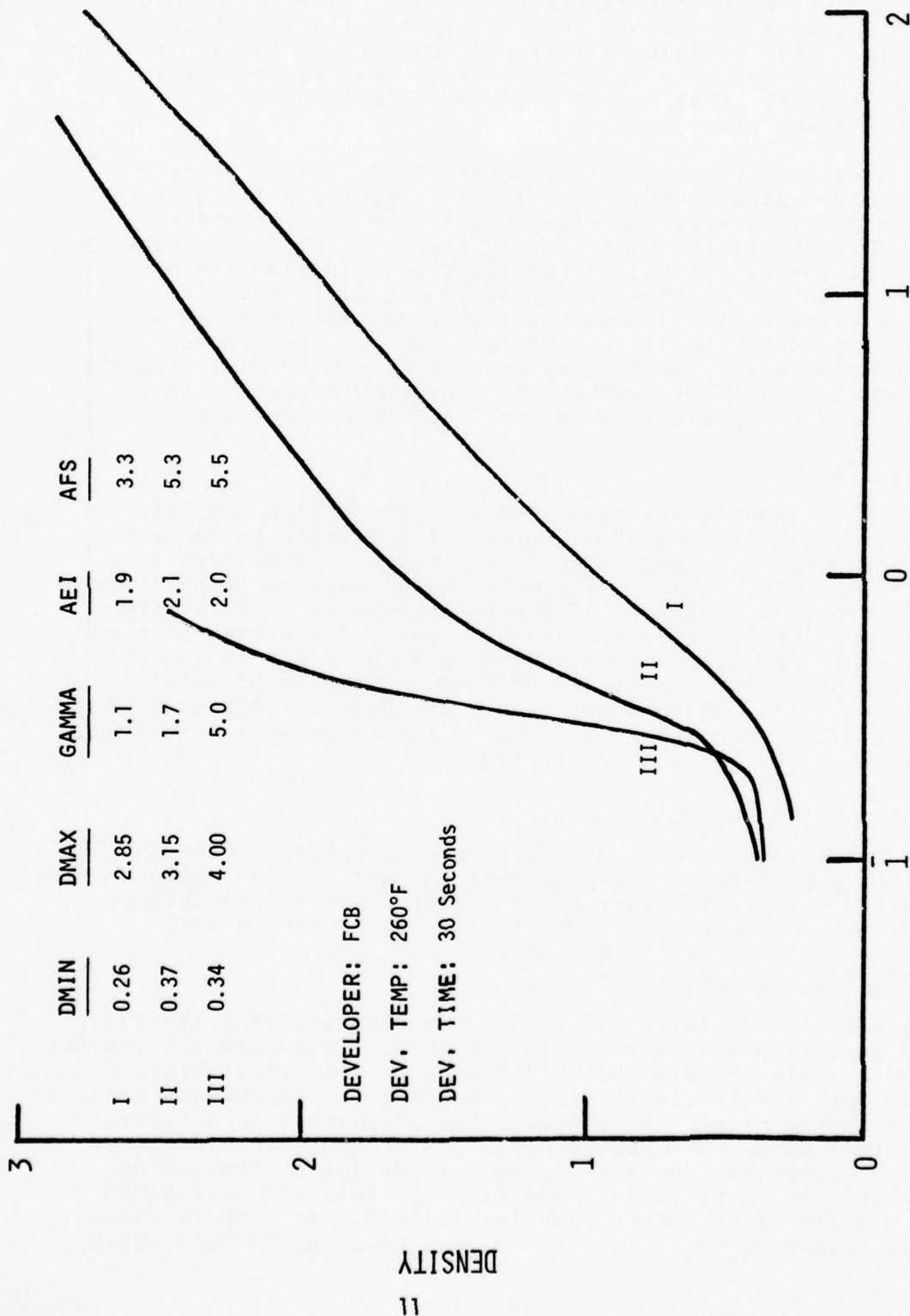


FIGURE 5 - The Effect of Developers on the Sensitivity and Gamma of Dry Silver. (The light sensitive component is the same for the three samples while the developer/toner system is varied.)

SECTION IV

RESULTS

The discussion of the contract's results will be divided into areas of laboratory results and the final contract sample. This division allows a discussion of the sensitivity achieved at a laboratory scale besides the level of sensitivity that can be produced at a larger scale with a pilot plant coating.

All sensitometric exposures, unless specifically notated, were made using an EK101 sensitometer having a 0-4 neutral density carbon wedge, modified to include a series 335 ATC-Shawnee digital timer for shutter control. A Corning 5900 filter was placed in the light path to simulate daylight. Output of this system was measured at the film plane using a United Detector Technology 21A power meter equipped with a NO 242 radiometric sensor. Development of samples was by mechanical immersion in a 3M FC-43 fluorochemical bath (FCB), continuous thermal processor with a 3M Model 259, or development in a 3M Model 199 sheet processor.

Laboratory

With the added sensitivity necessary for AEI-20 Dry Silver material, the interactions between the formula parameters become more important. Therefore, certain laboratory formulations with either critical chemical ratios, temperatures, agitation, or equipment were not suitable to produce film in the pilot plant coater at this time. Additional testing and equipment fabrication are being done to obtain sensitivity by the previously mentioned methods. Examples of sensitivity obtainable at a laboratory scale are shown in Figures 6 and 7. These films were very sensitive to their chemical ratios and the method of formula preparation.

Final Contract Sample

The final sample for the Camera Speed Dry Silver Film AEI-20 contract represented the state-of-the-art of pilot plant coating. This film represents a reproducible formulation that is of camera speed sensitivity, at the AEI=1.6 level.

Spectral Sensitivity

The film is sensitive through 700 nanometers. The absolute spectral response is shown in Figure 8. This data was generated with a series of blocked narrow band pass filters obtained from Optics Technology, Inc. Sensitometric output was measured at the film plane. Development was 30 seconds in a fluorochemical bath. The energy necessary to generate a density of 0.3 above fog and a gross density of 1.0 at the various wavelengths is given in Table 1. This data was determined at a point of no reciprocity law failure. As seen in Table 1 or Figure 8, the film met the goal of a panchromatic film.

Results - continued

Photosensitivity

The measurement of sensitivity for the contract was the Aerial Exposure Index, AEI, of the film. AEI is defined as $1/2E$ where E is the exposure in metercandle seconds to a 5900-6100 Kelvin light source required to produce a density response of 0.6 slope of the measured gamma. The goal of the contract was an AEI of 20. Sensitivity was also measured in terms of Aerial Film Speed AFS. AFS is defined as $3/2E$ where E is the exposure in meter candle seconds to a 5900 to 6100 Kelvin light source at a density response at 0.30 above base fog.

As determined at 3M, the film, when initially coated, had an AEI of 1.6 and an AFS of 3.4 with 30 second development in a fluoro-chemical bath. An optimum characteristic curve for a daylight source is shown in Figure 9. An optimum characteristic curve for a tungsten light source is shown in Figure 10.

The sensitivity is dependent on exposure time as shown in Figure 11 reciprocity curves. Measurements for this involved a series of exposures from 99.99 seconds to 10^{-5} seconds with a known output. Measurements at times greater than 0.20 seconds were done on an EK101 sensitometer. Measurements done at times less than 0.20 seconds were done on an EG&G Mark VII. Exposure times for the EG&G Mark VII were determined by oscilloscope calibration. Optimum exposure time was found to be 10^{-3} seconds (Figure 9).

Gamma

The gamma of Dry Silver film is essentially formula dependent. There exists a small range in variation of gamma with varying development. However, other characteristics of the film are also changed such as D_{min} , D_{max} , and general shape of the characteristic curve. The gamma, contrast response of the film, is from 1.2 to 2.1 and the bar gamma is from 1.1 to 1.9 as shown in Figure 12. This is within the contract goals of a gamma of 1.2 to 1.6.

Image Color

The image produced upon development is a neutral black image both on reflectance and transmittance.

Useful Log Exposure Range

The useful log exposure range was determined with a 100 mcs exposure similar to Figure 9. With this exposure the useful log exposure range is 2.6 log E units. However, as shown in Figure 13, the true D_{max} of the film is 4.20 and, therefore, the log exposure range is greater than 2.6 log E units.

Results - continued

Image Resolution

All resolution was measured by contact imaging of a MIL-STD-150A test target pattern; high contrast (> 2.0 density difference), medium contrast (0.8 density difference), and low contrast (0.22 density difference.) Maximum resolution found was:

203 cycles/mm high contrast
128 cycles/mm medium contrast
32 cycles/mm low contrast

Resolution versus exposure figures, Figures 14, 15, and 16 for high, medium, and low contrast respectively, show the exposure ranges for optimum resolution. Resolution meets the goal of 200 cycles/mm high contrast.

Film Base and Coating Thickness

The film is coated on clear polyester film base nominally 3 mil thickness. The photosensitive coating thickness is 1 mil. There is no backside coating. Thus the film thickness is a nominal 4 mil.

Film Processing

The film is processed by heat. The mode of heating is unimportant. However, optimal development time and temperature will depend on the equipment used. The goal of the contract was thermal development to a stable image form in 30 seconds or less time at temperatures not exceeding 300F. The film was tested in a 3M FC-43 fluorochemical bath, a 3M Model 259 roll film processor, and a 3M Model 199 sheet processor.

Development temperatures for the fluorochemical bath were chosen to encompass a use range. The test temperatures were 250F, 260F, and 270F. Development series for 250F, 260F, and 270F are given in Figures 17, 18, and 19 respectively.

Optimum curves using Dmin and speed as the criteria for these three temperatures are given in Figure 20. The fluorochemical bath temperature for the other tests of this report was 260F. The choice was made solely for convenience since this temperature is common to other Dry Silver film products. From Figure 20 it can be seen that increasing development temperature can reduce processing time.

Results - continued

The 3M Model 199 sheet processor can also be used to develop the film. The nature of this processor is such that longer development times are required. A development series was generated for 250F, Figure 21, and 260F, Figure 22. Optimum development time or temperature is dependent on the sensitometric parameters desired.

Roll film was processed on the 3M Model 259 roll processor. The optimum conditions for development were higher temperatures with shorter dwell times. Figures 23 through 27 show development series for temperature settings of 265F, 270F, 275F, 280F, and 290 F respectively. A recommended setting for optimum development is 290 F at a development time of 15 feet/minute. A comparison of characteristic curves in Table 2 and shown in Figure 28, which is the optimum development times for five temperatures, demonstrates the optimum condition of 290 F. The development latitude on a Model 259 processor is shown in Figure 29.

The goal for film processing was 30 seconds. This time is easily met since a dwell time for 15 feet/minute development is only ten seconds on the Model 259. The other methods of developing individual samples requires times greater than 30 seconds. However, since this material is a roll material, the important development conditions are roll development conditions which are well within the goals of the contract.

Latent Image Stability

To determine latent image of the film, samples were exposed and stored in black envelopes at 70F and 50% relative humidity. Samples were developed at predetermined times over a 24 hour period at the same development conditions. A sensitivity loss of 0.3 log E units occurred in four hours. The loss over 24 hours is shown in Figure 30. The latent image decay was decreased with a decrease in temperature.

Image Permanency

Exposed and processed film was subjected to two light intensities; room light and Richard's Photointerpretation Light Table. Room light conditions were 100 footcandles and 72F with normal room humidity. The Richard's Light Table was operated at the maximum output which was 1400 footcandles and 100F on the surface of the table. Figures 31 and 32 show the increase of density when exposed to light. As shown in the figures, there is an increase in Dmin of the film, however, there is no degradation of the image. The image color remains a neutral black while the unimaged regions progress to a brown with a density increase of 0.40 when subjected to the Richard's Light Table for one hour. The sample has no Dmin increase when subjected to the same heat conditions without light.

Results - continued

Useful Package Shelf Life

Shelf life was tested at three combinations of temperature and humidity. The three conditions are defined as 70F/50% RH, 80F/80% RH, and 120F/50% RH. All samples were stored in black envelopes which allowed the film to equilibrate to the test condition. The results are shown in Figures 33, 34, and 35 for 70F/50% RH, 80F/80% RH, and 120F/50% RH respectively. As shown in these figures, a speed loss is evident. This speed loss is directly related to the temperature. When the film is stored at 14F (-10C) or below, the shelf life is indefinite at the present state of the testing.

CAMERA SPEED DRY SILVER FILM

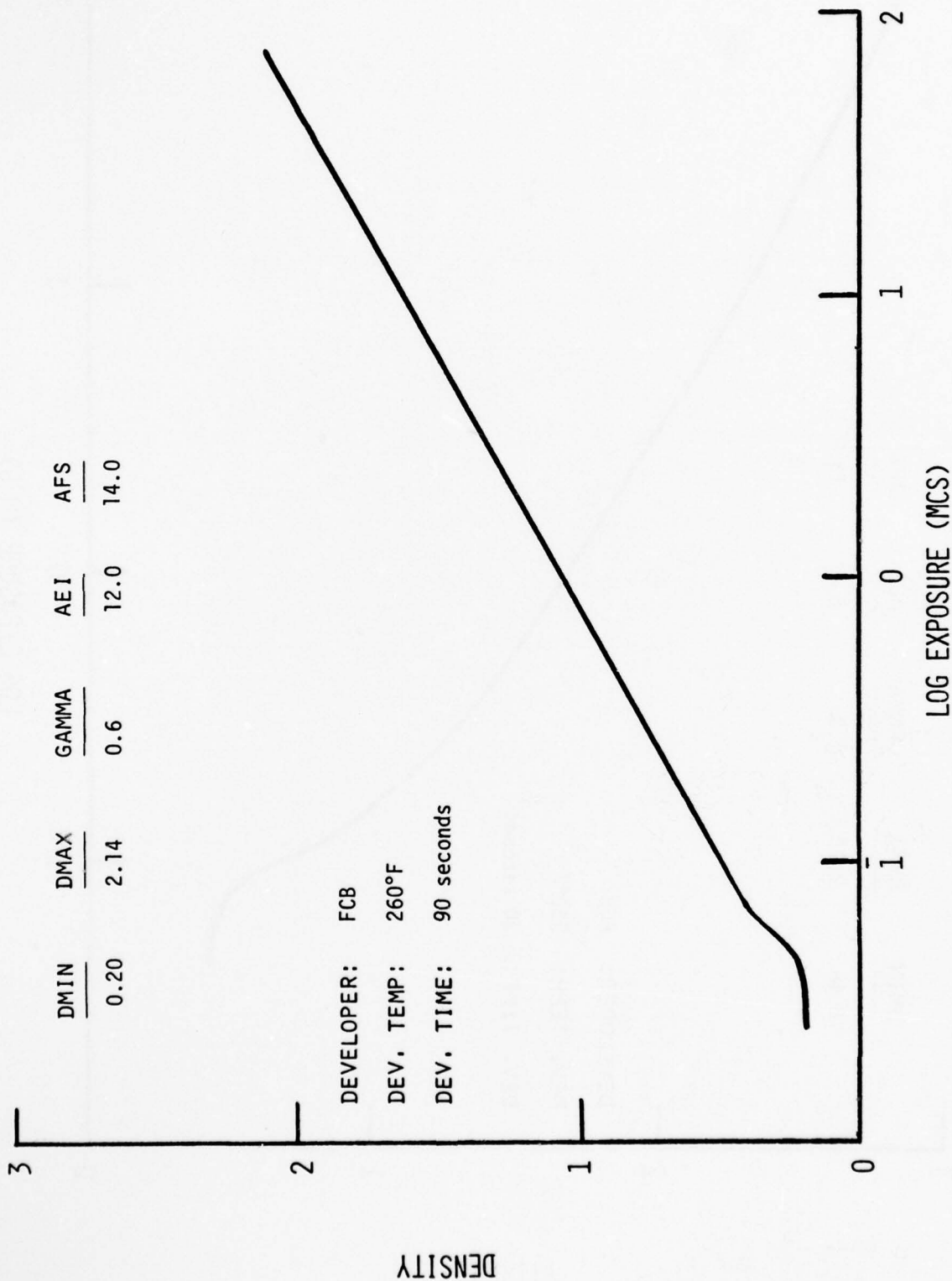


FIGURE 6 - Highest Photosensitive Lab Coating with Extended Thermal Development

CAMERA SPEED DRY SILVER FILM

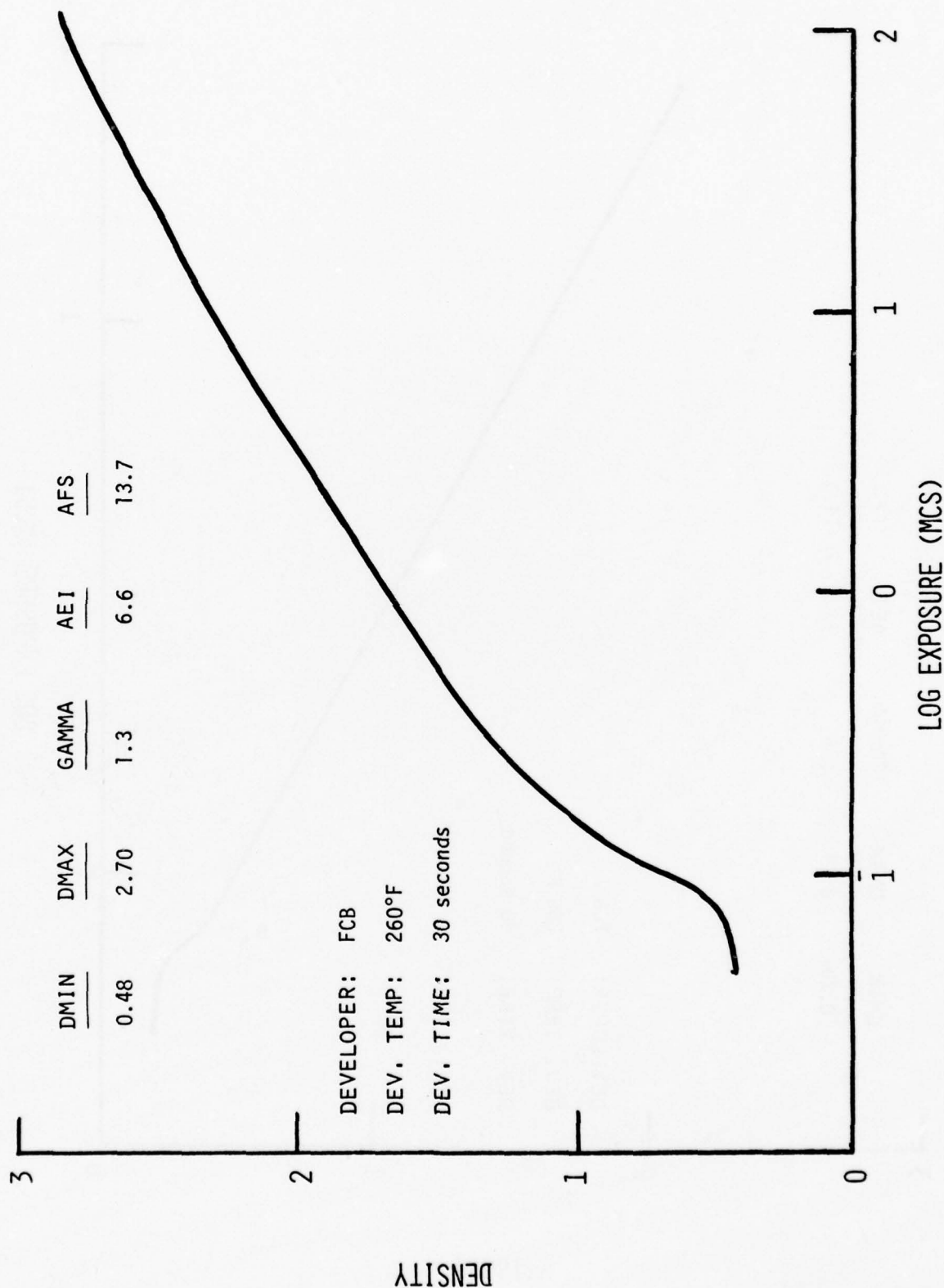


FIGURE 7 - Highest Photosensitive Lab Coating with Standard Thermal Development

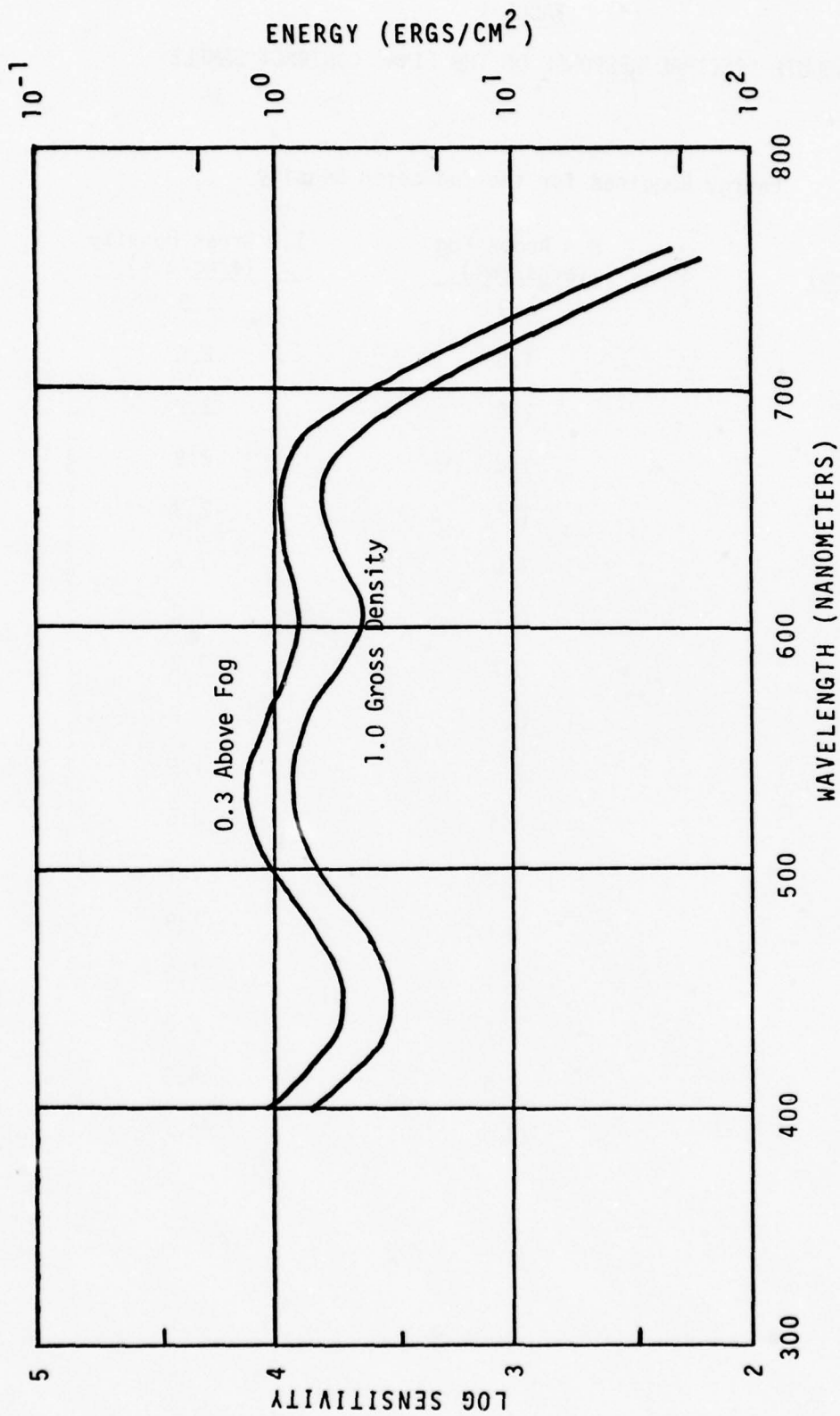


FIGURE 8 - Absolute Spectral Response of the Final Contract Sample
Sensitivity = reciprocal of the exposure at point of no reciprocity law failure in millijoules/cm² required to produce the indicated density (D).

TABLE 1

ABSOLUTE SPECTRAL RESPONSE OF THE FINAL CONTRACT SAMPLE

Energy Required for the Indicated Density

<u>Wavelength (nm)</u>	<u>0.3 Above Fog (ergs/cm²)</u>	<u>1.0 Gross Density (ergs/cm²)</u>
400	0.9	1.5
420	1.5	2.6
440	2.0	3.2
460	1.8	2.9
480	1.4	2.3
500	1.0	1.6
520	0.7	1.2
540	0.7	1.3
560	0.9	1.2
580	1.1	1.9
600	1.4	2.5
620	1.3	2.1
640	1.1	1.9
660	1.1	1.6
680	1.2	2.0
700	2.6	4.3
750	27.5	45.7

CAMERA SPEED DRY SILVER FILM

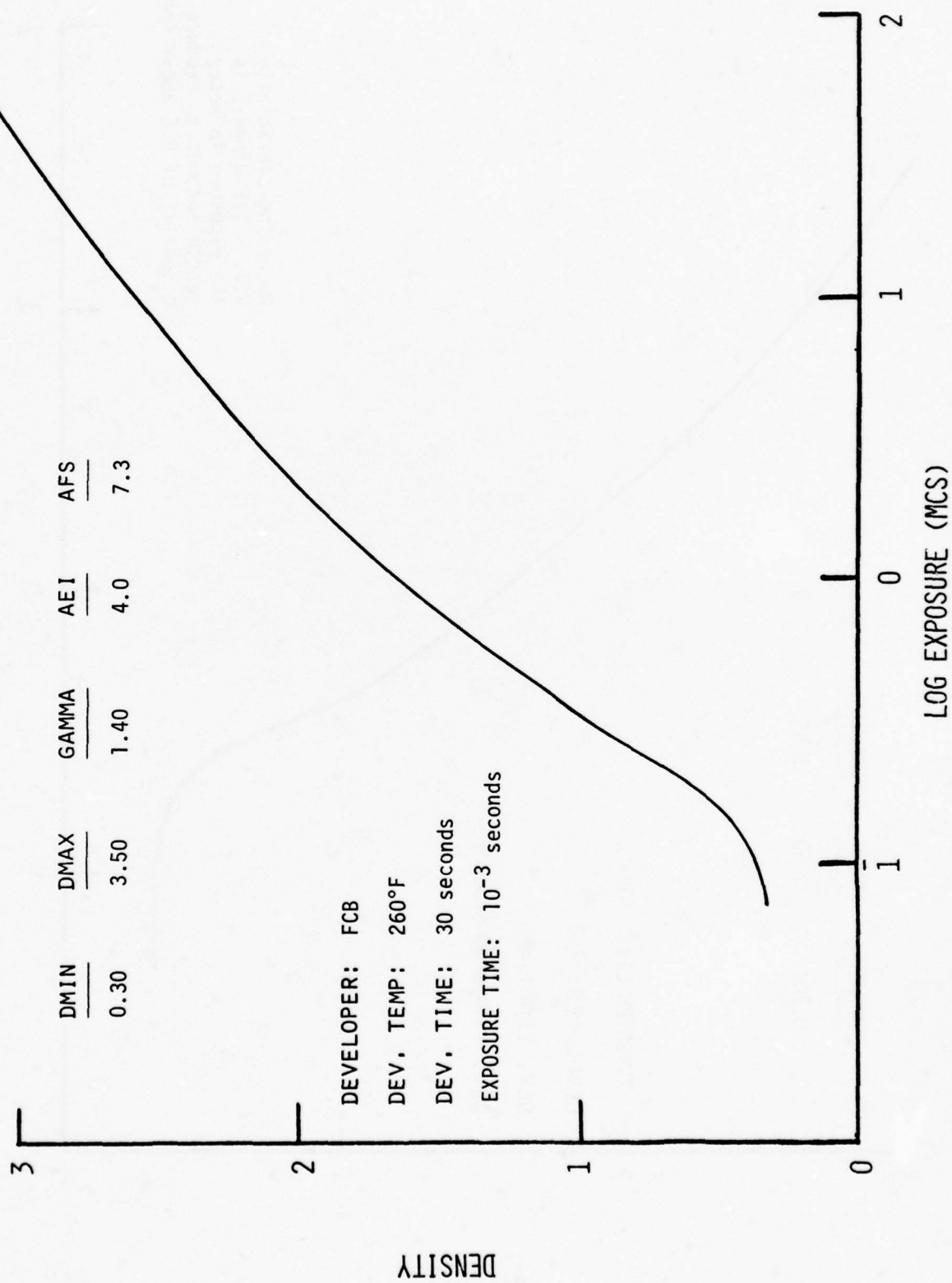


FIGURE 9 - Optimum Characteristic Curve for the Final Sample, Daylight Light Source.

CAMERA SPEED DRY SILVER FILM

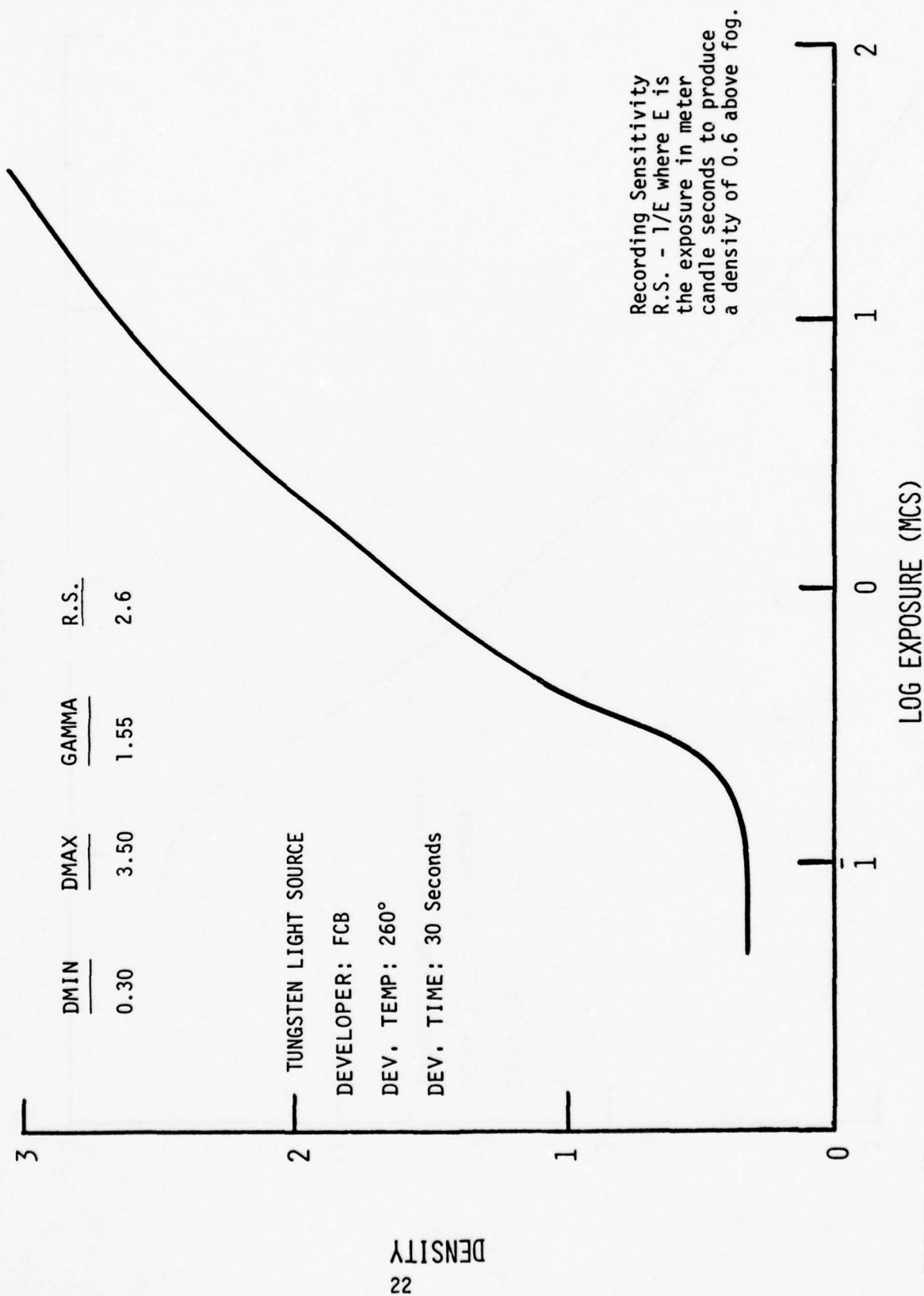


FIGURE 10 - Optimum Characteristic Curve for the Final Sample, Tungsten Light Source

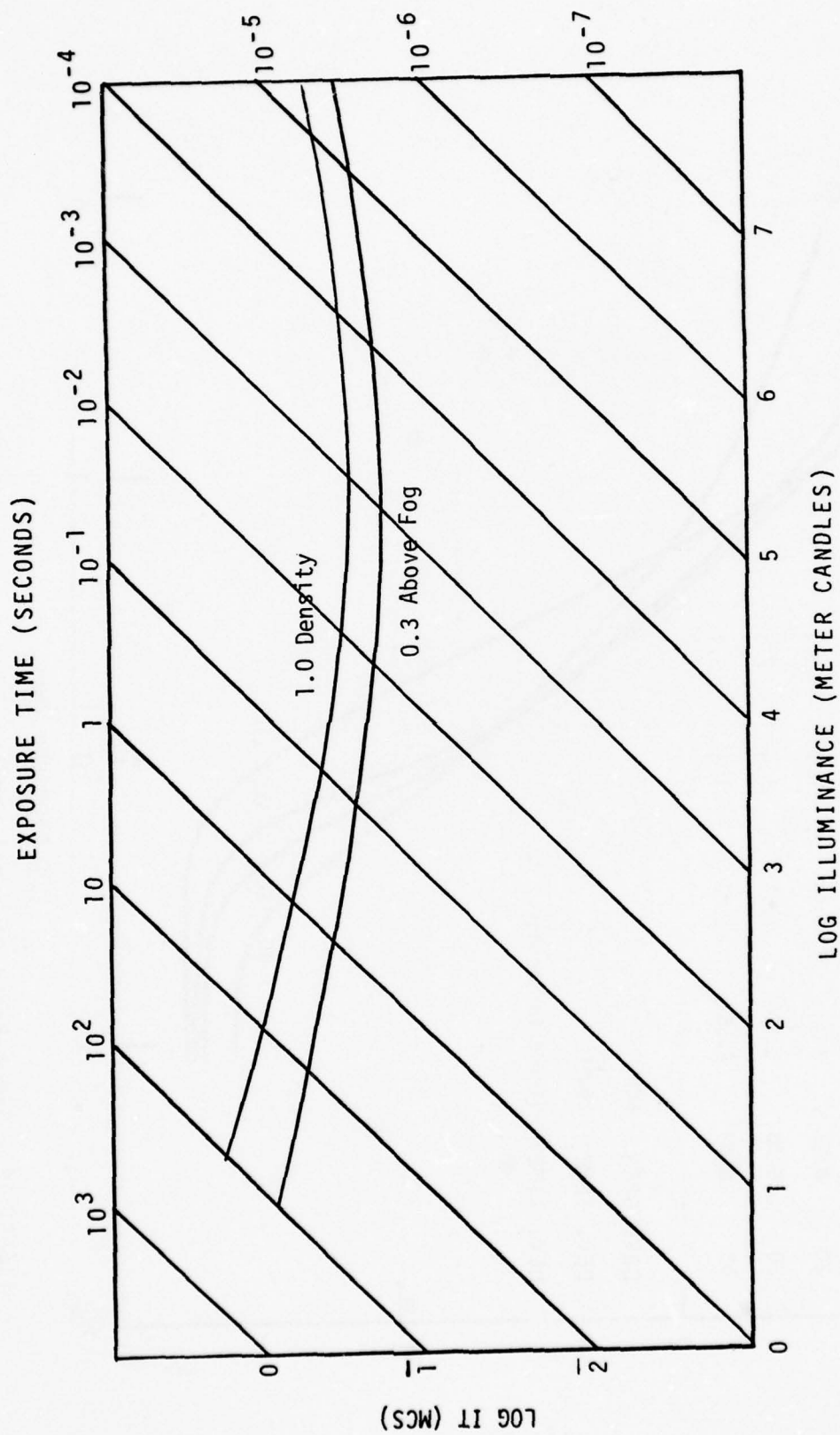


FIGURE 11 - Reciprocity Curves for the Final Sample.

CAMERA SPEED DRY SILVER FILM

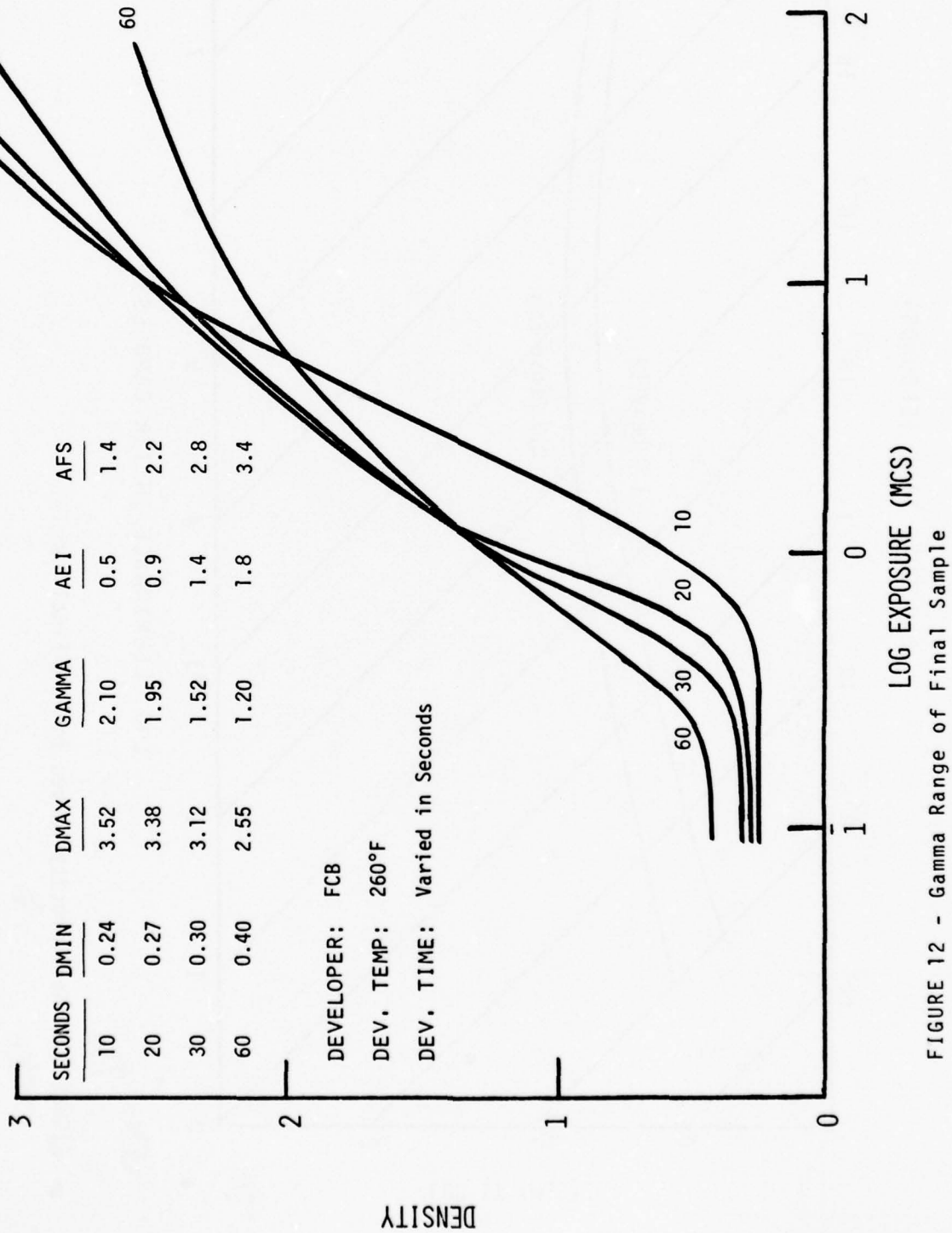


FIGURE 12 - Gamma Range of Final Sample

CAMERA SPEED DRY SILVER FILM

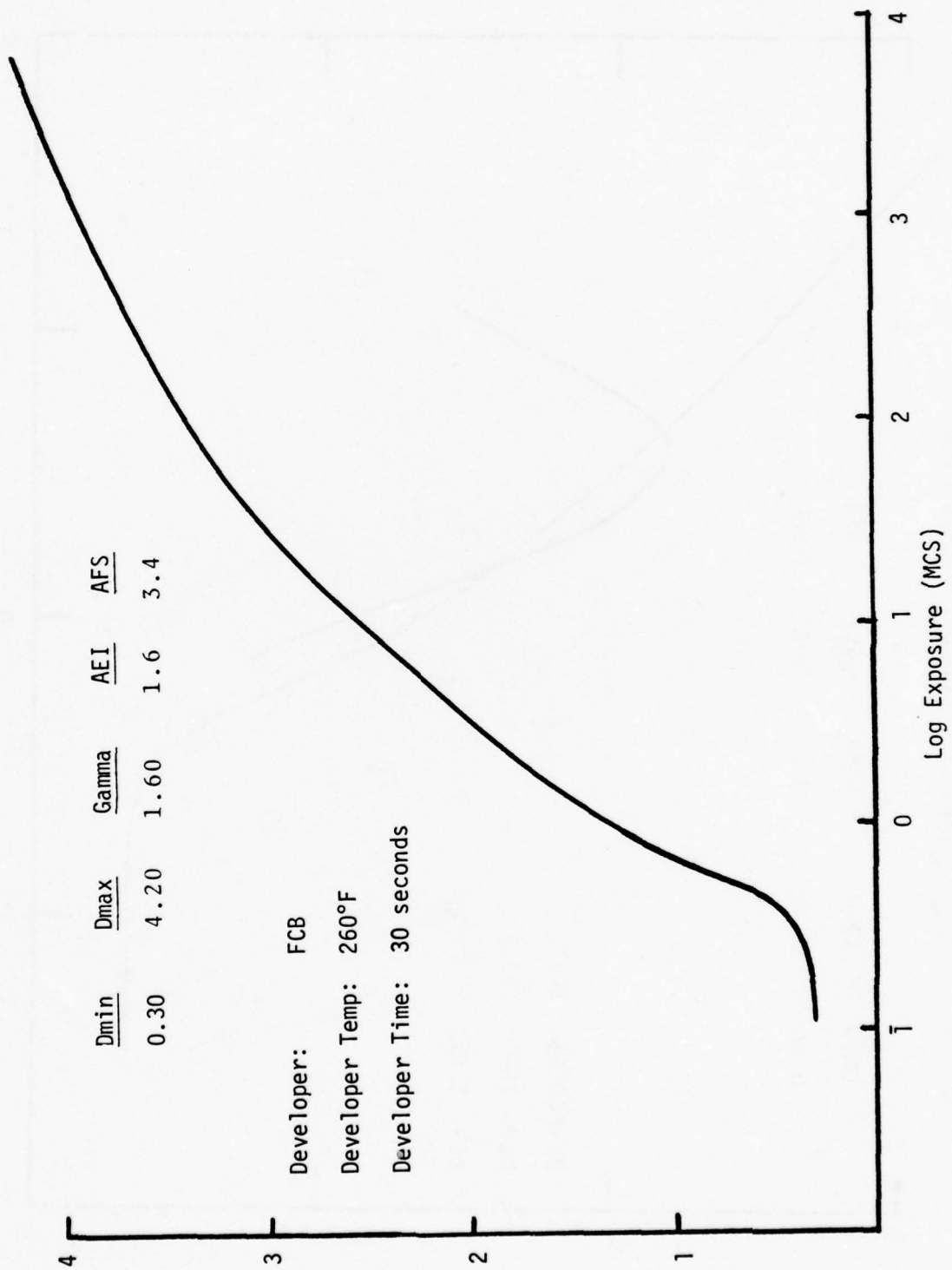


FIGURE 13 - Exposure Range to Reach Dmax for the Final Sample

CAMERA SPEED DRY SILVER FILM

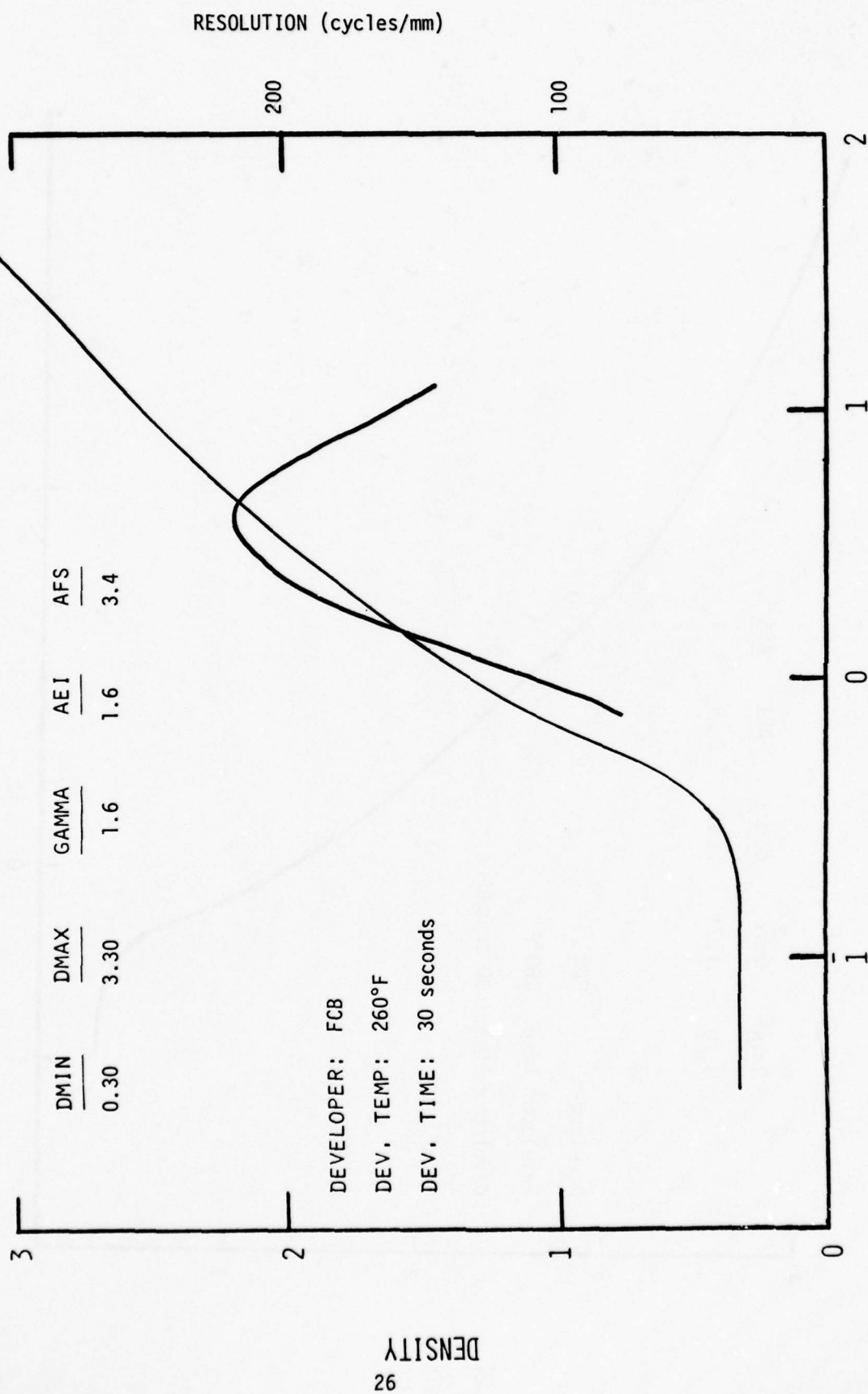


FIGURE 14 - High Contrast Contact Resolution versus Exposure

CAMERA SPEED DRY SILVER FILM

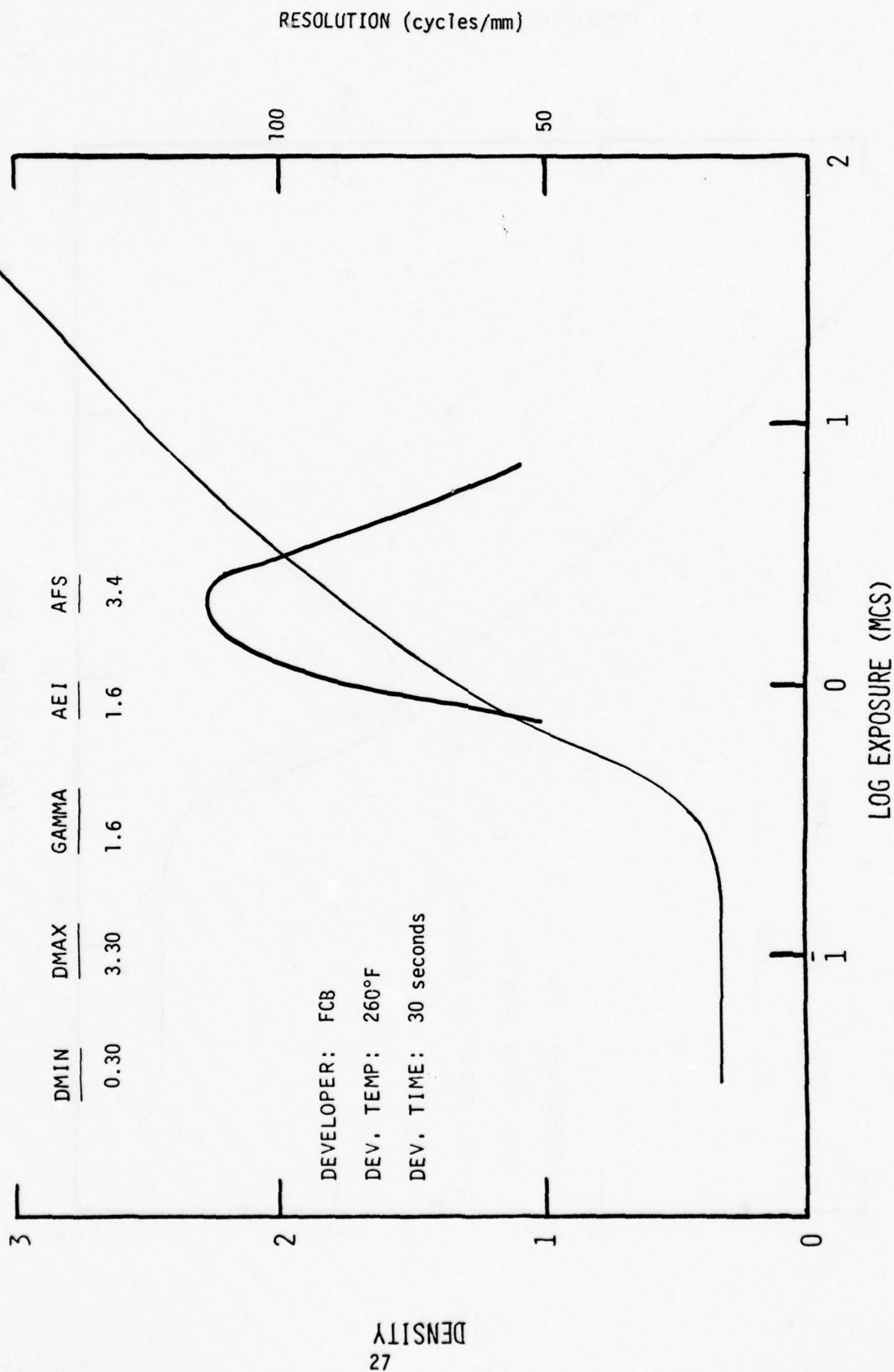


FIGURE 15 - Medium Contrast Contact Resolution versus Exposure

CAMERA SPEED DRY SILVER FILM

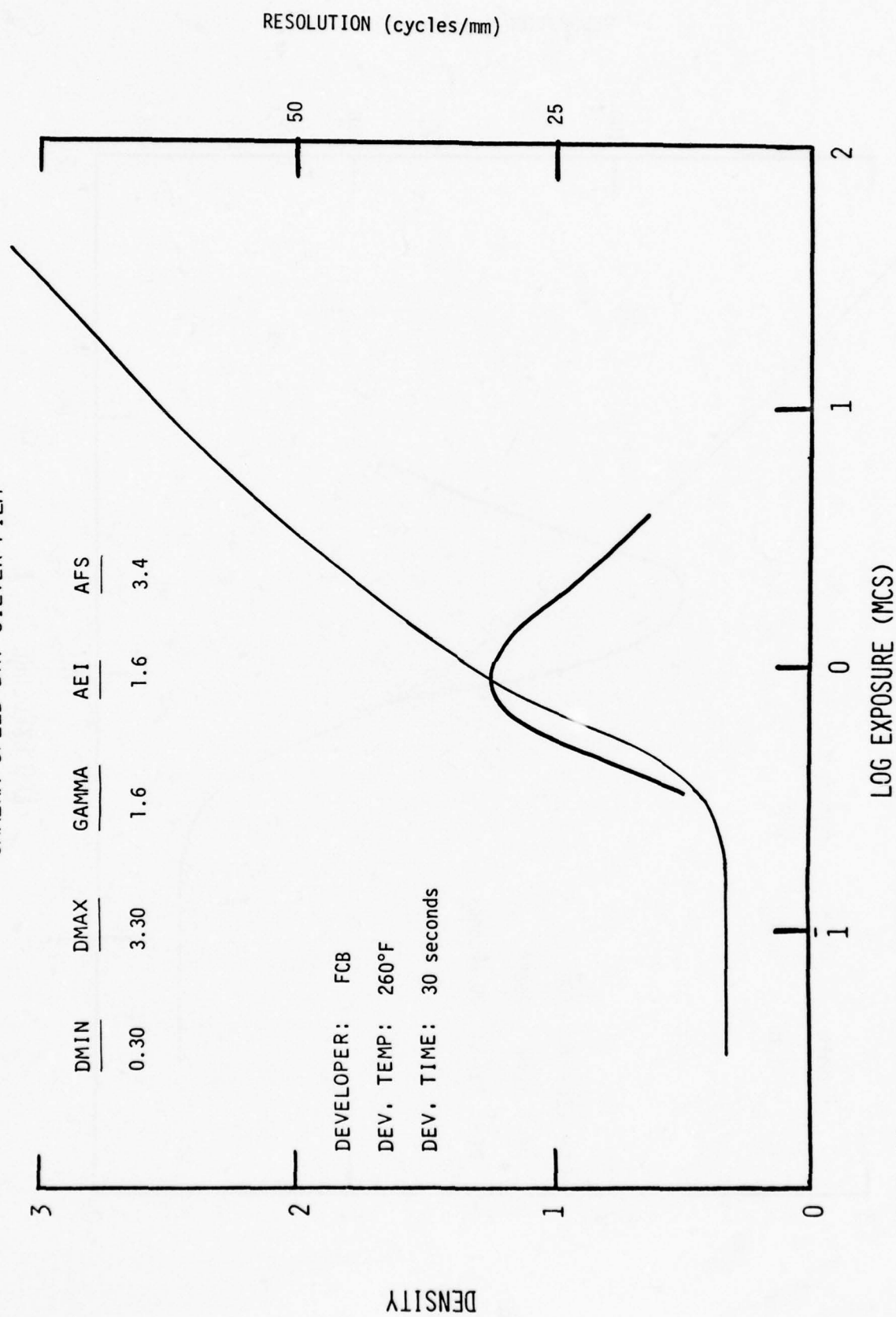


FIGURE 16 - Low Contrast Contact Resolution versus Exposure

CAMERA SPEED DRY SILVER FILM

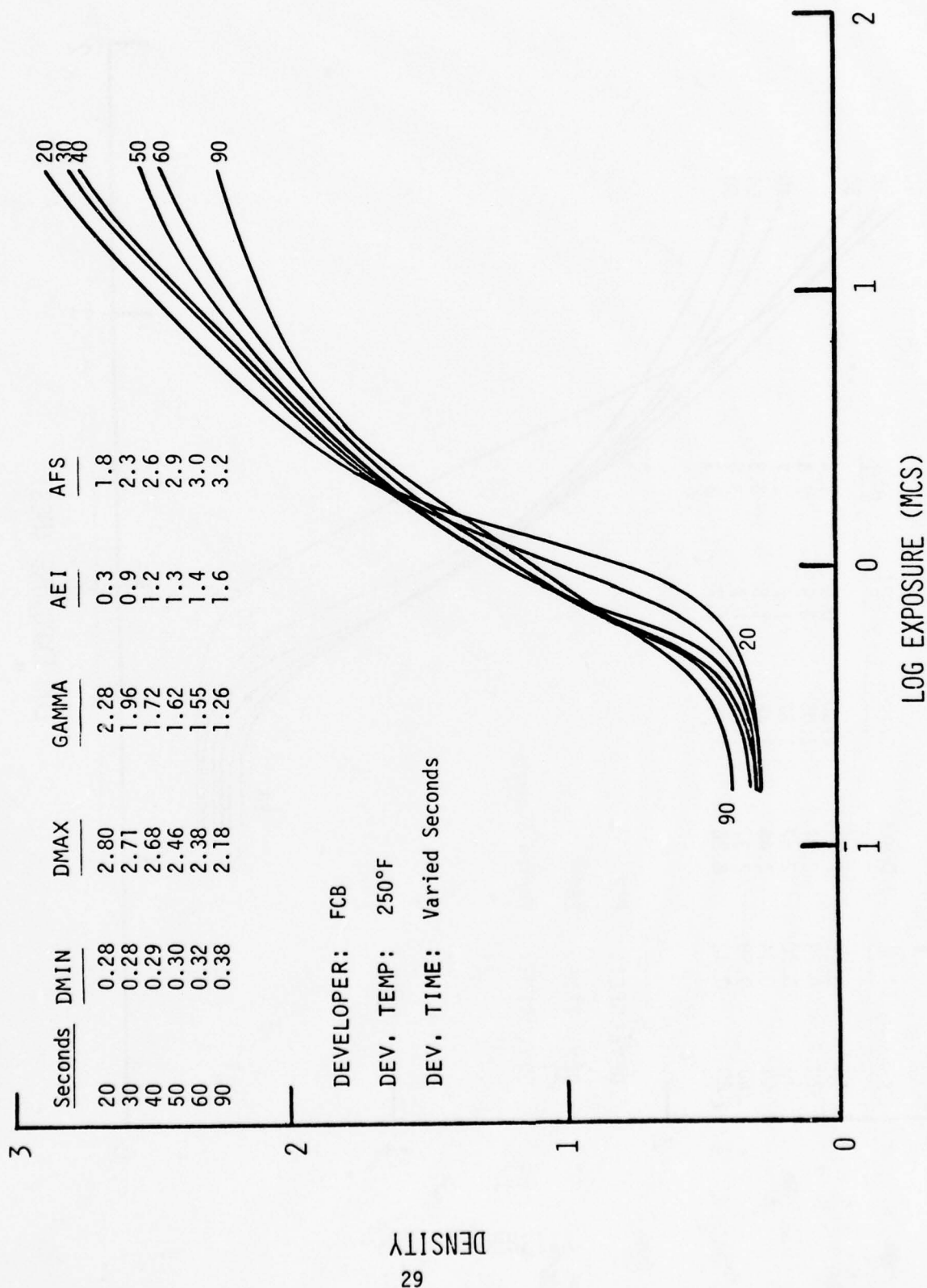


FIGURE 17 - Development Series at 250°F in a Fluorochemical Bath for the Final Sample

CAMERA SPEED DRY SILVER FILM

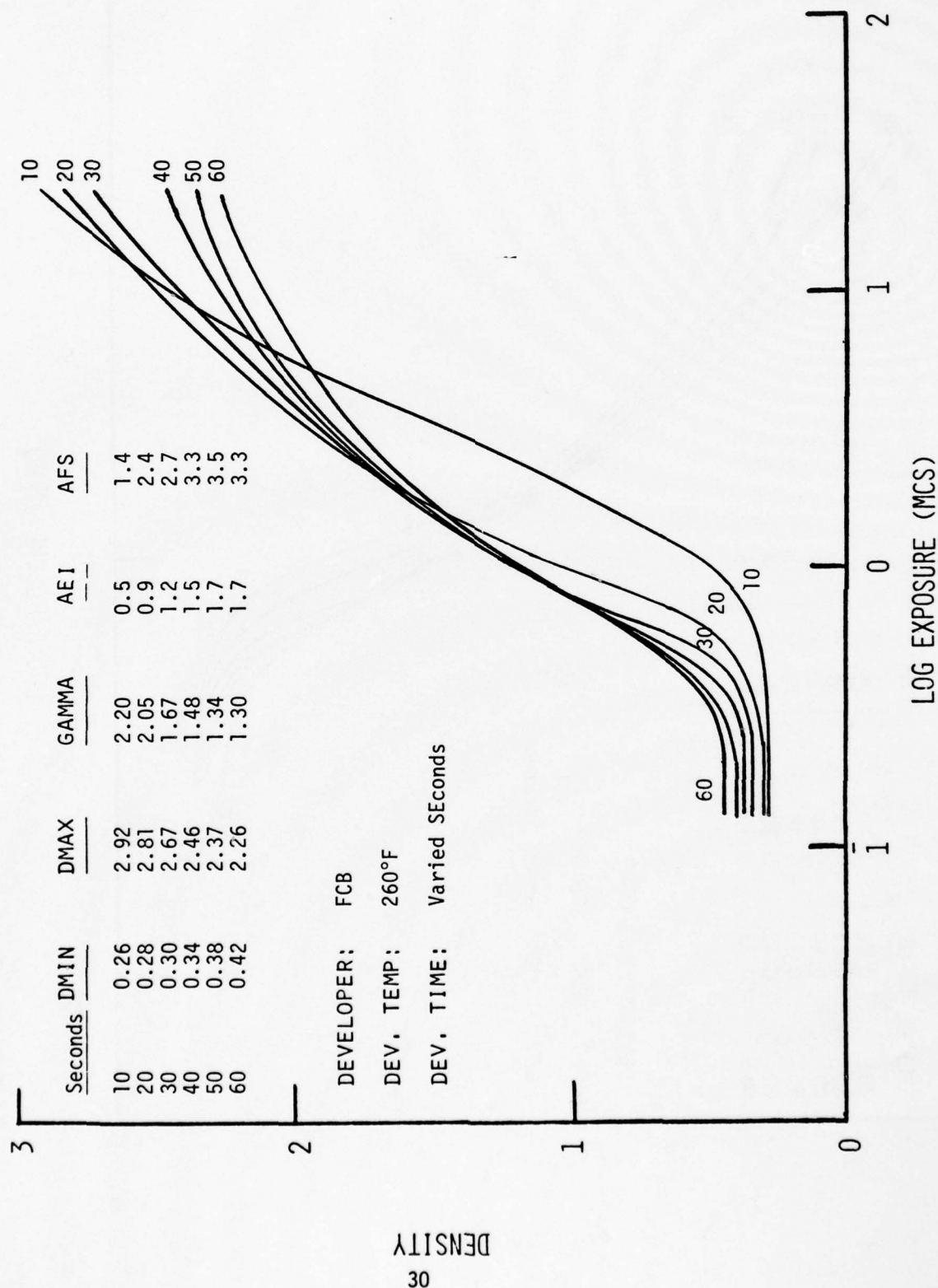


FIGURE 18 - Development Series at 260°F in Fluorochemical Bath for the Final Sample

CAMERA SPEED DRY SILVER FILM

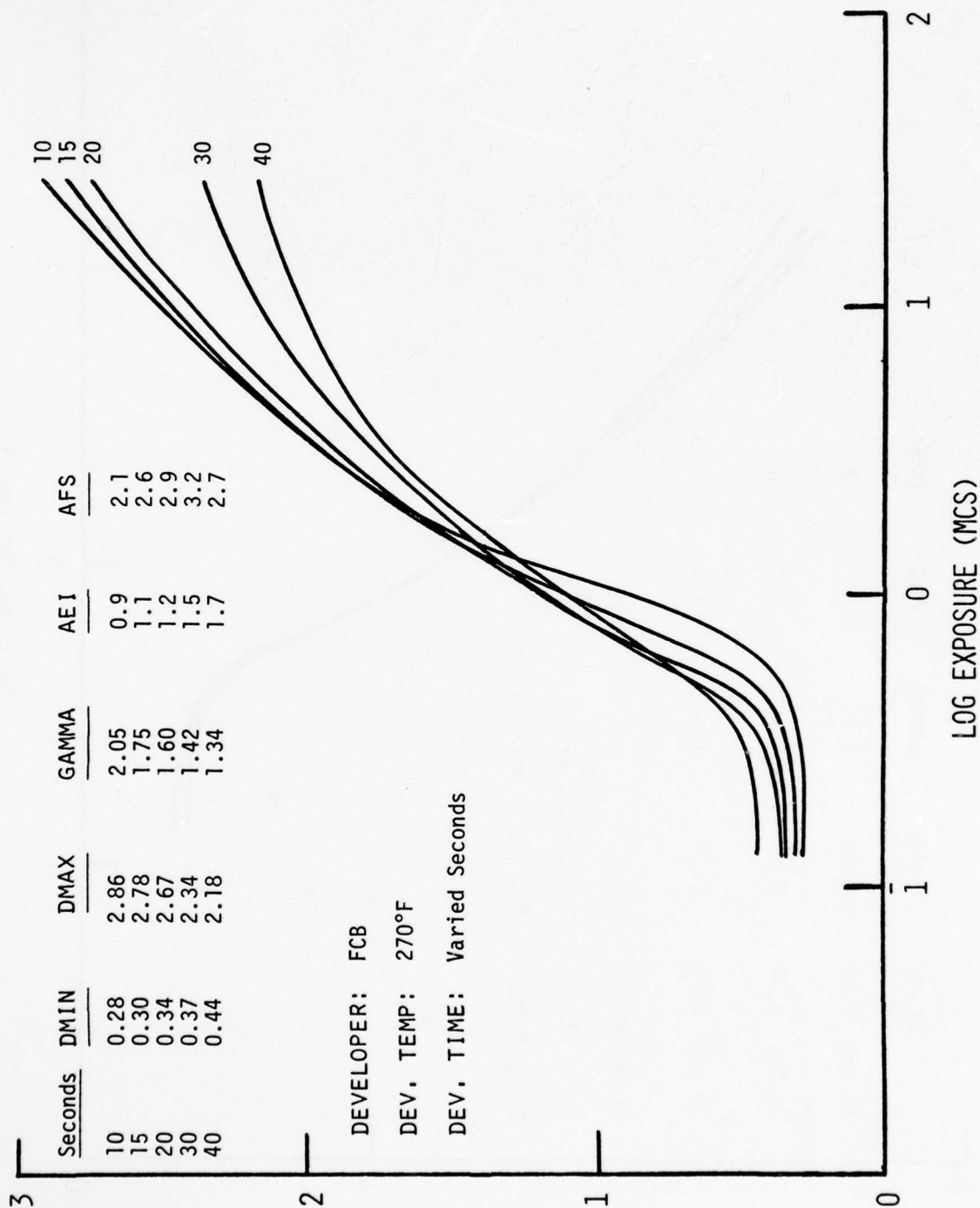
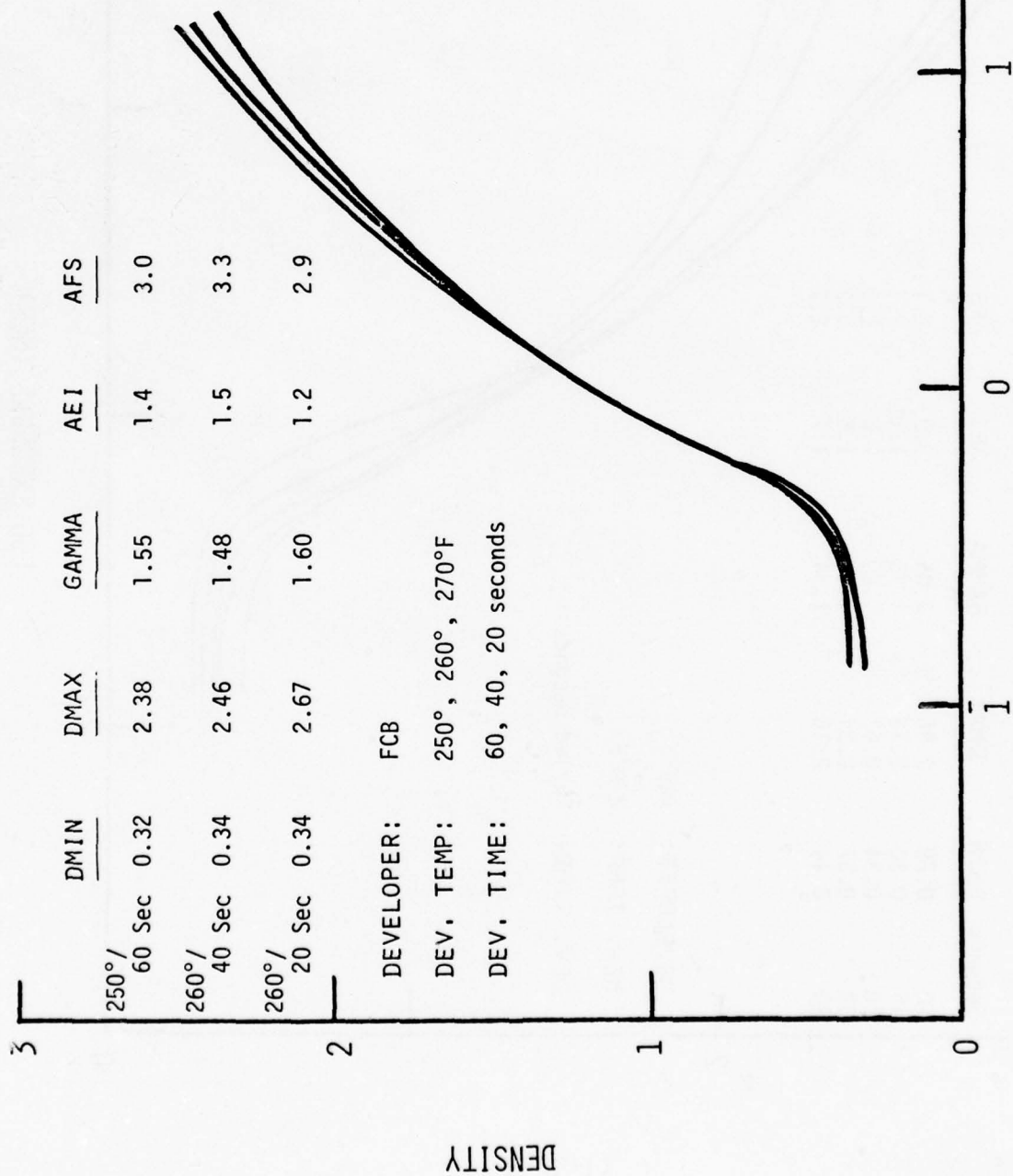


FIGURE 19 - Development Series at 270°F in a Fluorochemical Bath

CAMERA SPEED DRY SILVER FILM



LOG EXPOSURE (MCS)

FIGURE 20 - Optimum Development in Fluorochemical Bath (250°F, 260°F, and 270°F) for the Final Sample

CAMERA SPEED DRY SILVER FILM

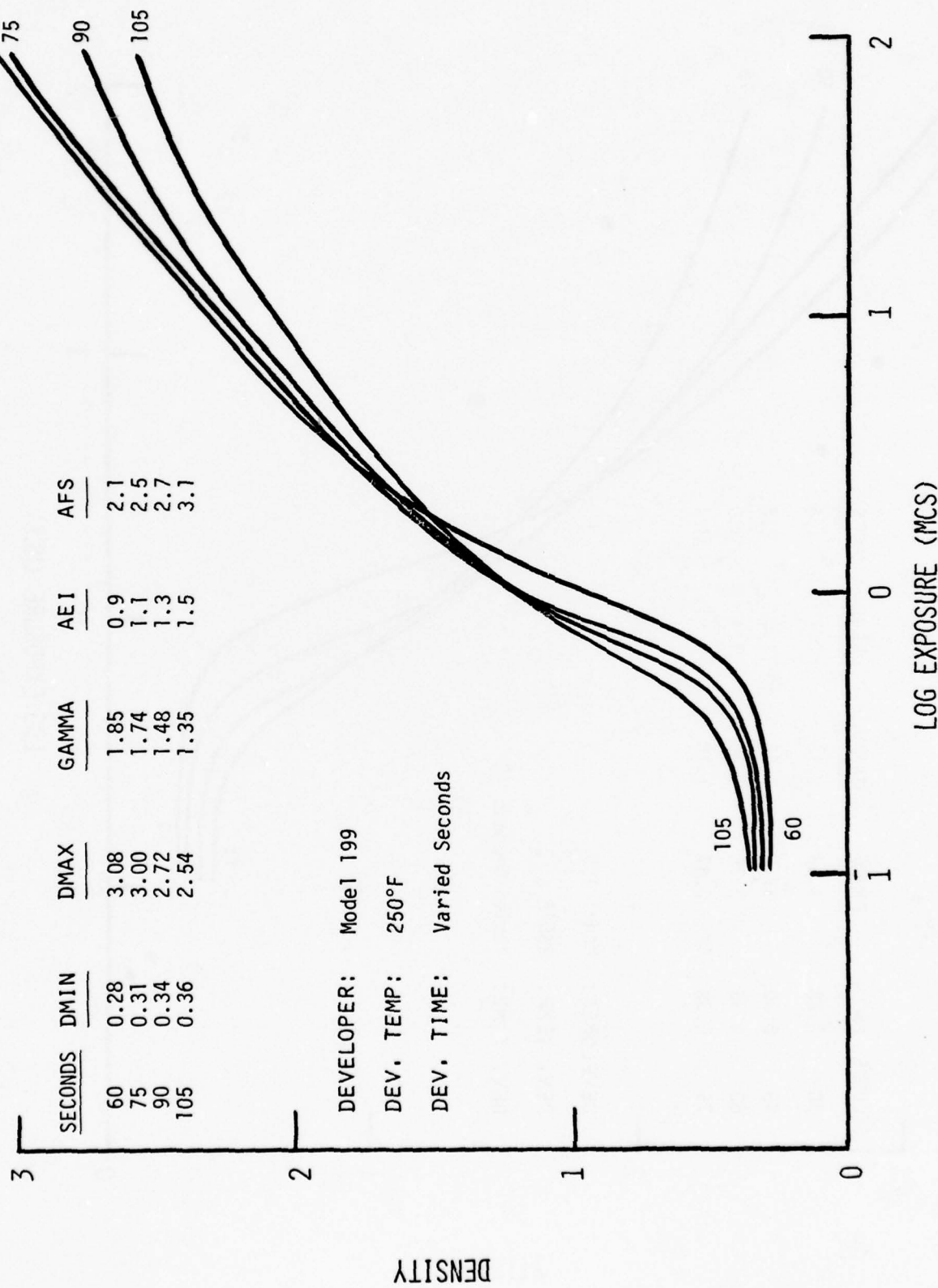


FIGURE 21 - Development Series in a 3M Model 199 Sheet Processor at 250°F for the Final Sample

CAMERA SPEED DRY SILVER FILM

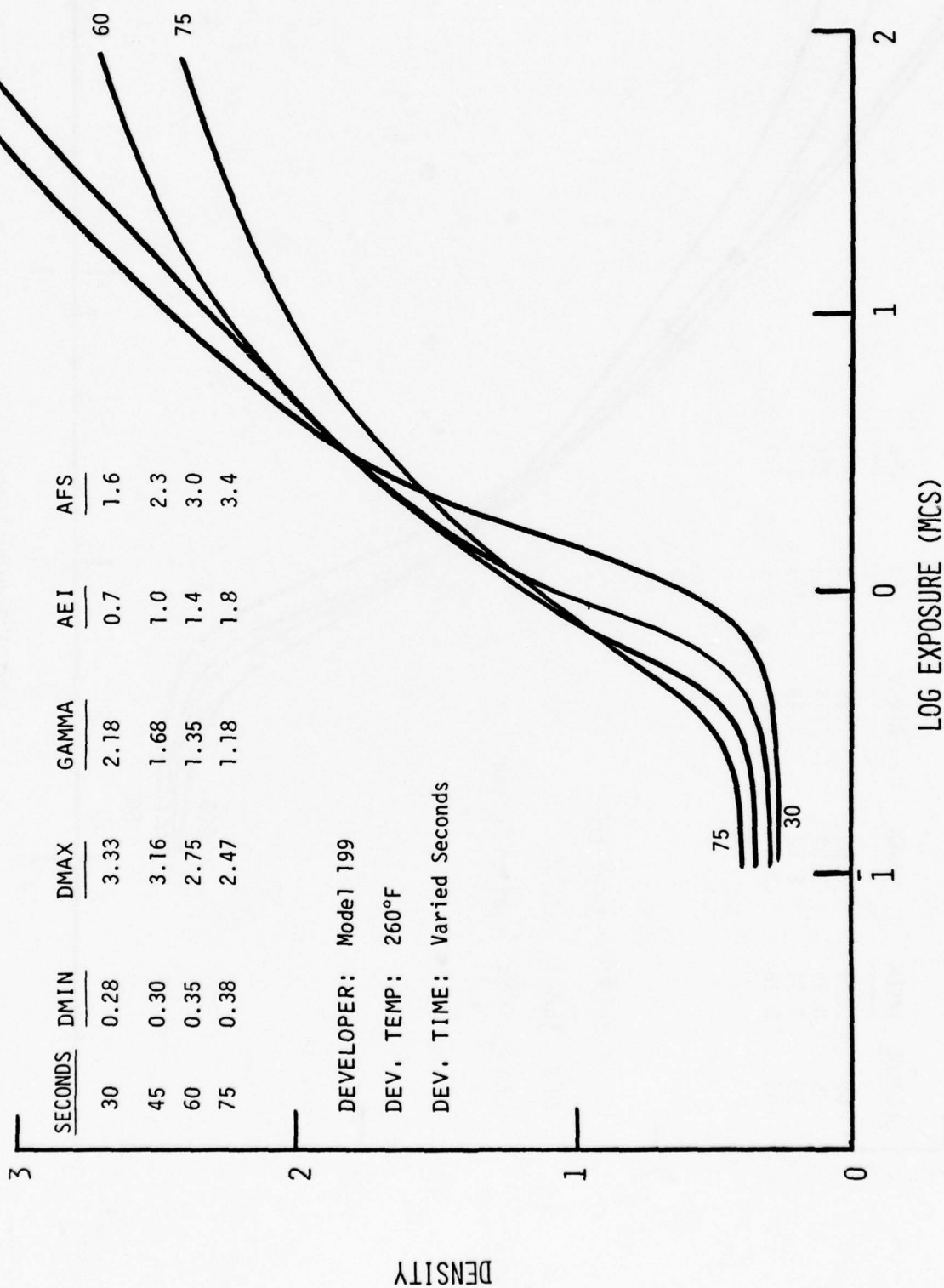


FIGURE 22 - Development Series in a 3M Model 199 Sheet Processor at 260°F for the Final Sample

CAMERA SPEED DRY SILVER FILM

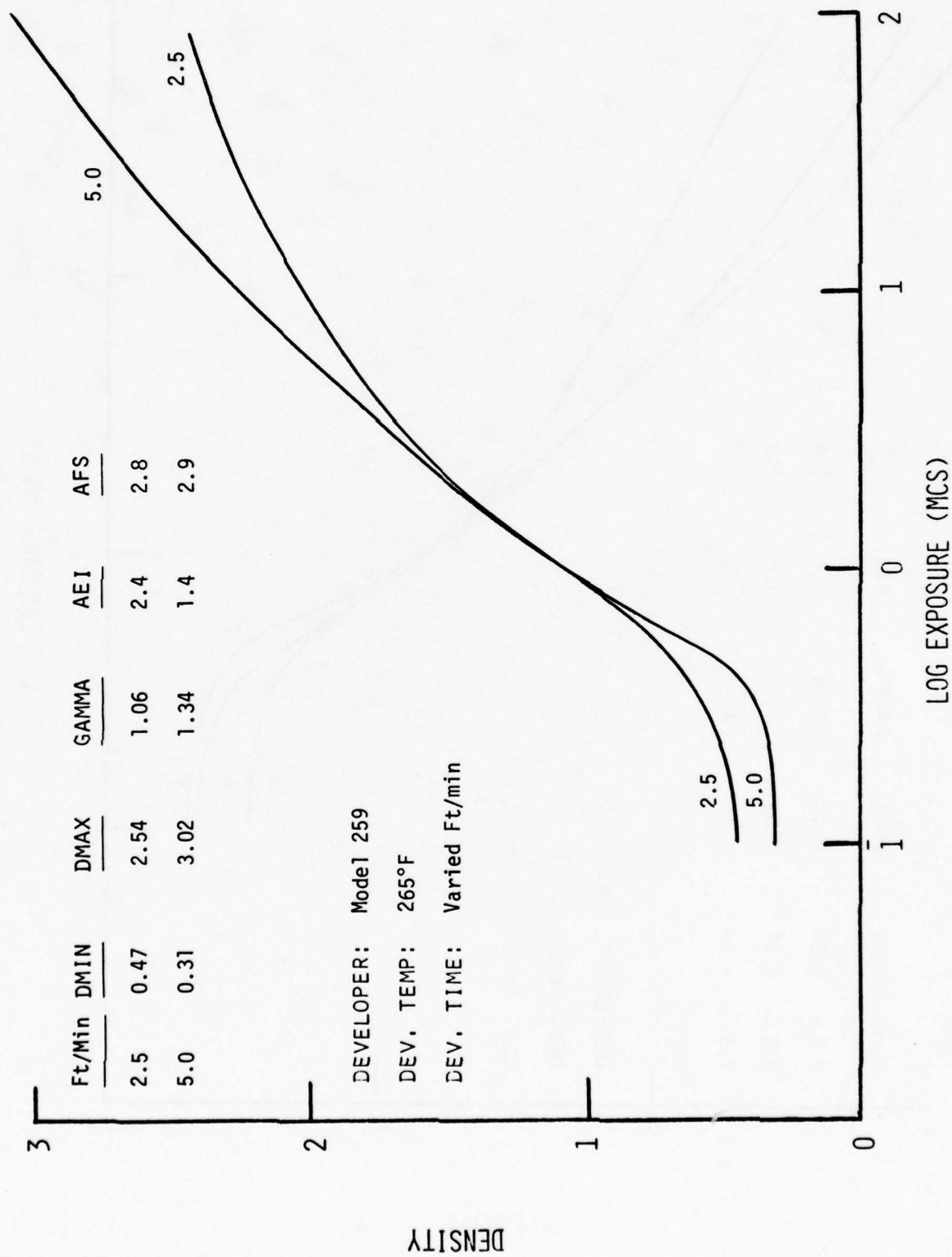


FIGURE 23- Development Series on a Model 259 Ro11 Processor at 265°F

CAMERA SPEED DRY SILVER FILM

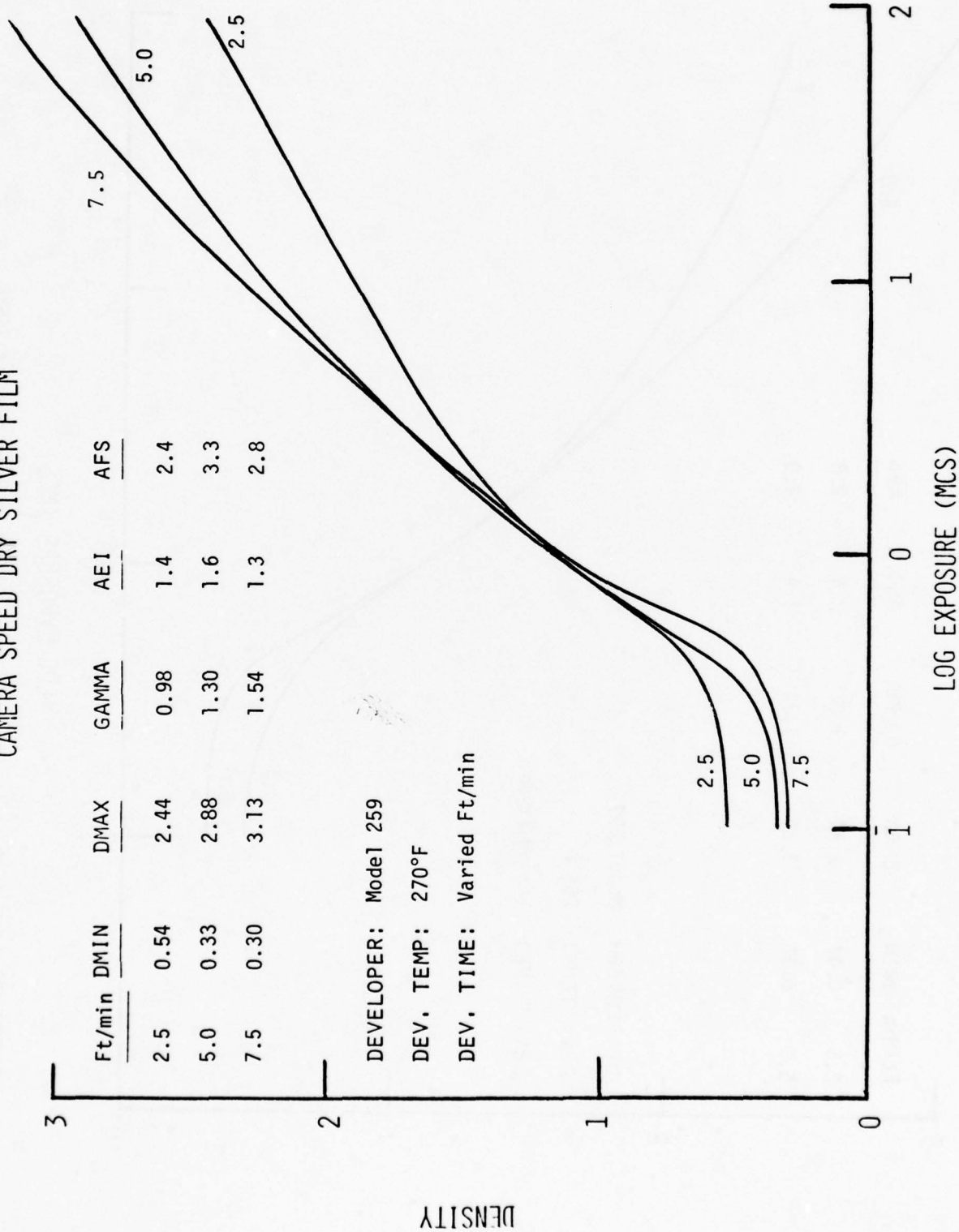


FIGURE 24 - Development Series on a Model 259 Roll Processor at 270°F

CAMERA SPEED DRY SILVER FILM

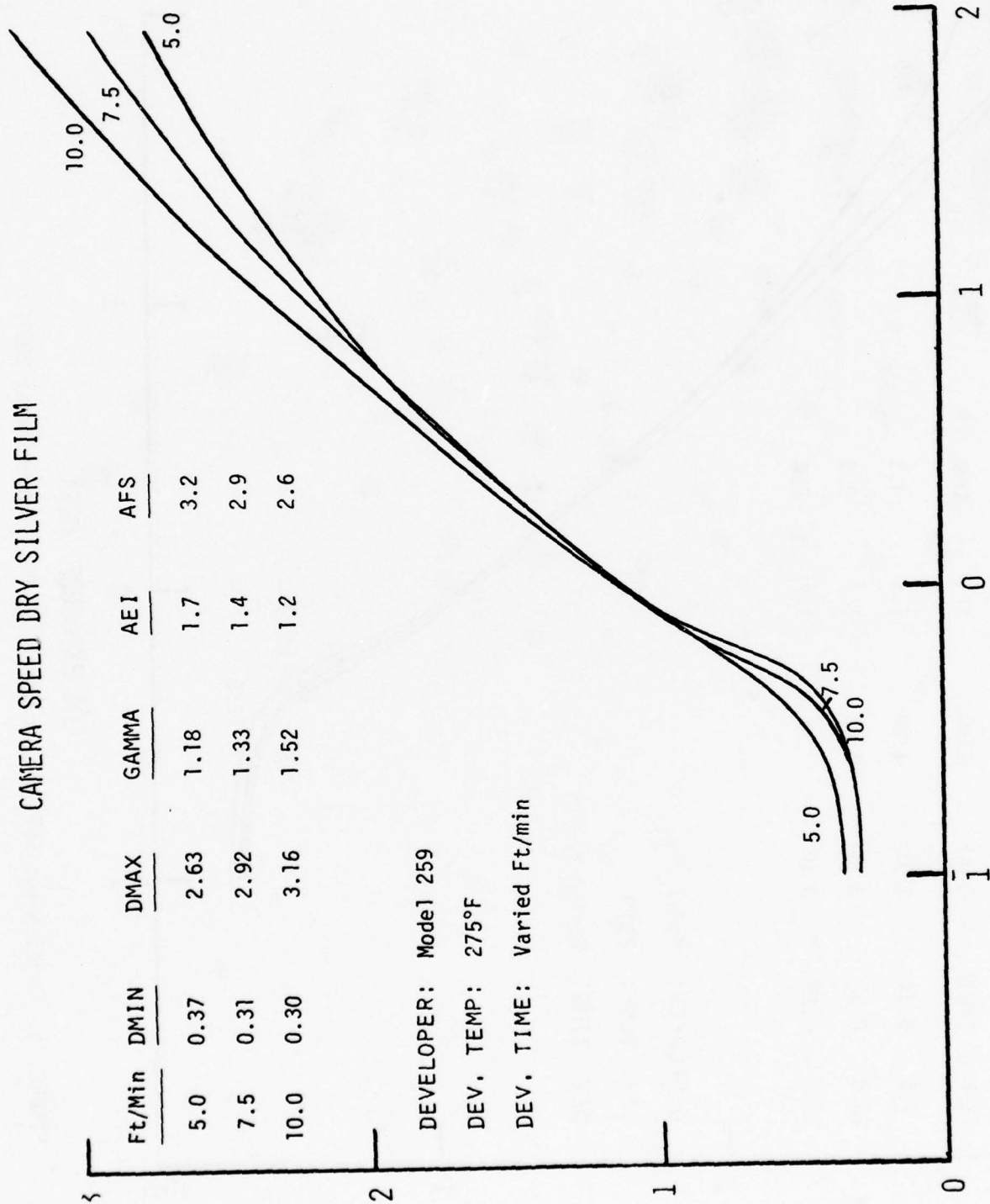


FIGURE 25 - Development Series on a Model 259 Roll Processor at 275°F

CAMERA SPEED DRY SILVER FILM

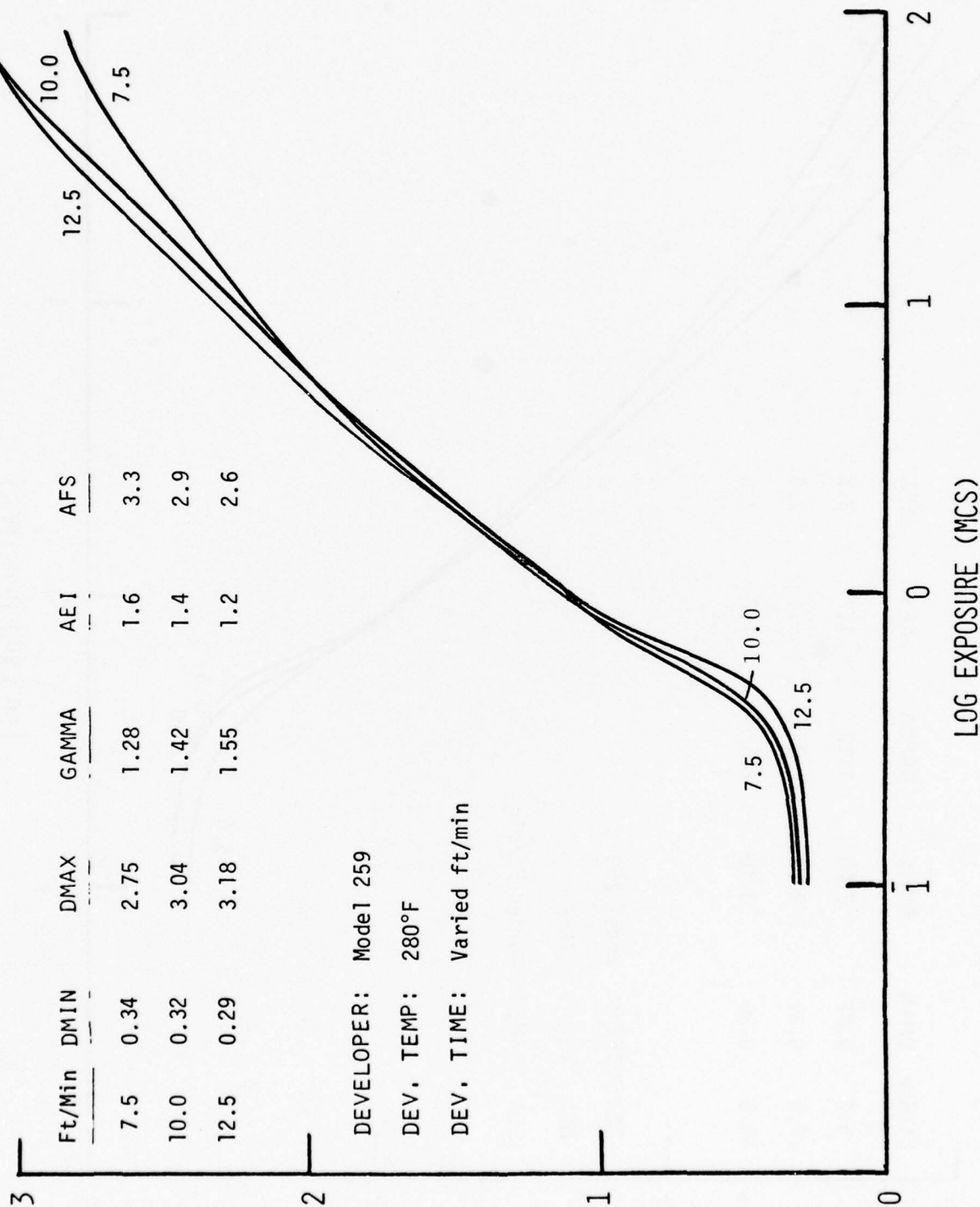


FIGURE 26 - Development Series on a Model 259 Roll Processor at 280°F

CAMERA SPEED DRY SILVER FILM

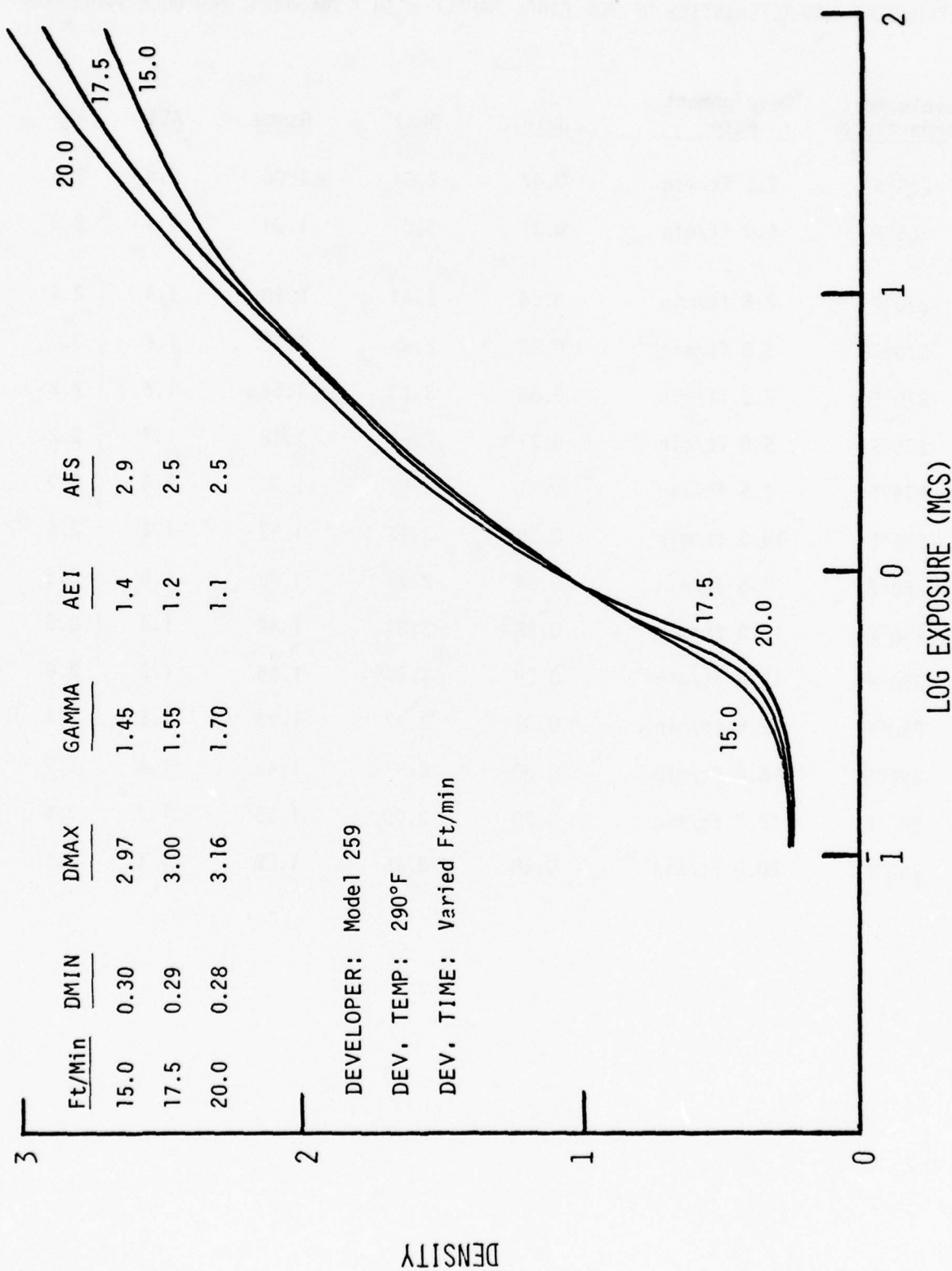


FIGURE 27 - Development Series on a Model 259 Roll Processor at 290°F

TABLE 2

DEVELOPMENT CHARACTERISTICS OF THE FINAL SAMPLE WITH A 3M MODEL 259 ROLL PROCESSOR

<u>Development Temperature</u>	<u>Development Rate</u>	<u>Dmin</u>	<u>Dmax</u>	<u>Gamma</u>	<u>AEI</u>	<u>AFS</u>
265°F	2.5 ft/min	0.47	2.54	1.06	2.4	2.8
265°F	5.0 ft/min	0.31	3.0	1.34	1.4	2.9
270°F	2.5 ft/min	0.54	2.44	0.98	1.4	2.4
270°F	5.0 ft/min	0.33	2.88	1.30	1.6	3.3
270°F	7.5 ft/min	0.30	3.13	1.54	1.3	2.8
275°F	5.0 ft/min	0.37	2.63	1.18	1.7	3.2
275°F	7.5 ft/min	0.31	2.92	1.33	1.4	2.9
275°F	10.0 ft/min	0.30	3.16	1.52	1.2	2.6
280°F	7.5 ft/min	0.34	2.75	1.28	1.6	3.3
280°F	10.0 ft/min	0.32	3.04	1.42	1.4	2.9
280°F	12.5 ft/min	0.29	3.18	1.55	1.2	2.6
290°F	12.5 ft/min	0.31	2.77	1.33	1.5	3.1
290°F	15.0 ft/min	0.30	2.97	1.45	1.4	2.9
290°F	17.5 ft/min	0.29	3.00	1.55	1.2	2.5
290°F	20.0 ft/min	0.28	3.16	1.20	1.1	2.5

CAMERA SPEED DRY SILVER FILM

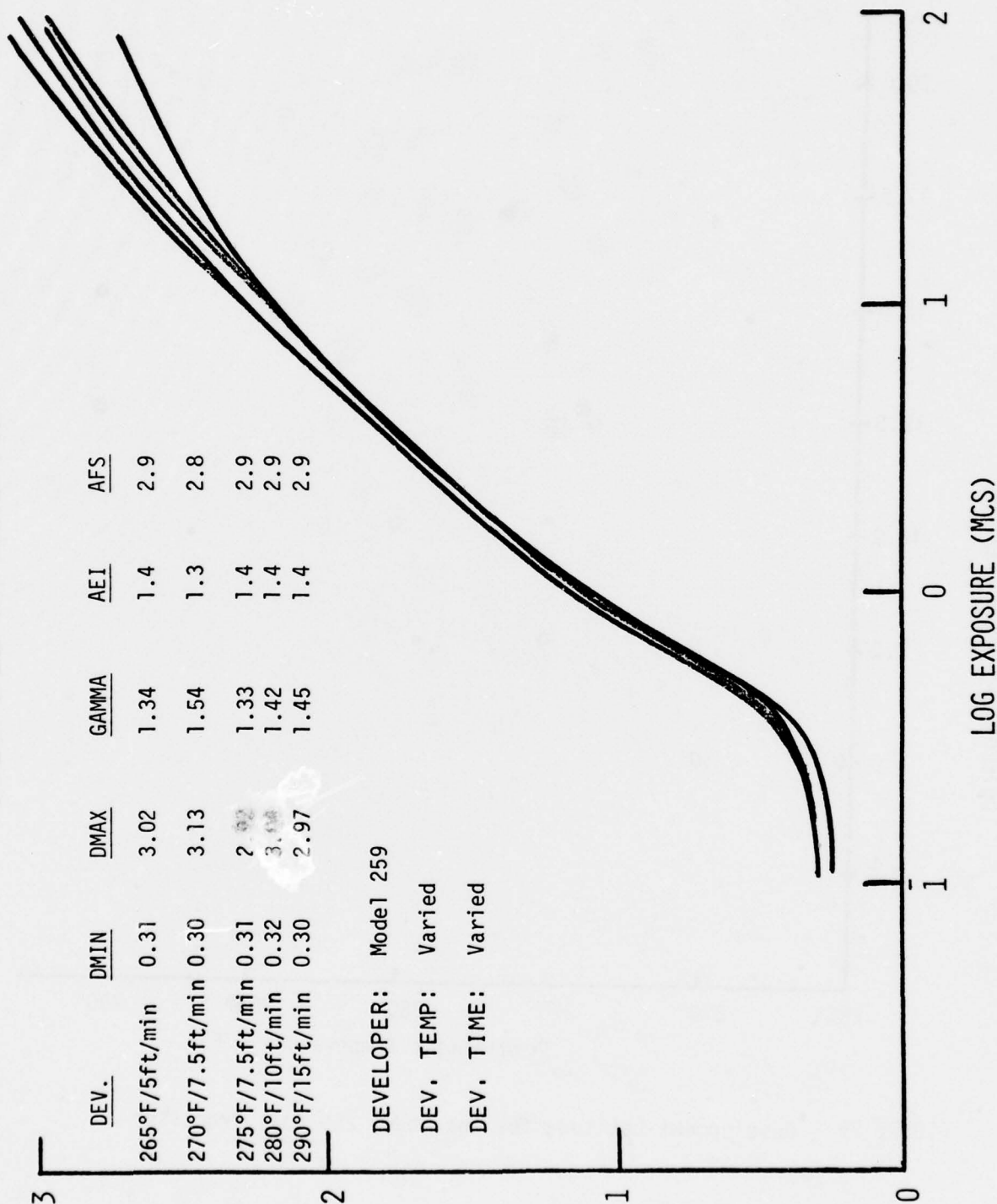


FIGURE 28 - Optimum development conditions on a 3M Model 259 Roll Processor

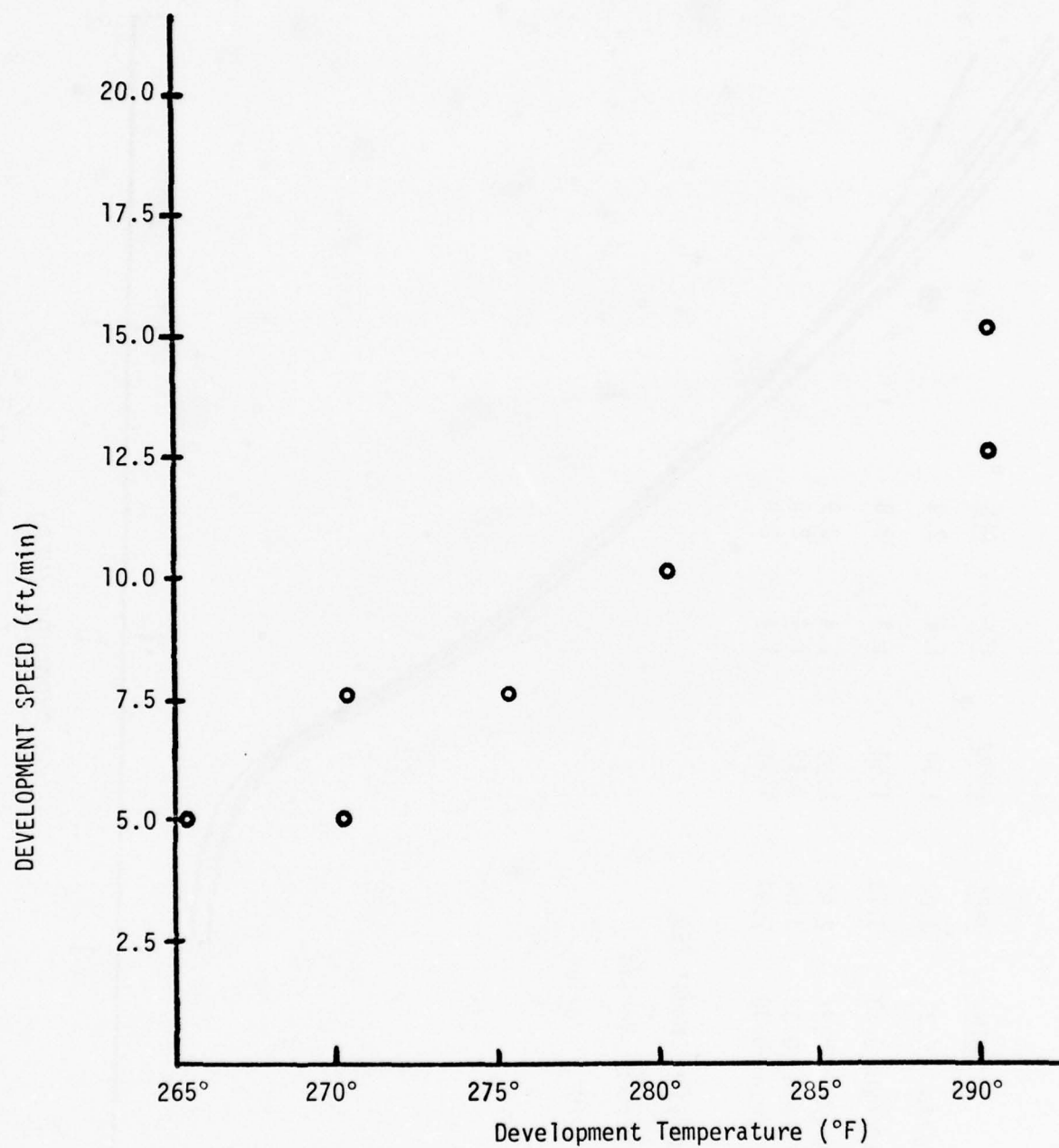


FIGURE 29 - Development Latitude for the Model 259 Roll Processor

CAMERA SPEED DRY SILVER FILM

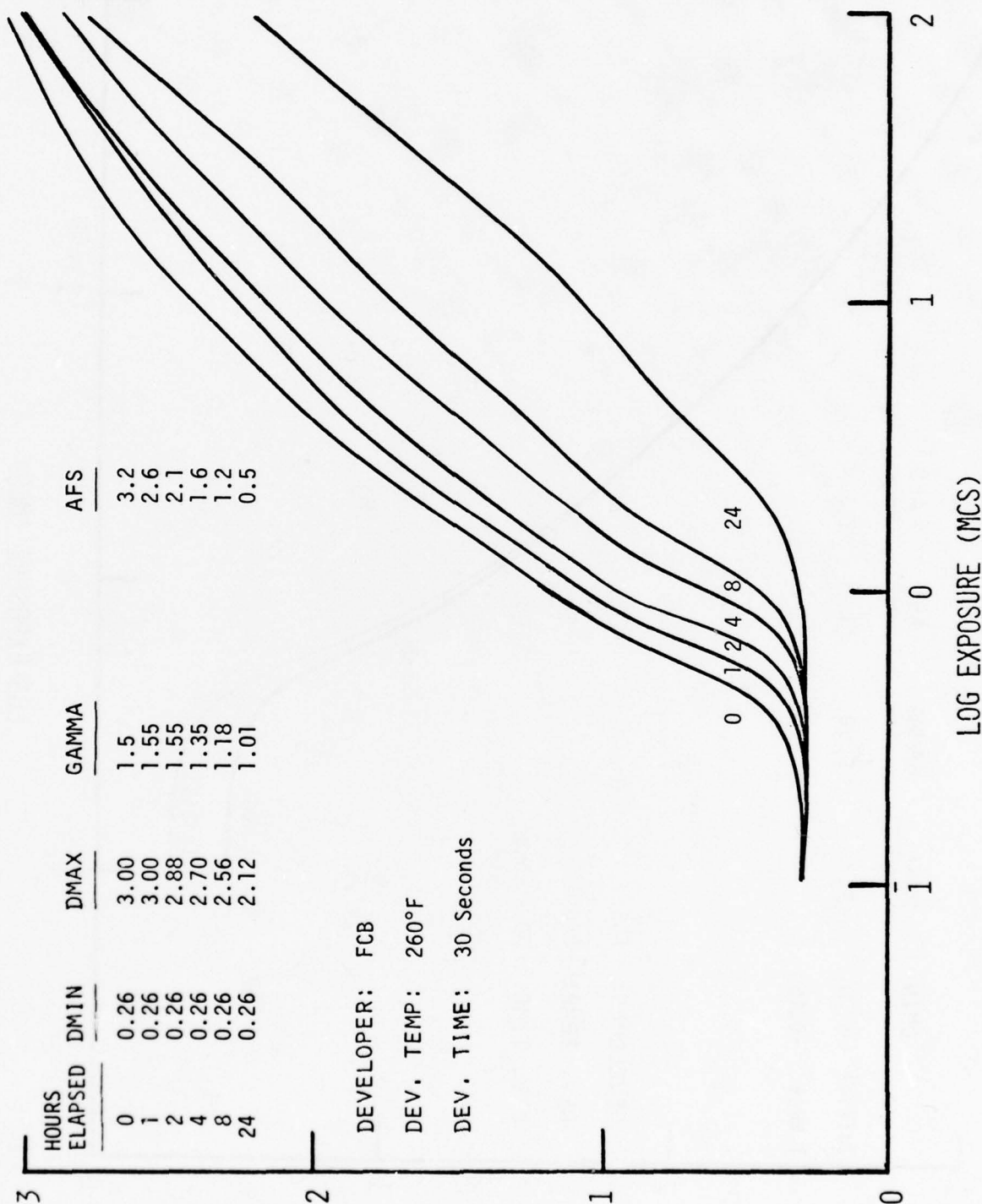


FIGURE 30 - Latent Image Study at 70°F, 50% Relative Humidity.

CAMERA SPEED DRY SILVER FILM

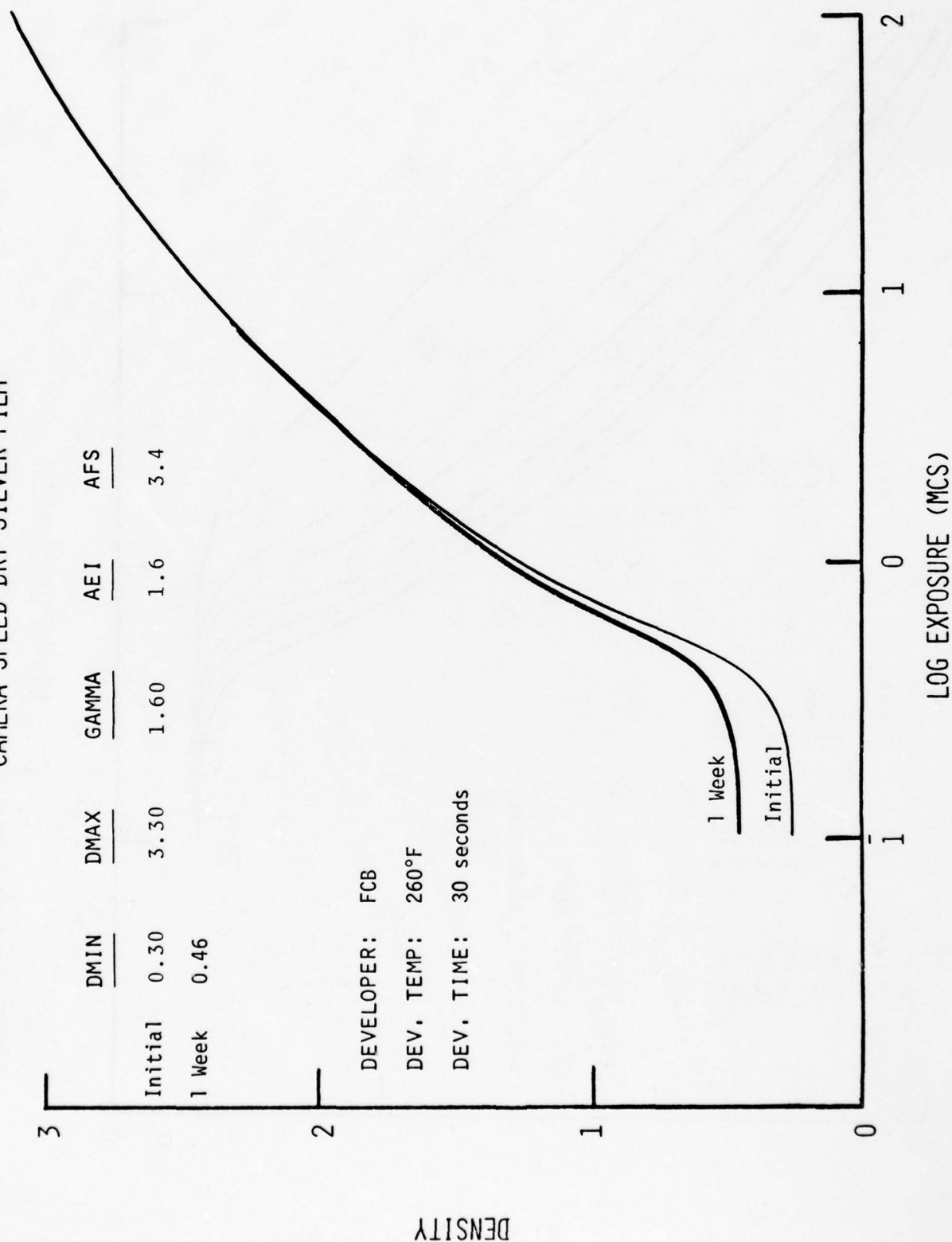


FIGURE 31 - Light Stability of Developed Film, Roomlight

CAMERA SPEED DRY SILVER FILM

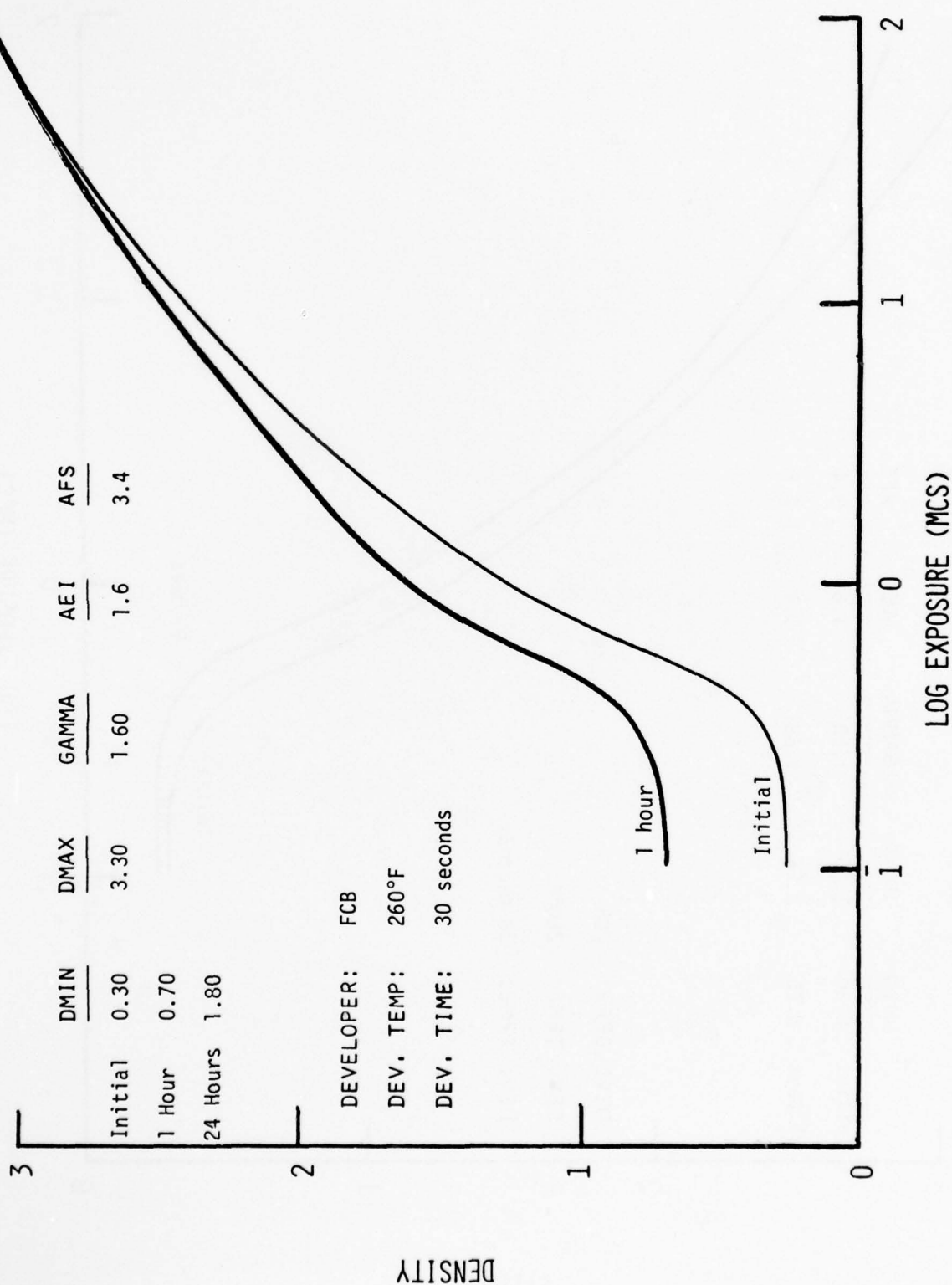


FIGURE 32 - Light Stability of Developed Film, Richard's Photointerpretation Light Table

CAMERA SPEED DRY SILVER FILM

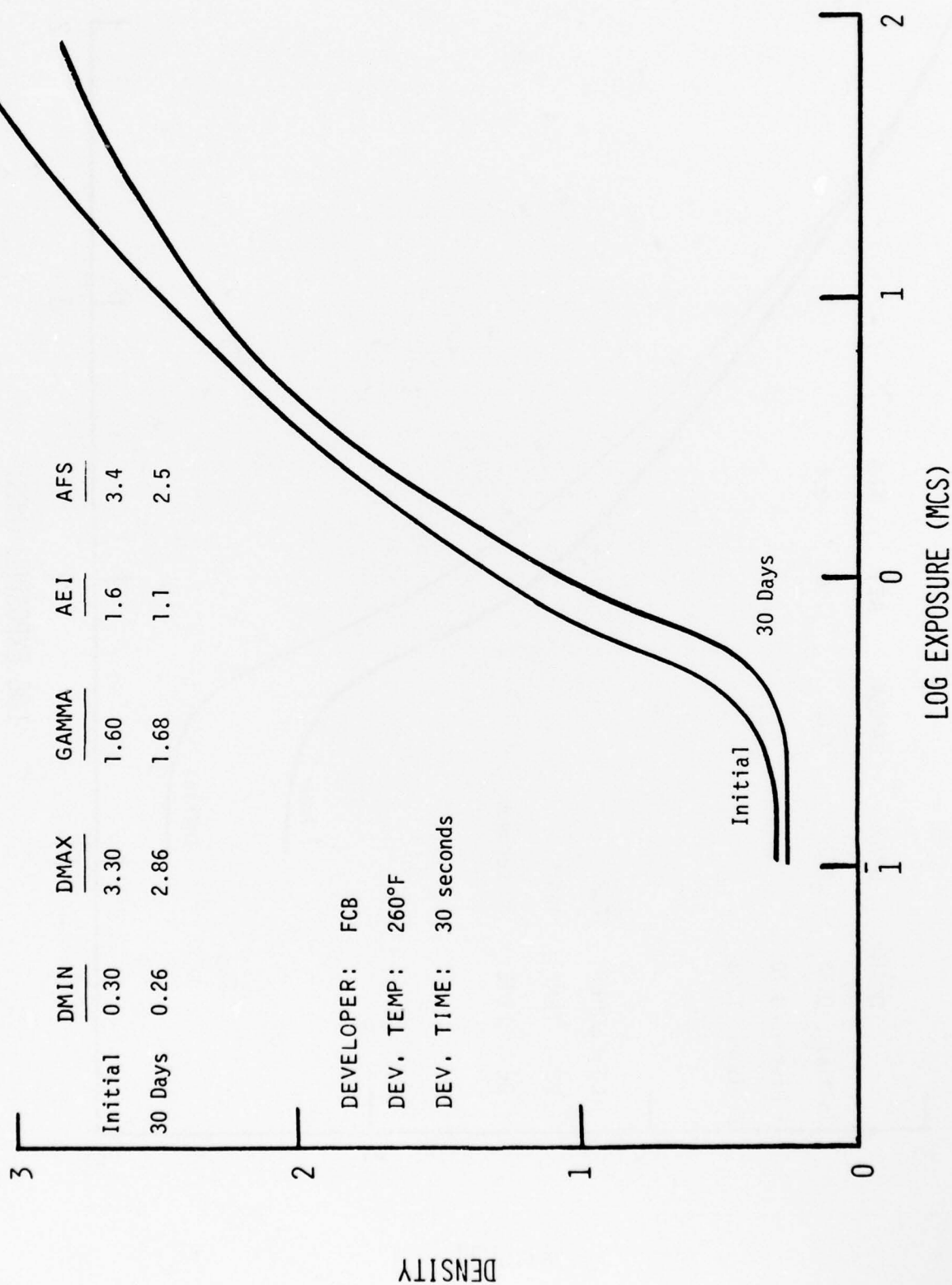


FIGURE 33 - Shelf Life at 70°F, 50% Relative Humidity

CAMERA SPEED DRY SILVER FILM

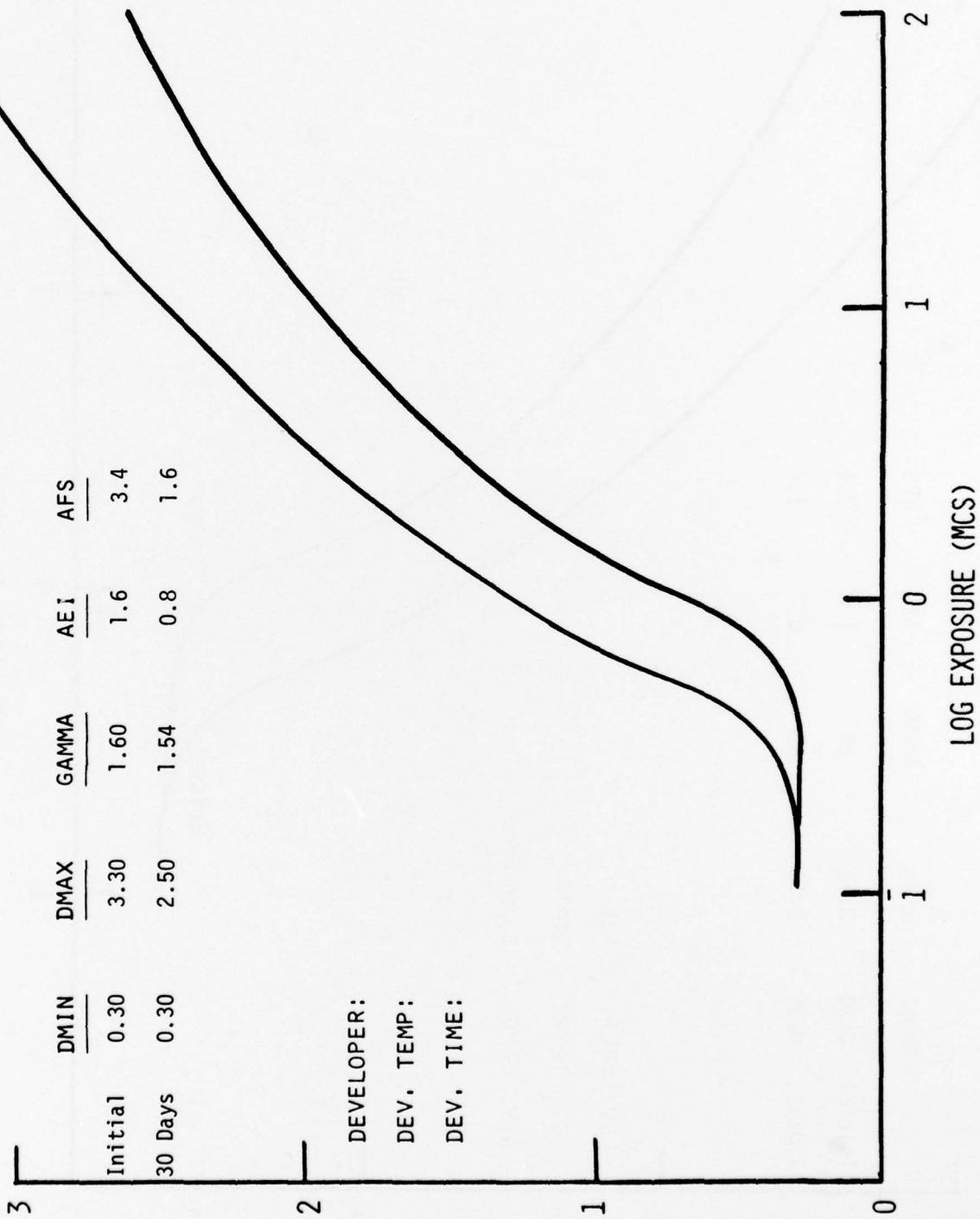


FIGURE 34 - Shelf Life at 80°F, 80% Relative Humidity

CAMERA SPEED DRY SILVER FILM

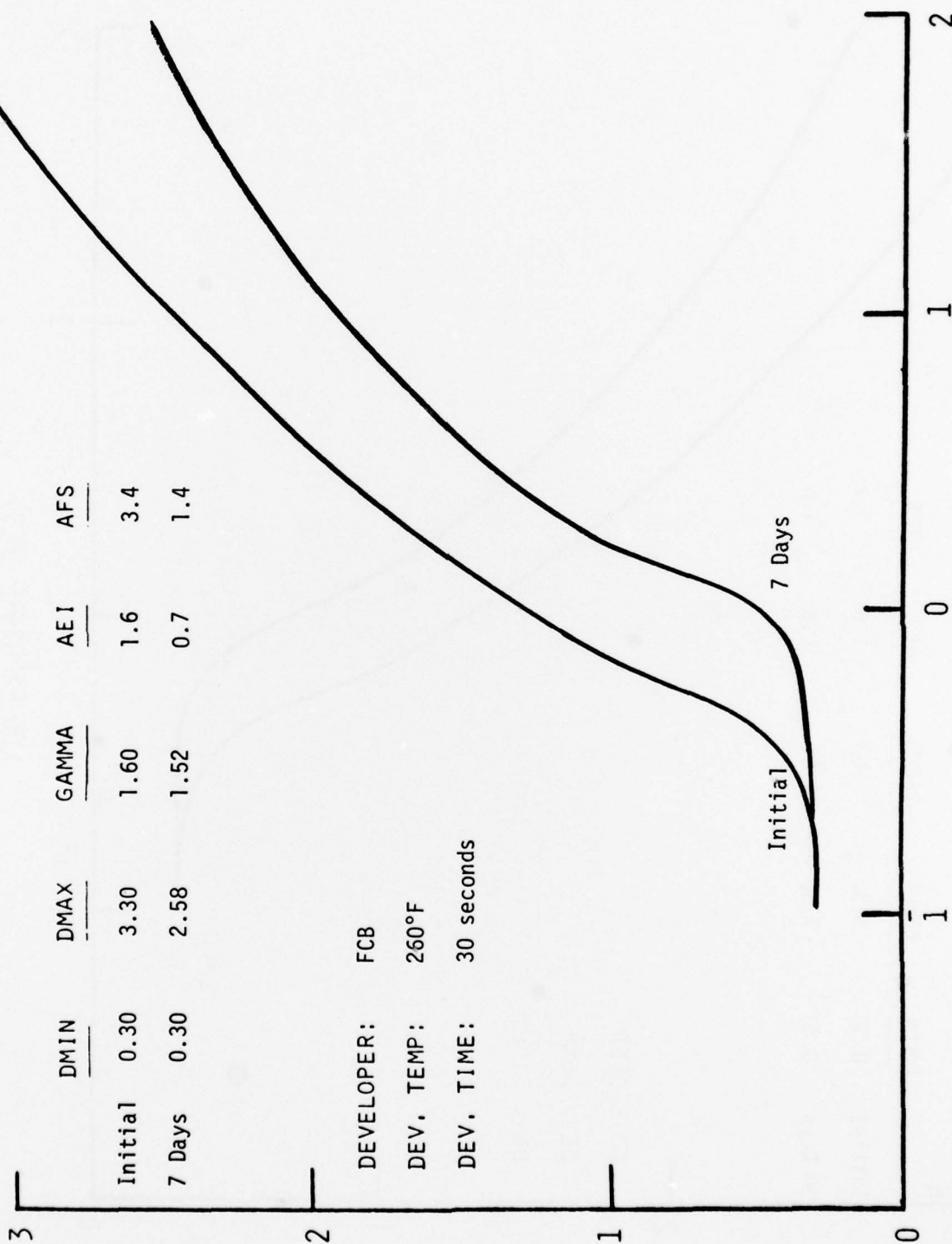


FIGURE 35 - Shelf Life at 120°F, 50% Relative Humidity

DISCUSSION

The objectives of this contract were designed to increase the sensitivity of camera speed Dry Silver film. Prior to this contract, film had been supplied to the Air Force which placed 3M Dry Silver film within the lower sensitivity range of aerial film. The contract was successful in producing sensitivities in the range of AEI 1.6 - 12.0 in the lab. The adaptation of the procedures to obtain this sensitivity will require more effort in a laboratory and pilot plant situation. The final sample represents a level attainable consistently in the pilot plant. A tabulation of the results is given in Table 3.

Although the final sample was not sensitive enough to meet contract goals, the contract was successful in studying other aspects of dry silver film. Resolution was improved in film formulations by acutance and antihalation systems. Shelf life was also studied and the knowledge from our commercial products shelf life was applied to this research program. The other aspects of the film which had not been controlled before are now being studied; latent image, reciprocity, light stability, and shelf life.

In previous contracts, the camera speed Dry Silver formulations had used the conventional formulating techniques of commercial Dry Silver films with only small increases in sensitivity being achieved. The longer term of this contract allowed new techniques in formulation, coating, and equipment to be considered for camera speed Dry Silver. This study allowed new areas of knowledge to be applied to camera speed Dry Silver.

Future work will be devoted to increasing the lab sensitivity while also transferring the current laboratory sensitivity to a pilot plant coater. A further sensitivity increase might be possible over the contract final sample if we successfully transfer our current knowledge to the pilot coater. The knowledge from this work can be applied to other less sensitive Dry Silver in the areas of acutance, stability, dye sensitivity, etc.

TABLE 3

OBJECTIVES AND RESULTS OF CONTRACT

OBJECTIVE	RESULT	
	Lab	Final Sample
Spectral Sensitivity	400-720nm	400-720nm
Photosensitivity	AEI=20	1.6
Gamma	1.2 to 1.6	
Image Color	Neutral Black	Neutral
Useful Log E Range	2.0	Greater than 2.5
Resolution High Contrast	200 cy/mm	203 cy/mm
Film Processing	30 seconds or less at temps below 300 F	Standard Condition 30 seconds 260 F
Latent Image	No loss in 24 hours	0.3 Log E in 4 hours
Image Permanency	0.1 Dmin increase 7 days - Richard's Light Table	1.8 Dmin Increase
Shelf Life	1 Year below 50 F	Data unavailable at this time.

(1) No data is given for these lab results. Films have been prepared in the lab which realize these objectives but not all three at once.

APPENDIX I

3M'S DRY SILVER TECHNOLOGY

David A. Morgan
3M Company
St. Paul, Minnesota

I am indeed pleased to have this opportunity to address the Photo Scientists of Japan.* We in the United States are aware of the Japanese tradition and respect for change and progress. I hope this paper will help satisfy your desire to know more about 3M's Dry Silver process and stimulate your interest in this technology.

Photo scientists have worked for years trying to develop a simple, dry photographic process. In most cases efforts were made to generate water in situ to satisfy the essential need for moisture during processing. Commercially, the most successful approaches have been diffusion transfer techniques and various viscous processing methods. We did not attempt to use these approaches as we began our research to develop a dry photographic process. Instead we started with a thermally induced reaction that would produce silver images and added a method of light activation or stimulation. In other words we started with a dry process and made it light sensitive.

A brief list of the essential elements that make up a Dry Silver media are: 1.) a silver halide latent imaging forming material in catalytic proximity to a light stable, heat reducible silver compound, 2.) stable chemicals that will reduce silver compounds during heating. All of these materials must be white or colorless and must remain stable at room temperature for long periods of time.

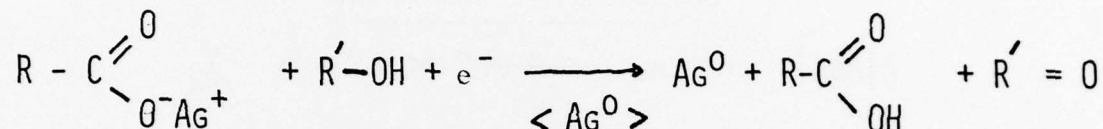
Today I would like to explain how the process functions and give you a better understanding of the various constructions and compositions. The process as we can define it is catalytic. Light energy is used to produce the catalyst and heat energy is used to produce the silver image. It's worthy to note that the catalyst is not consumed or changed in any manner in the chemical reaction between the reducing agents and the silver salts contained in the coating.

When a Dry Silver media is uniformly heated over its entire surface, the chemical reaction occurs at a tremendously accelerated rate in the light struck areas. This, of course, is analagous to normal photography.

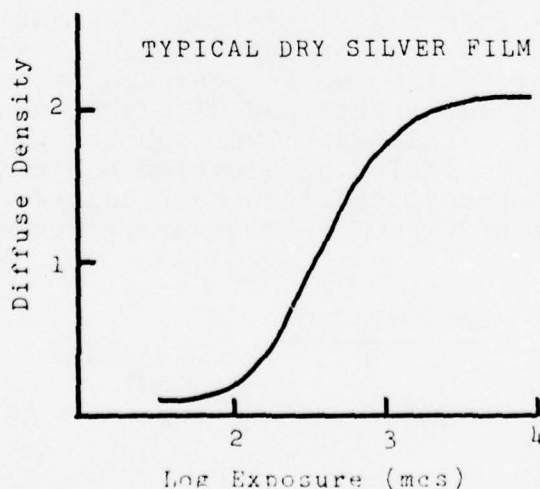
*Paper presented at the 1973 SPSE International Tokyo Symposium

The developing rate is normally between 3 and 20 seconds and at temperatures between 250-280F. The following chemical reaction occurs:

IMAGE DEVELOPMENT

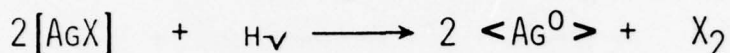


The rate of reaction for any given formula of Dry Silver material is dependent on the amount of catalyst generated through exposure and the time and temperature used to heat the material. The reaction between the reducing agents and the silver salt is quite effective and it is not necessary to remove any by-products; therefore it is not necessary for any vapors or particulate materials to be removed from the coating during the heating. In fact, the presence of the reaction products, as well as the type and amount of chemicals used, gives a measure of control or latitude to the reaction. This reaction also can be controlled by the type and quantity of chemicals used to produce images at varying gradients. In other words we can control contrast, gray scale, or gamma through chemical selection. An example of the normal contrast of Dry Silver films is shown on this next slide.



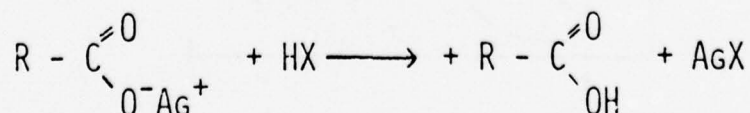
I will now discuss briefly the latent image forming reaction. I will not, however, go into theoretical detail because I believe theories of latent image formation in silver halide crystals have been discussed thoroughly in the literature by experts. The material that forms the light sensitive part of our media is silver halide, and we believe this silver halide to respond to light in much the same manner in our system as it does in wet silver halide systems.

LATENT IMAGE FORMATION

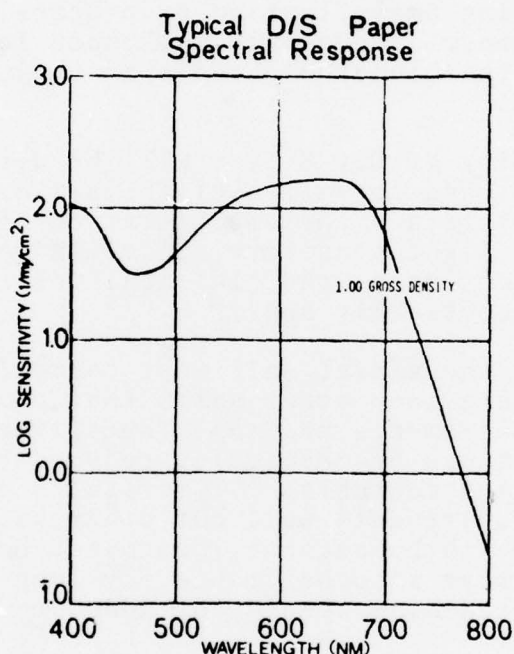


We have made Dry Silver materials using chloride, bromide, or iodide and they have the spectral response you would expect. Most commercial Dry Silver products have the latent image forming capability in the form of silver bromide. The more sensitive materials use both silver bromide and silver iodide. The necessity for catalytic proximity of the latent image to the silver source from which the silver image is generated is a vital factor in making the system work. One way to achieve this is by using a single silver compound as the progenitor for both the silver halide and the silver metal image. We have developed a technique for doing this by forming the silver halide in situ from silver compounds. Silver behenate is one of our preferred silver salts. We cause the in situ silver halide formation by reacting the silver behenate with bromide and iodide ions. We control this reaction by carefully monitoring the concentrations of ions, temperature and contact time. The importance and significance of the in situ formation is evidenced by the performance of the material as those of you who have experimented with this system can verify. The source of halide ions can be from any practical source and is practically unlimited. The halide source may be organic or inorganic and the associated cation often has significant desirable or undesirable effects such as fog or antifogging. The in situ formation of the silver halide can be achieved by several techniques, each having advantages and disadvantages. During our experimenting we have reacted silver sources with halide ions in a variety of ways including slurried and dispersed, powdered and silver compounds that had been coated on a base of paper or film. All of these techniques are workable.

HALIDIZATION

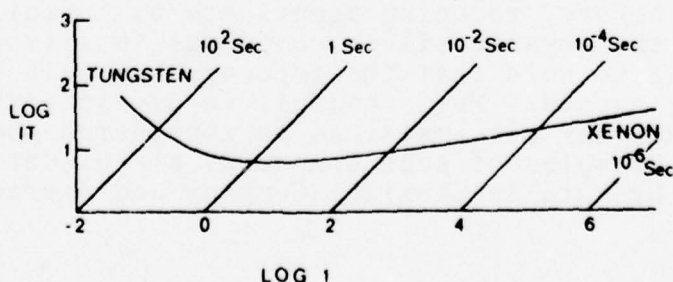


Nominally we convert 50 to 10 percent of the silver to silver halide, but the percentage may vary considerably from the norm. Some products have less than one percent converted and some more than 10 percent. The low end is limited by lack of sensitivity and the high end by poor stability and image density. This silver halide in catalytic proximity to a silver salt when exposed to light will form a latent image we believe to be similar to other silver halide latent images. It can be dye sensitized to specific parts of the visual spectrum or it can be made panchromatic. In most respects, it has characteristics similar to a normal silver halide latent image. This next slide shows the response of a dye sensitized Dry Silver paper.



The reciprocity law conformance of Dry Silver materials can be illustrated as follows:

**3M DRY SILVER FILM
RECIPROCITY**



The efficiency of the latent image is dependent on how well it can be amplified. The sensitivity of a Dry Silver media also depends on how well the latent image can be amplified. The amount of silver halide used and the energy needed for a given density indicates a surprising efficiency. For instance, a typical film would contain a total silver content of 100 mg/sq. ft. of which 10 mg is silver halide. This film can be imaged to a Dmax of 2.0 with an exposure of approximately 100 ergs/cm². One rewarding approach to improving the sensitivity of our material has been to improve the efficiency of the latent image. For example, we've achieved a 1000-fold sensitivity increase without changing the latent image forming mechanism. This improvement in sensitivity came from a more efficient utilization of the latent image and was accomplished mainly by keeping fog levels down during amplification or processing. One way to do this is to choose a developer with much less tendency to cause fog, such as the hindered phenols in conjunction with various antifoggants.

The future sensitivity of Dry Silver will be determined by two factors; the control and formation of the silver halide latent image forming materials and the amplification of that latent image. Because the light sensitive silver is not used as a source for the silver image, the classical relationships and approaches do not necessarily apply.

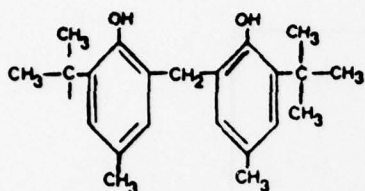
As I had mentioned, the silver salt most commonly used is silver behenate but there are many other salts that can be used as well. The fundamental requirements are that the silver salt be stable to light and that it can be chemically reduced by heating, and must be economical and coatable. Silver salts of long chain acids meet these requirements well but other silver salts can be used: phthalate, phthalazinone, benzoate, benzotriazole, etc. These silver salts must also be stable for long periods of time in the presence of reducing agents for adequate shelf life at normal temperatures.

There is also a need for adequate resins for dispersing and coating the Dry Silver materials. Fortunately there are many such resins commercially available. Preferred resins are vinyls and acetates. Examples of these are Eastman Kodak's cellulose acetate 398-6 and Monsanto's Butvar B-76. Up to now we have preferred to use solvent soluble resins but this is not essential. We have also developed water soluble systems that perform well.

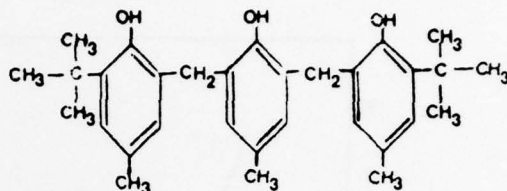
As mentioned before, reducing agents are essential for the reduction of the organic silver compounds to silver metal. It is interesting to note that the type of chemicals used are very weak reducing agents. Most commonly we use antioxidants that would not under any circumstances be considered photographic developers. Examples of such compounds are hindered phenols manufactured by Catalin Chemical Company and American Cyamide, respectively.

The next slide illustrates two such examples. Although the standard type developers could be used, their high level of activity is not optimum for good stable dry processed materials and it is difficult to keep fog levels down. However, the first commercial products used hydroquinone, a very powerful developer as you well know.

Example 1

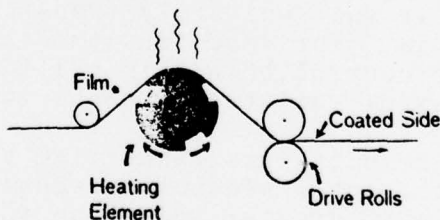


Example 2



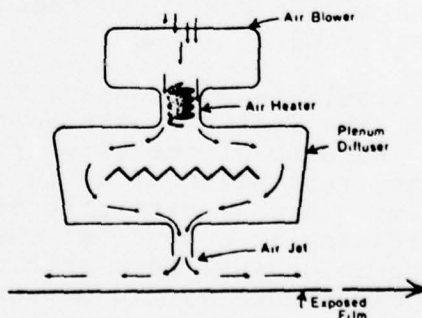
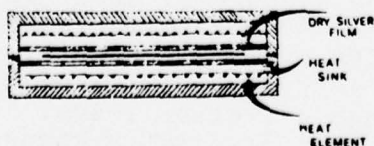
I would now like to go into more detail in the area of processing or developing of Dry Silver materials. As mentioned earlier, the actual temperature of the materials determine the development rate, not the temperature of the device or media being used. Therefore, the coefficient of heat transfer is the dominant factor. Dry Silver materials can be developed in several ways over a wide range of time and temperature to reproduce exactly the same response. It matters very little if conductive or convective processing techniques are used. Examples of existing methods include a heated mandrel, convection oven or heated air. The results are essentially identical. The indicated time and temperature will not be the same due to difference in the heat transfer rate. This visual illustrates my point.

HEATED MANDREL



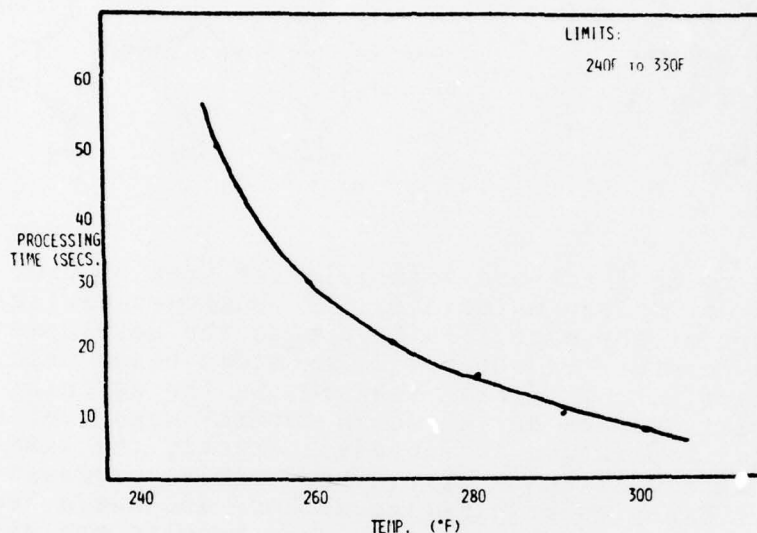
Heated Air Development

CONVECTION OVEN



For any given material there exists a wide range of dwell times and temperatures that can be used to give the same results. For instance, if a very short dwell time is needed, a relatively high temperature must be used. Conversely, if low temperature is applied, a relatively long dwell time must be used.

PROCESSING TIME VS. TEMP.

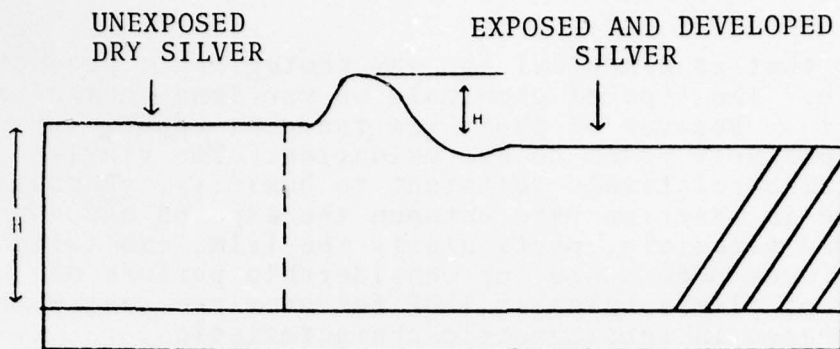


This type of relationship can be developed for each method of development.

Because the rate of heat transfer is important, uniform contact is most important. Even though a device may have adequate temperature control, it must uniformly transfer this energy to the Dry Silver media. For good sensitometric reproducibility, temperature should be controlled within $\pm 1.5^\circ\text{C}$ although for many applications, larger variations can be tolerated.

And for very precise results less than $\pm .5^\circ\text{C}$ variation is desirable. Because the interface between coating and base is uniform and the base much greater mass than the coating, it matters very little as to which side is heated from a kinetic point of view. There are limitations, however, because of physical distortions of the coated surface of some medias if it is touched during processing.

The image itself is, of course, composed of finely divided particles of silver. The particles are on the average of about $100\text{\AA}$ in diameter. Because of the size of the silver particles and their compactness, resolution of up to 1000 cy/mm are possible on Dry Silver films. A characteristic of a Dry Silver image that is most interesting is a physical change or relief image. Dry Silver images have an unusual edge effect that displays a change in thickness of as much as 2.5 microns . This phenomena is still being studied, but it can be illustrated as follows.

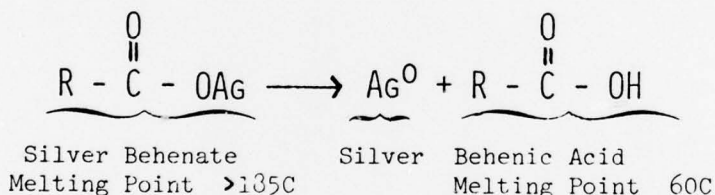


H = 10 MICROMETERS

h = 1.5 MICROMETERS

Relief resolution of up to 100 cy/mm has been recorded by measuring the change in thickness of images made by exposing the samples from a resolution target, processing and measuring the physical change. This relief image will diffract a laser beam and there are some applications utilizing this characteristic.

There are other physical and chemical differences in the images and non-imaged areas. This can be illustrated in the following slide.



The stability of Dry Silver materials is, of course, a question that must be discussed. Because it is a catalytic process with nothing added or removed the potential for a continued reaction, after development, is there, particularly in the non-imaged or background areas. Once a Dry Silver media is exposed to light, the potential for the reduction of the silver salts exists if heating should occur.

The reaction can be controlled and optimized for elevated temperatures so that it is not necessarily a serious problem for normal use products. From a fundamental point of view, however, it is one of the characteristics that we are trying to improve. This problem is certainly not unsolvable and various approaches are being used. One technique involves adding chemicals which generate oxidizing agents upon exposure to light. This, of course, offsets the reduction potential that does exist and tends to destroy the residual latent image. Other approaches are the generation of stabilizers by chemical reaction or physically mixing preformed stabilizers, both methods being initiated by heat.

A characteristic that is essential for any photographic product is its shelf life. The type of chemicals we use lend themselves to good shelf life. Because of their low reducing capability they are not excessively prone to air oxidation. The vinyl resins used are also relatively resistant to humidity. Because of the difference in reaction rate between the exposed and non-exposed areas, the materials, particularly the film, can tolerate elevated temperatures before use for considerable periods of time. We have kept film samples at 110F for over one year with essentially no change in sensitometric characteristics.

In this paper I have tried to explain a relatively complex process with general but hopefully meaningful explanations. It would appear that products developed from this technology will find applications where certain characteristics are essential, these characteristics being dryness and rapid access in conjunction with continuous tone and high resolution. We do not believe Dry Silver technology is a replacement for wet silver halide photography at this time. It should be applied where its present characteristics fit. What the future will bring forth is still uncertain.