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Second Report on

Reflection Reducing Coatings for Glass and Transparent Plastic Surfaces

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WASHINGTON, D. C.

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Prepared by:

Peter King, Associate Chemist

Reviewed by:

Allen L. Alexander, Chemist

P. Borgstrom, Head Chemist
Superintendent Chemistry Division

Approved by:

A. H. Van Keuren, Rear Admiral, USN
Director

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INTRODUCTION

(a) Authorization

1. This study was authorized by Bureau of Aeronautics letter Aer-E-2573-ERM, F38-2, 181359, dated 21 December 1942.

(b) Statement of Problem

2. The investigation directed by the authorizing letter was to study application of reflection reducing coatings to transparent plastic surfaces. However, as a result of the work done on plastics it was found possible to apply very good reflection reducing coatings to glass as well. The Bureau of Aeronautics is confronted with the problem of reducing reflection from glass and plastic surfaces on instrument panels; therefore, this report is submitted for consideration by the Bureau as it contains directions for applying films to instrument window glasses.

(c) Known Facts Bearing on the Problem

3. Methods for the preparation of reflection reducing coatings on glass surfaces have been known for several years. At present, the Navy has many of the lenses and prisms of its optical instrument coated with magnesium fluoride films. The method of doing this is well established and it is now used on a large scale. The evaporation process of depositing magnesium fluoride films is expensive and requires an elaborate evacuated system. The films obtained by this method are excellent, even, and very hard. The reduction in reflection amounts to about 70 percent.

4. It has been found possible to obtain good films by depositing silica on glass or plastics thereby obtaining a reduction in reflection as high as 98 percent. The American Optical Company has prepared reflection reducing coatings with this material and obtained excellent results. However, in order to obtain reasonably hard films two applications of material are necessary. The results reported here require only one application and are offered as a simplification of the method used before.

(d) Previous Work Done at this Laboratory

5. This Laboratory has previously written three reports on reflection reducing coatings. These are: Naval Research Laboratory Report No. H-1692 entitled "Metallic Fluoride Films of Low Reflectivity," dated 10 February 1941, NRL letter C-A13 (422) (451-PK) of 12 November 1942, and a report on "Reflection Reducing Coatings for Transparent Plastic Surfaces," No. P-1987, dated January 22, 1943. The first of these reports deals with evaporated films and gives specific directions for obtaining them. The second deals with films of silica produced from three different solutions. The third report mentions briefly the kind of film reported here and describes in detail the method for spraying material to obtain reflection reducing coatings.

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METHOD

(a) Apparatus

6. A small synchronous motor was used to remove the glass panels from a suspension of the coating material. This motor made one revolution a minute and a 1-5/16 inch pulley was mounted on the shaft. This gave a linear movement of about 4 inches per minute. The coating material can be held in any convenient container. It is preferable that this container not be made of metal as the solution contains a small amount of hydrochloric acid.

(b) Material

7. The concentrated suspension from which these films were made was designated simply as solution No. 115 by the American Optical Company. This material was diluted with solvents which are commonly used in the paint industry and ordinary commercial grades proved to be of sufficient purity for this application.

8. A small amount of ethyl silicate was also required. This product was manufactured by Carbide and Carbon Chemical Corporation and was used without purification. The hydrochloric acid needed to hydrolize the silicate was the ordinary concentrated acid supplied by J. T. Baker and Company.

EXPERIMENTAL

(a) Application of Films

9. Before applying the film to glass or plastic it was necessary to wash the surface and remove all dirt. This was done with hot water and soap, after which they were dried by one of several convenient methods. When clean, the material to be treated was placed in the coating suspension and removed at a constant rate by means of a synchronous motor. With the particular suspension used here, the linear rate of removal was about 4 inches per minute. Care should be taken that the surface of the liquid comes to rest after inserting the panel and before the withdrawal of the panel has started. If this precaution is not observed, the film will be uneven and will have bright streaks in it which have a rather high reflectivity. With a small amount of effort very even films can be produced.

(b) Composition of Suspensions

10. The suspensions used were prepared from the No. 115 suspension furnished by the American Optical Company. This is very concentrated as received and must be diluted before application. One suspension made with solution No. 115 had the following composition.

46 cc. Cellosolve
25 cc. Butanol
10 cc. Butyl Cellosolve
240 cc. Ethyl Alcohol
77 cc. No. 115 Suspension

This combination gave very good films which reflected between 5 and 8 percent of white light. These were even and had about the correct color, but were very soft. These films could be removed when washed with soap and hot water. Although the films possessed good optical properties they were not satisfactory, being much too soft and for that reason, not permanent.

11. In order to harden the film, a small amount of ethyl silicate was added to the suspension along with some hydrochloric acid. Many combinations were tried with the result that a suspension was obtained which gave films with excellent optical properties and at the same time were reasonably hard. The composition of this suspension is the following:

46 cc. Cellosolve
25 cc. Butanol
10 cc. Butyl Cellosolve
390 cc. Ethyl Alcohol
87 cc. No. 115 Suspension
10 cc. Ethyl silicate
1 cc. HCL

12. This suspension gives films which are even and have very good optical properties. After application the coated glass should be baked at a temperature between 100-300°C. After this treatment the films are hard enough to withstand washing with a sponge, hot water, and soap.

13. When coating plastics, it may be necessary to add a small amount of the No. 115 suspension over that required for coating glass. A temperature of about 80°C is sufficient to harden the coating in about thirty minutes. However, if the coating remains at room temperature for a few days it will harden without the application of heat. The films produced on plastic are the same as those on glass and will withstand washing with hot water and soap.

(c) Resistance of Films to Weathering

14. Glass panels coated with these films have been exposed in salt spray for four hundred hours without damaging their reflection reducing properties. At the end of this period part of the film was removed by washing and the remaining part was unchanged. There is a possibility that the glass was dirty when coated thus accounting for the portion washed off. The films on glass have also withstood 220

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hours in an accelerated weathering unit without damage.

15. In order to try to avoid the dilution of No. 115 suspension with the mixed solvents, other diluents were tried. The cellosolve butanol, and butyl cellosolve were replaced with an equal volume of alcohol. Although this combination gave films which were hard, the optical properties were not good. The reflection from film so obtained was approximately 10 percent which is twice that of the best ones prepared with other dilution. This difference may be due to a difference in surface tension in the two suspensions. In one case it may be such as to deposit a film of the correct thickness and in the other the thickness is not great enough.

(d) Surface Tension of Two Suspensions

16. The first suspension (alcohol alone) has a surface tension of 25.3 dynes per cm. and the second (mixed solvents) 27 dynes per centimeter. This difference can cause a variation in the thickness of the film deposited under the conditions used. Whether all the differences between the films produced is due to this is not known.

(e) Measurements of Reflection and Transmission

17. Light from a monochrometer was allowed to fall on the coated panels and reflected from these to a photocell. The photocell was in such a position that the light rays were as near normal to the surface as possible. An uncoated panel was substituted for the coated one at each wavelength and the ratio of the galvanometer deflections taken as the percent of light reflected. For measurements on transmission a stack of four plates was used. In this case, the light from the monochrometer was made parallel by a lens and focussed by a second lens on a slit in front of the photocell. The stacks of plates were introduced into the beam between the two lenses.

18. Results showing the amount of light reflected from a single panel and the amount transmitted by a stack of four panels are given in Plates 1 and 2. From these curves it is obvious that the amount of light reflected reaches a minimum value of 2% at 5750A° and remains at the same value to 6250A. At 5500A° the amount is only three percent. The maximum sensitivity of the eye is around 5500A° and it will disregard the higher values at each end of the curves. The low value of two percent refers to an uncoated piece of glass and represents only 0.16 percent of light reflected from two surfaces.

(f) Electron-microscope Pictures of Surfaces

19. Plates 3, 4, and 5 are pictures of the films at 15,000, 60,000 and 78,000 magnification. The first of these is on a silica background while the other two are on Formvar. Care should be taken in interpreting the latter two since there may have been some interaction between the solvents used and the supporting film. This picture shows that the film is not continuous but made up of very small particles. Some of these are certainly only 200A° in diameter and there are many which seem a

great deal smaller than this. Further study should be made on different films of varying thickness to determine if the effectiveness of the film depends on the relative amount of covered and open spaces.

20. Plates 6 and 7 are pictures of an evaporated magnesium fluoride film on silica. These show a very even film with no large irregularities as is found in the films produced here.

DISCUSSION OF RESULTS

21. The results obtained in these experiments show that it is possible to coat either glass or plastic in one operation with a reflection reducing film which is at least semi-permanent. The film will not be damaged by washing provided a sponge is used with soap and water but no abrasive. The resistance of the films to salt spray and artificial weathering is such that further tests, particularly service tests, should be made.

22. The hardening effect of the ethyl silicate may be due to two factors, a bonding of the material to the glass and the formation of a resistance polymer over the surface of the film. This action should be investigated further.

23. The solvents used as diluents in producing these films have a great bearing on their effectiveness. It is not thought that the particular combination mentioned here is the only one that will work, however, the results do indicate that some mixture is probably necessary. The solvents used do produce good results and indicate that additional work should be done with various solvents.

RECOMMENDATIONS

24. It is recommended that these coatings be given a test on instrument panels.

25. It is also recommended that the project be continued with the object of making the film extremely hard perhaps approaching the hardness of glass itself.

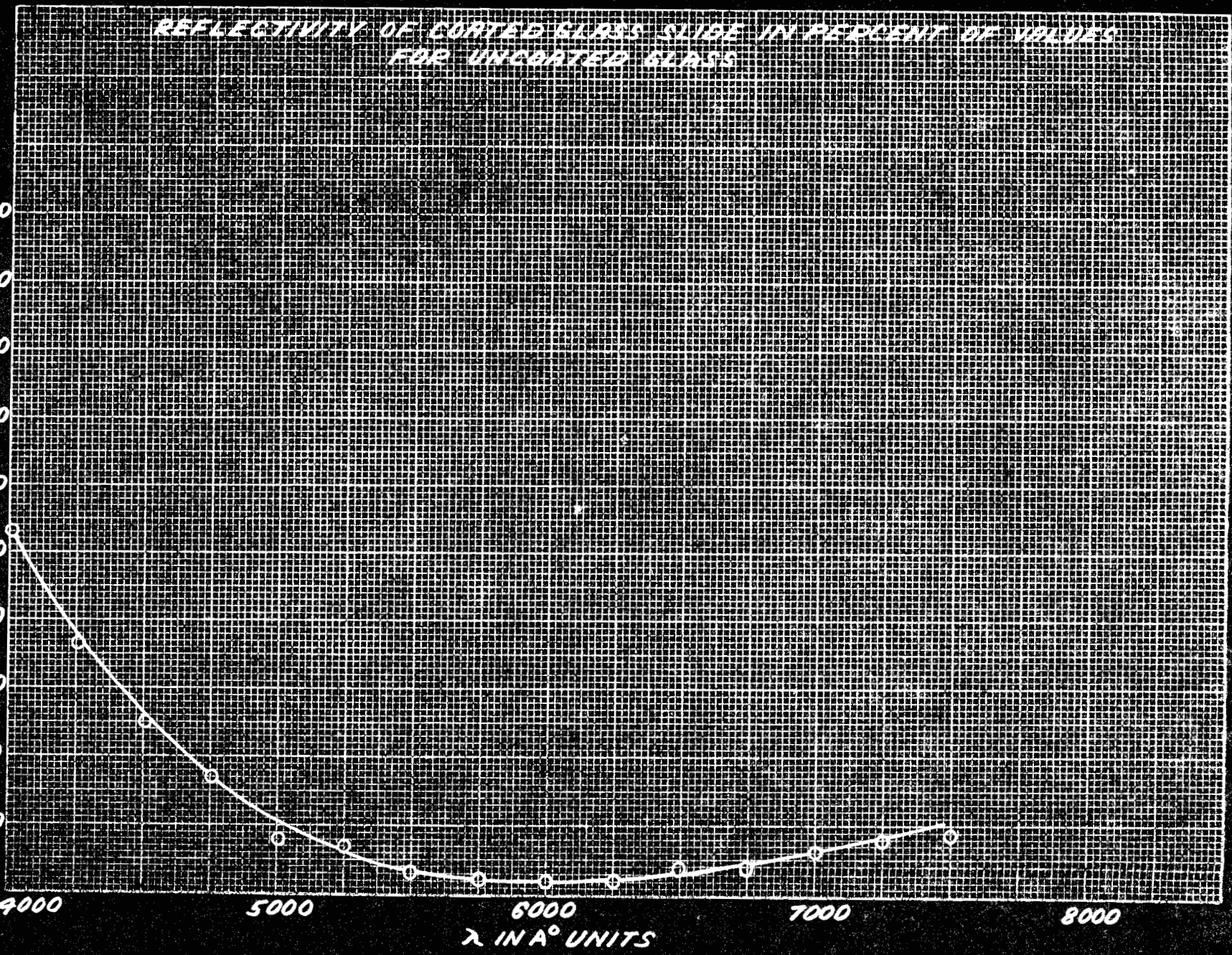


PLATE I

U.S. NAVAL RESEARCH LABORATORY
BELLEVUE, D. C.

DATE

N. R. L. 34A

U. S. NAVAL RESEARCH LABORATORY
BELLEVUE, D. C.

DATE

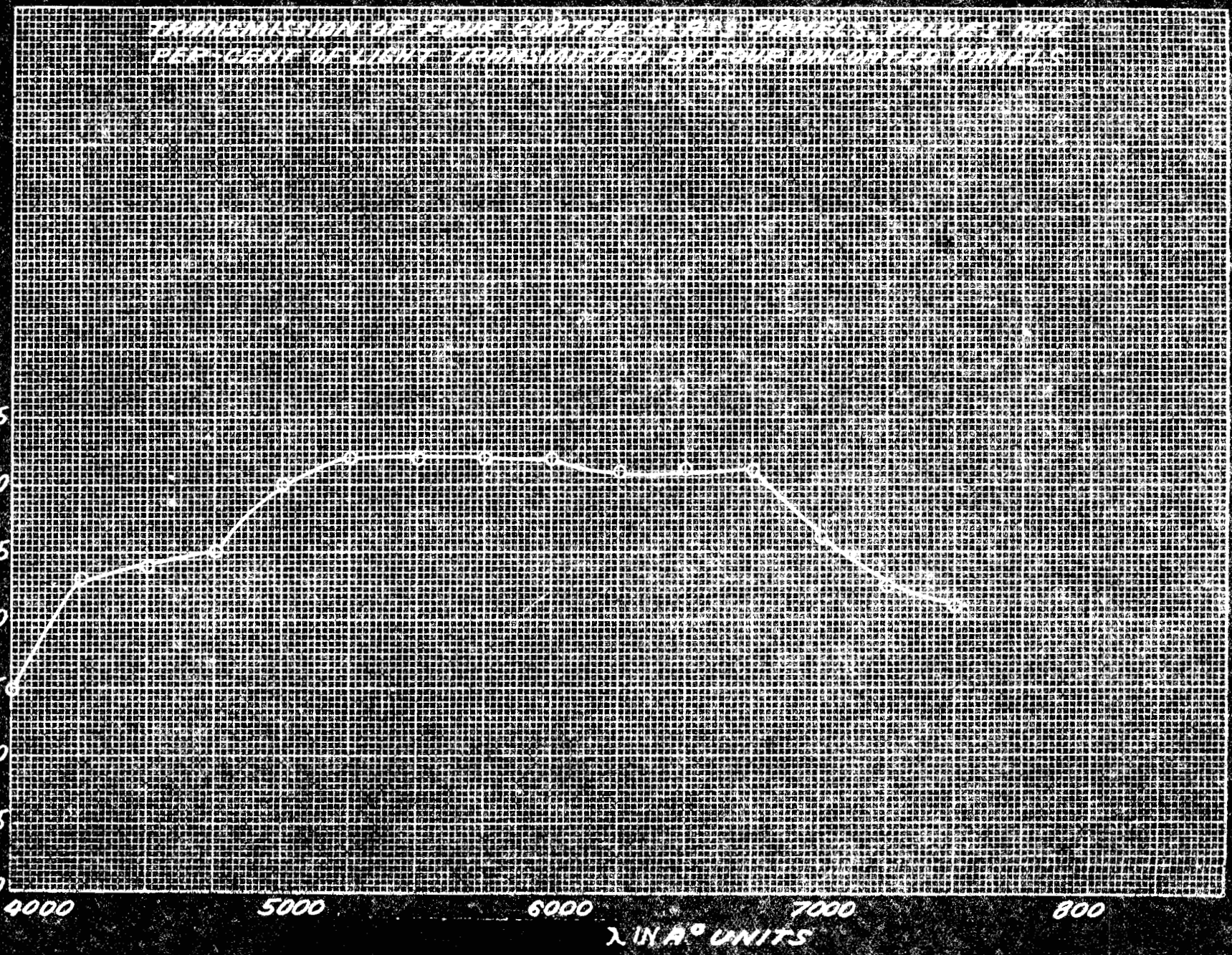
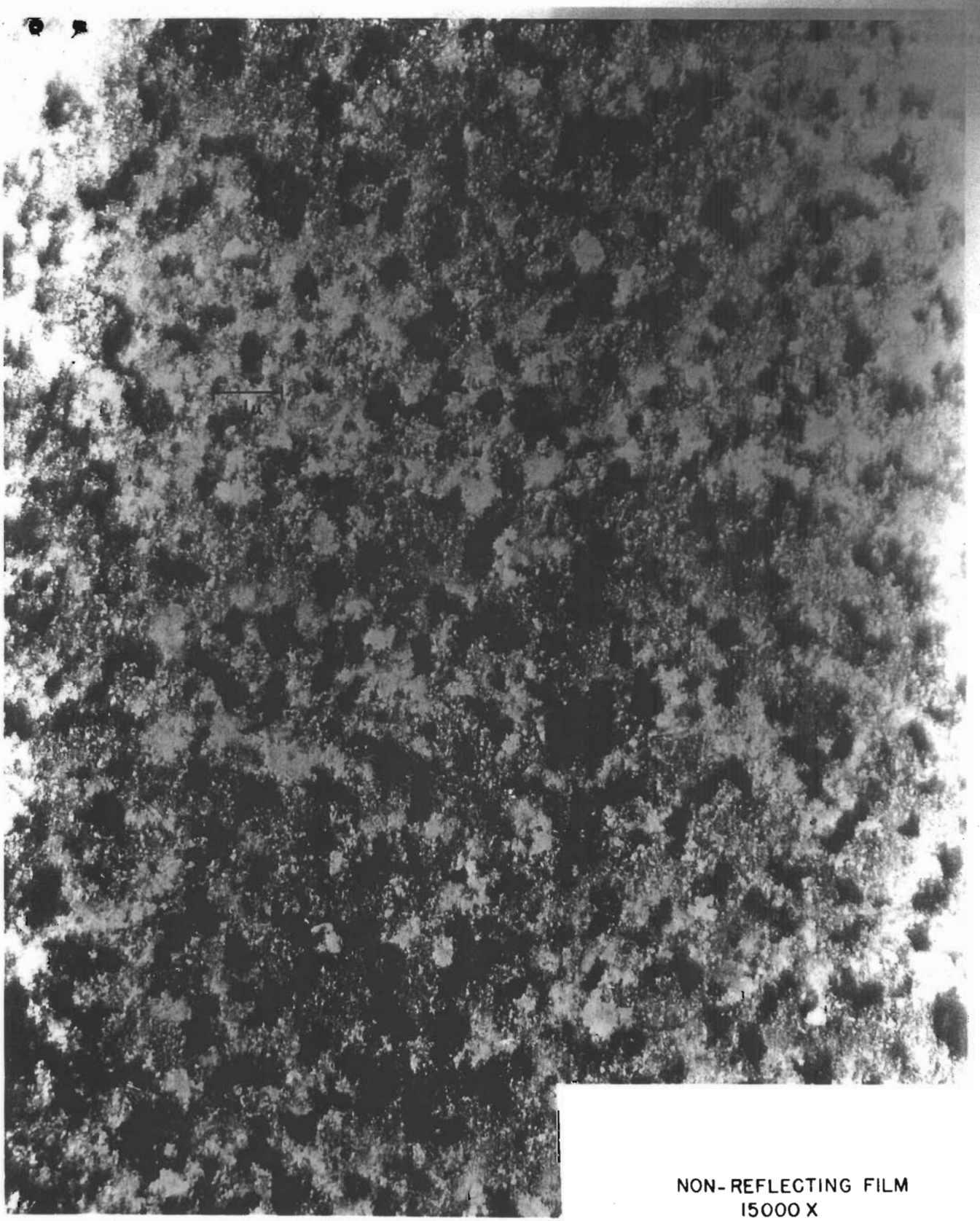


PLATE 2



NON-REFLECTING FILM
15000 X

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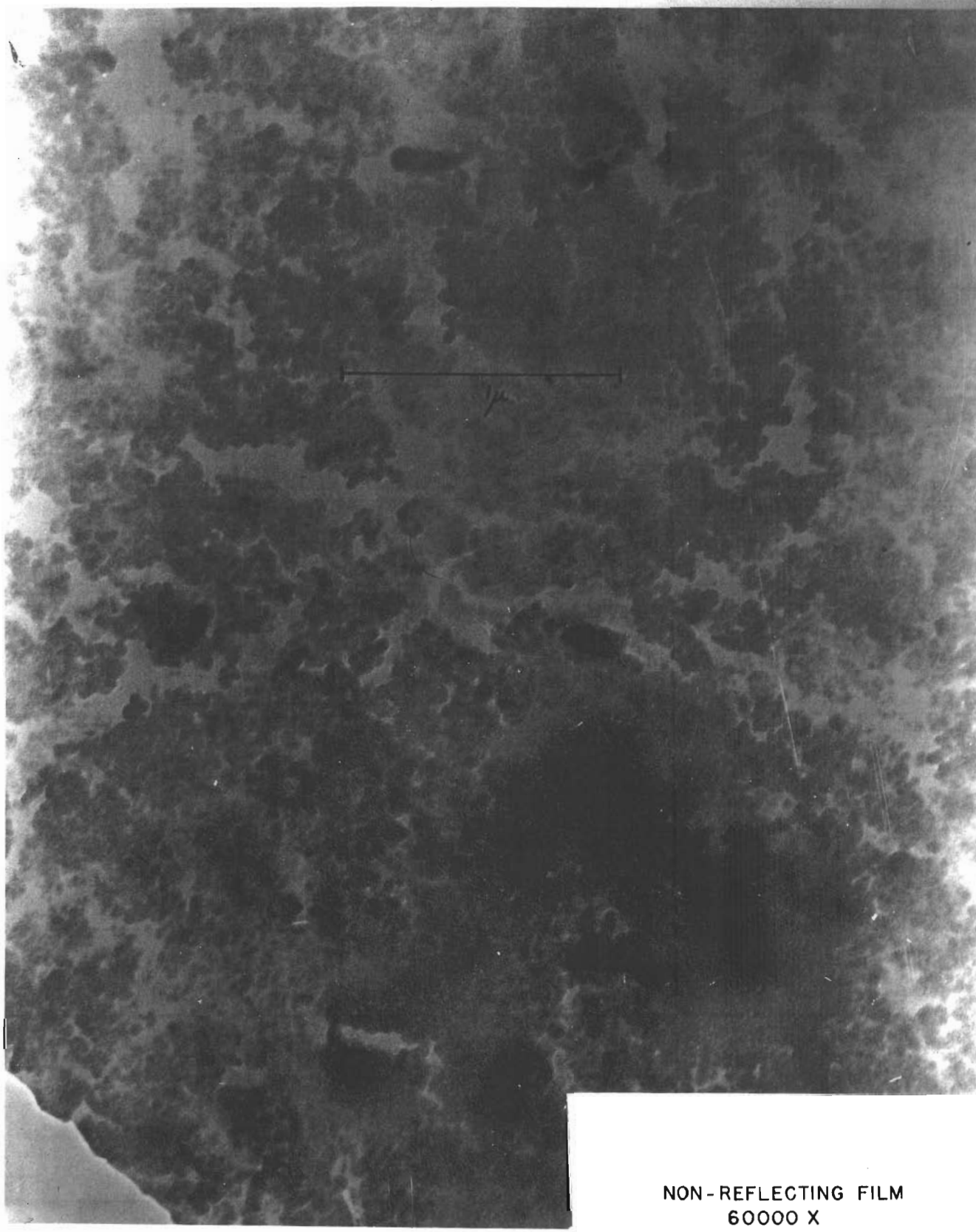

NON-REFLECTING FILM
60000 X

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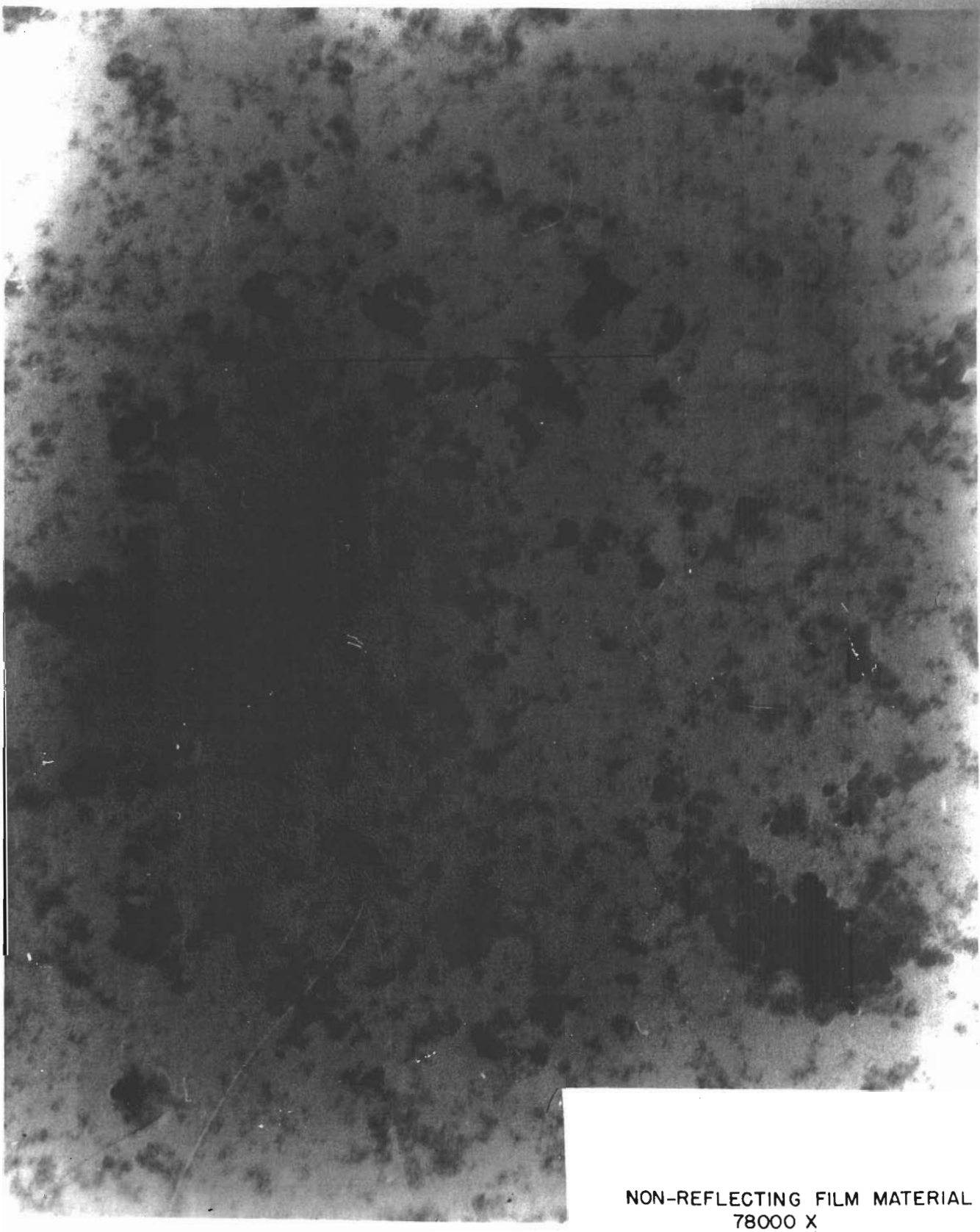

NON-REFLECTING FILM MATERIAL
78000 X

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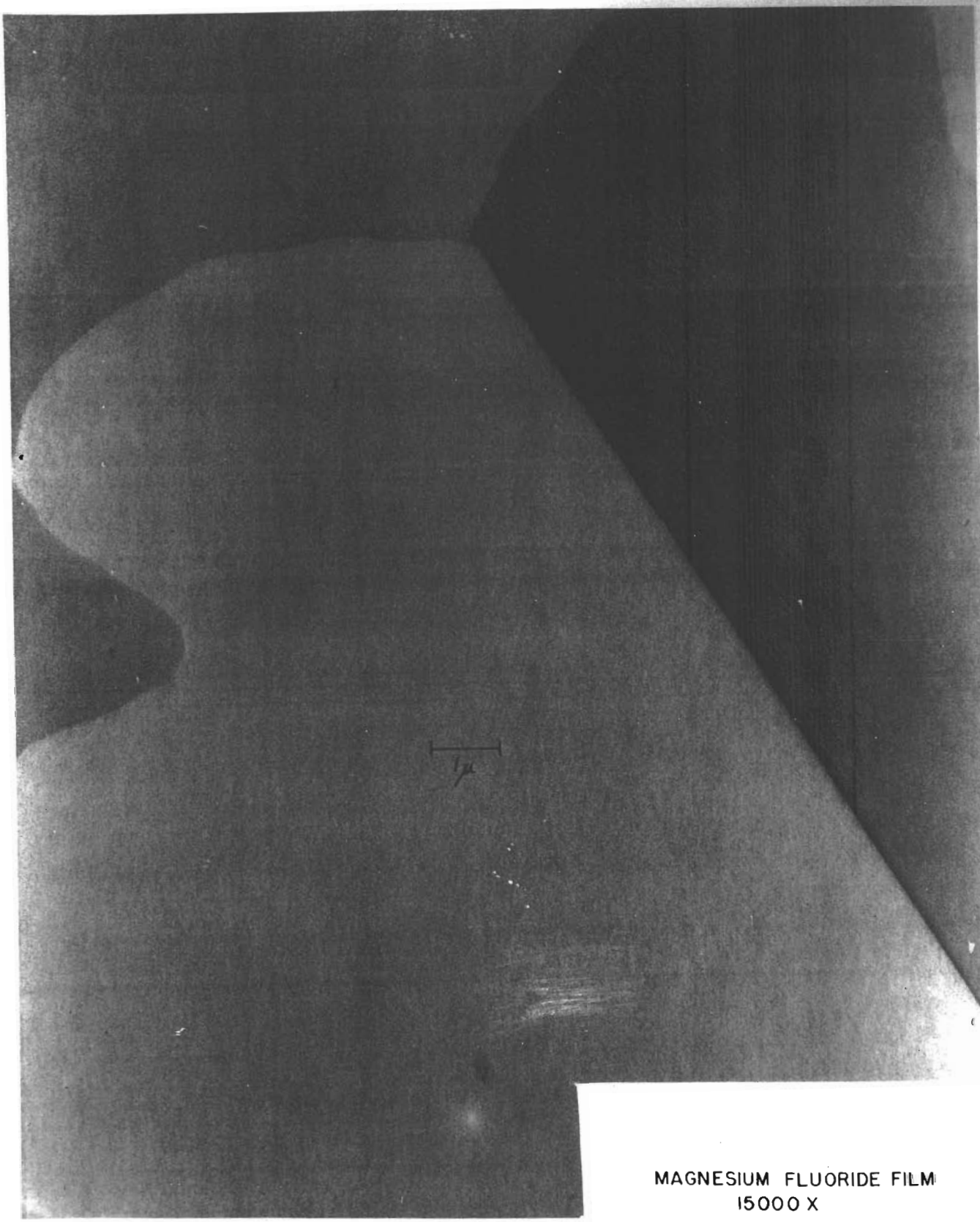
PLATE 5



MAGNESIUM FLUORIDE FILM
15000 X

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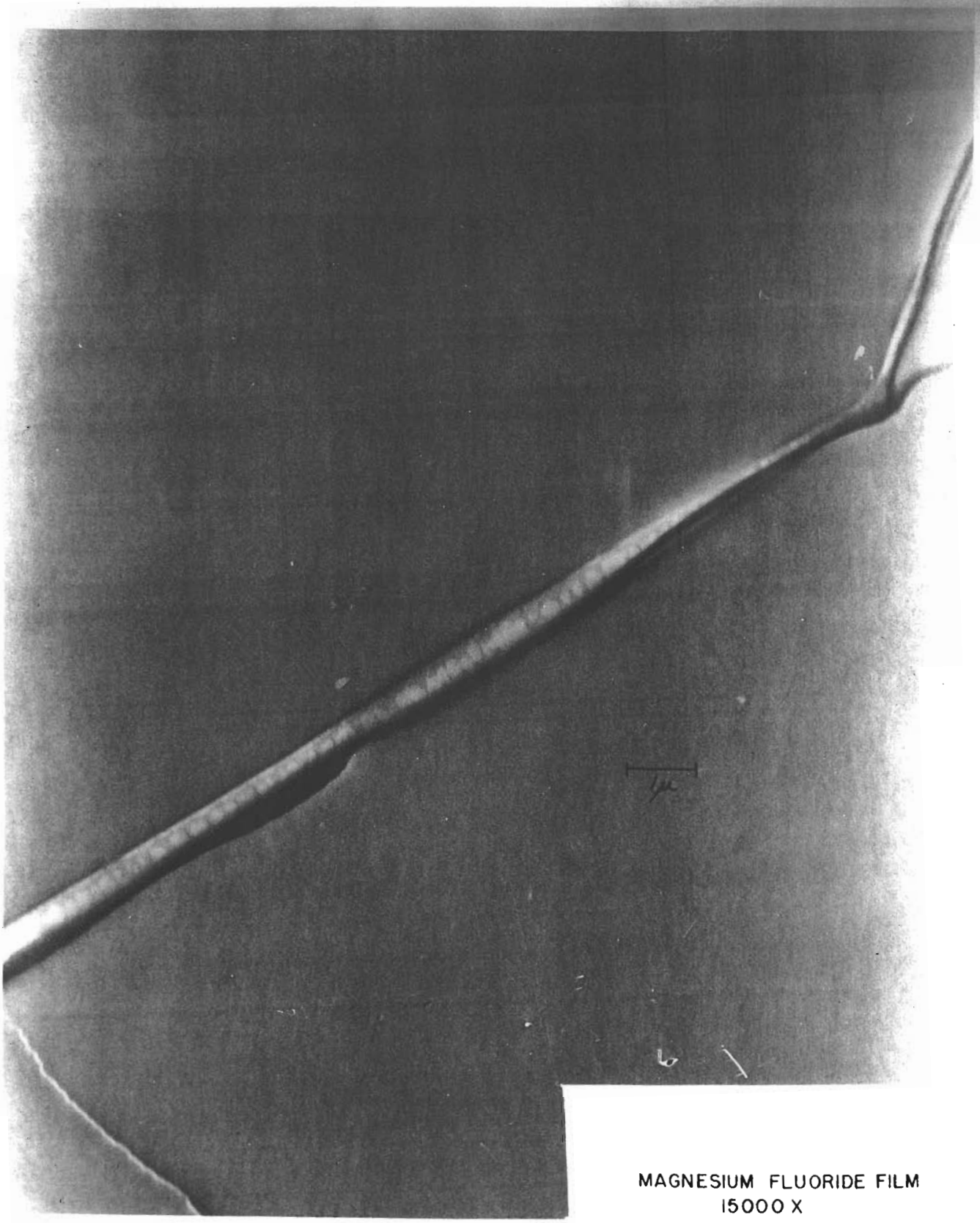

MAGNESIUM FLUORIDE FILM
15000 X

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