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EVALUATION OF A FLASH X-RAY TECHNIQUE FOR RECESSION MEASUREMENTS IN ABLATIVE MATERIALS

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16. Abstract

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SUMMARY

A flash X-ray technique has been developed that permits the determination of the surface and interface recessions of ablative materials during testing in arc-jet facilities. Use is made of a system with which several radiographs of a specimen can be made during a single test. The flash X-ray system and measurement techniques are discussed. Surface and interface recessions are obtained for three charring ablators tested in an arc-jet facility. The data obtained with the X-ray system are compared with results obtained by direct measurement on sectioned specimens after testing and with analytical predictions. The agreement among the results is good. Thus the X-ray technique can be used effectively to obtain reliable recession measurements. For the phenolic-nylon materials tested, the char density increases significantly toward the front surface. An epoxy-base material tested shows no char density gradient. Also, there appears to be a reduction in density of the charring materials before any visible change takes place.

INTRODUCTION

Vehicles reentering the earth's atmosphere are commonly protected from the heating environment by various types of ablative materials. Since it is not practical to flight test each promising material, the ablative materials are usually evaluated from test results obtained in simulated reentry environments produced by arc-heated test facilities. To evaluate the performance of materials in these environments, it is necessary to know the front surface recession and the recession of the interface between the charred and uncharred material during a test. In the past, the surface and interface recessions have been determined by testing several specimens in the same environment for various periods of time. (See ref. 1, e.g.) At the end of each test, the specimen is sectioned, and the locations of the front surface and the interface are determined by direct measurements. This procedure is time consuming and costly because a minimum of four tests is usually required to obtain sufficient recession data. Also, uncertainties in the measurements could arise because of possible variations in material properties among the test specimens, the inability to duplicate the test conditions in the separate

tests, and the difficulty of actually locating the interface by observing the change in physical appearance of the materials (color, structure, etc.).

A technique has been developed which reduces the magnitude of some of these uncertainties and eliminates others. The technique makes use of a flash X-ray system with which several radiographs of a specimen can be made during a single test. This paper describes the measurement techniques and the flash X-ray system and gives some results obtained with the system. Surface and interface recessions were obtained for three ablative materials tested in an arc-jet facility. The data obtained with the X-ray system are compared with results obtained by direct measurement on the specimens and with analytical predictions.

The units used for the physical quantities in this paper are given in the International System of Units (SI). (See ref. 2.)

APPARATUS

X-Ray Source

The X-ray source is a cold-cathode X-ray tube. X-rays are produced when electrons, accelerated by a high-voltage pulse applied between the cathode and the anode, strike the anode. The apparatus used for this study can be operated with tube voltages ranging from 150 to 300 kV at currents of 2500 to 5000 A, respectively. The pulse duration is about 18 ns. The very short exposure time is necessary to permit the recording of the dynamic processes at any given time. The source can be fired at rates up to 10 pulses per second for 10 pulses. At lower pulse rates, the total number of pulses can be increased. A photograph of the X-ray source, the high-voltage pulser, and the control panel is shown in figure 1.

Film Transport System

The number of radiographs taken during a test is limited to the number of film plates which can be appropriately positioned near the specimen during the test. Two types of film transport systems have been used with good results. One is a 10-frame system, described in reference 3; the other, used for the present tests, is a four-frame system. This apparatus has four 20- by 25-cm film cassettes arranged in a box configuration (fig. 1) and is rotated at 1 rad/s during a test. One to four film sheets can be irradiated during a single rotation of the transport assembly. The interval between radiographs can be varied by allowing the transport assembly to rotate any desired number of times before making a radiograph. Intensifying screens, placed in contact with the film, convert the X-rays to visible light and thus increase the film sensitivity.

Film Readout System

A recording microphotometer is used to obtain recession data from the radiographs. As the film is passed by the slit of the microphotometer, relative film transmission is recorded as a function of distance along the film from some reference point. The slit width is adjustable from 0 to 50 μm, and the slit length is adjustable from 0.5 to 1.2 mm. The film rate across the slit can be varied from 0.001 to 0.4 mm/s.

TEST SPECIMENS AND CONDITIONS

To evaluate the flash X-ray technique for recession measurements, three charring ablators were tested in apparatus C of the Langley entry structures facility described in reference 4. All specimens were flat, circular disks 7.6 cm in diameter. The materials tested were a high-density (1200 kg/m^3) phenolic-nylon, a low-density (550 kg/m^3) phenolic-nylon, and an epoxy-base material in a fiber-glass honeycomb; these materials were exposed to heating rates of 1.4, 0.7, and 0.45 MW/m^2, respectively, in a test stream of 3 percent oxygen and 97 percent nitrogen.

TEST SETUP AND PROCEDURES

A schematic diagram of the test setup is shown in figure 2. The X-rays are directed toward the specimen and the film. The lead shield absorbs X-rays not emitted directly toward the specimen. Thus, fogging of the film by X-rays which may be scattered onto the film from the surrounding apparatus is prevented. The specimen absorbs some fraction of the X-rays, and an image is produced on the film located behind the specimen. A photograph of the arc-jet facility with a specimen and the film transport in position is shown in figure 3.

The resolution of the system depends upon the source-to-film distance and the specimen-to-film distance. Good results have been obtained with a source-to-film distance of about 4.6 meters and a specimen-to-film distance of 30 cm. Perhaps better results could be obtained with a shorter specimen-to-film distance. However, if this distance is too short, the rather large heat outputs from the arc-jet stream and the specimen could damage the film or the film cassette.

The usual procedure is to make one radiograph of the specimen in the test position before the test, four at various times during the test, and one after the test. The exposed film is analyzed with the use of the microphotometer to obtain surface and interface recessions. After each test the specimen is sectioned and the locations of the surface and interface are determined by direct measurements.

DATA ANALYSIS

It is well known that materials absorb X-rays in amounts depending upon their densities and that, upon heating, most ablative materials undergo a considerable reduction in density. Therefore, in general, it is possible to differentiate between materials of different densities by examining radiographs of the materials and, in particular, to differentiate between charred and uncharred ablative materials. Consequently, surface and interface movements can be determined by analyzing radiographs of ablative materials taken at various times during a test.

Each radiograph is placed on the microphotometer, and a plot of relative film transmission against distance along the film is obtained. The microphotometer scale is set to include the range of film densities of interest. The radiographs are scanned along a line through the center of the specimen from the stagnation point to the metal mounting sting. Corresponding reference points are located on each radiograph to insure that the same line of scan is followed and that all measurements are made at the same locations on each specimen image.

The points on the microphotometer trace corresponding to the surface and interface are located simply by visual inspection of the trace. Rapid changes in relative film transmission are indications of the locations of the surface and interface. Measurements are made from these points to an appropriate reference point.

One factor which must be considered in all cases is the slight magnification of the specimen image due to the diverging X-rays and the fact that the film is some distance from the specimen. The magnification is easily accounted for by making a direct measurement on the film of some known distance on the specimen. The ratio of the distance as measured on the film to the actual distance is the magnification factor.

ANALYTICAL PROCEDURES

The equations governing the transient response of ablative materials to a heating environment have been programed for numerical solution on a high-speed digital computer. (See ref. 5.) The computer program yields temperature distributions within a material as well as dimensional changes of the material as functions of time for a material subjected to a heating environment. This program was used to calculate the surface and interface recessions for the materials used in this study. The results of the calculations are compared with the experimental results obtained from the X-ray data and from measurements made directly on the sectioned specimens after testing. Values for the thermal conductivity of high-density phenolic-nylon char and the specific heat of the gases of pyrolysis were obtained from reference 6. Other material property values used in the calculations are from references 7, 8, and 9.

RESULTS AND DISCUSSION

Figure 4 shows a series of six radiographs of a high-density phenolic-nylon specimen taken before the test, at various times during the test, and 300 seconds after the test. Also shown are the corresponding microphotometer traces. Corresponding points on the traces and on the radiographs are labeled; point A denotes the front surface and point B denotes the interface. In figure 4(a) (before test), the front surface and interface are, of course, at the same location. In all cases, the location of the front surface can be identified by the rapid increase in relative film transmission from the initial value. Although the locations of the points corresponding to the front surface appear to be somewhat arbitrary, good results are obtained if the points are chosen with consistency and with some thought to the physical situation.

For test times less than 10 seconds, it is somewhat difficult to locate the interface. However, definite changes in the slope of the trace occur as the front surface and interface are passed. (See fig. 4(b).) In this case, the inflection point on the trace is used for the interface location. This procedure has produced good results.

For test times greater than about 10 seconds (figs. 4(c) to 4(f)), the interface is easily identified as a low point in the relative film transmission, which corresponds to a low-density point in the material. Although a low-density char develops at pyrolysis, a significant amount of carbon is deposited in the char as a result of pyrolysis gas reactions as the gases flow through the char toward the surface. (See ref. 10.) This process accounts for the increase in char density as the surface is approached.

From the microphotometer traces in figure 4, it would appear that the char density attains a maximum value (corresponding to a peak in film transmission) at some small distance from the front surface. However, this behavior cannot be confirmed with the present data because even for the untested specimen (fig. 4(a)) the microphotometer trace does not have an infinite slope at the front surface as it theoretically should. The finite slope of the trace between the peak and the front-surface location may be caused by any one or a combination of several factors. For example, the X-ray source is not a point source, and since the film is some distance from the test specimen, some fuzzing of the edges results. Also, the intensifying screens used with the film, as well as the film itself, have some finite resolution associated with them. Additional factors to be considered are the change in shape of the specimen front surface from flat to slightly convex and the rounding of the corners of the specimen due to the test environment. As the specimen surface becomes curved, the X-rays have less material to traverse near the front surface, and fewer X-rays are absorbed. Therefore, the film transmission decreases as the front surface is approached.

The traces in figures 4(a), 4(d), and 4(f) are superposed in figure 5 to illustrate the recession of the front surface and interface with increasing time. No attempt was made

to determine material density from the heights of the curves in this figure. Although it may be possible to correlate relative film transmission with material density and obtain quantitative results, great care would be required in exposing, developing, and analyzing the film. The change in specimen shape with time, as well as apparent density changes as a result of charring on the sides of the specimen, would also have to be taken into account. Nevertheless, such a technique would be quite beneficial.

A typical radiograph and microphotometer trace of a low-density phenolic-nylon specimen are shown in figure 6 for a test time of 58 seconds. The trace is similar to those for the high-density phenolic-nylon specimen; again the low-density point associated with the interface is shown.

A post-test radiograph and corresponding microphotometer trace for the epoxy-base material are shown in figure 7. The honeycomb material can be readily observed in the radiograph. The trace shows that the char density is apparently fairly constant throughout its thickness, and a low-density point at the interface is not present in this material.

Figure 8 shows a comparison of the X-ray recession data with post-test direct measurements on the specimens and with the analytical data. For the front-surface locations of the high-density phenolic-nylon and epoxy-base specimens, the X-ray measurements agree very well with the direct measurements. (See figs. 8(a) and 8(c).) Since no post-test X-ray was made for the low-density phenolic-nylon, no comparison is made between the X-ray and direct measurements in figure 8(b). The analytical data for the high- and low-density phenolic-nylon surface recessions differed slightly from the measurements. The epoxy-base material was tested at a very low heating rate (about 0.45 MW/m^2), and there was virtually no surface recession. In this case, the experimental and analytical data agreed exactly.

For the interface measurements, the agreement between X-ray data and the analytical results is excellent. The direct measurement indicates an interface recession slightly smaller than that obtained from the X-ray data. This difference occurs because the direct measurement involves locating the interface visually on the specimen by observing the change in physical appearance of the material (color, structure, etc.), whereas the X-ray technique locates the interface essentially by the material density at that point. Therefore, there may be a considerable reduction in density of the material when heated before any visible change takes place.

Post-test X-rays were taken about 300 seconds after the tests had ended. The fact that the interface recession at this time is about the same as that predicted at the end of the test (120 seconds) may be an indication that there was negligible pyrolysis of the material after the test had ended.

CONCLUDING REMARKS

The front surface and interface recessions of three charring ablator specimens tested in an arc-jet facility are determined with the use of a flash X-ray system. The measurement technique makes use of a system which can produce several radiographs of a specimen during a single test. Results from the X-ray measurements are compared with analytical results and with measurements made directly on sectioned specimens after testing. The results of the measurements are in good agreement and demonstrate that the flash X-ray technique can be used effectively to obtain reliable recession data.

Although no attempts are made to obtain quantitative density measurements, a number of significant observations are made concerning the materials tested. Microphotometer traces of radiographs of high- and low-density phenolic-nylon indicate that the char density increases significantly toward the front surface. This observation reflects the deposition of carbon by the pyrolysis gases as they flow through the char. For an epoxy-base material in a low-heating-rate environment, the char density appears to be nearly constant throughout its depth.

Comparison of X-ray data with post-test measurements indicates that there may be a reduction in density of the materials before any visible change takes place. Therefore, care must be taken in attempting to locate the interface by visual inspection of specimens after testing.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., December 18, 1969.

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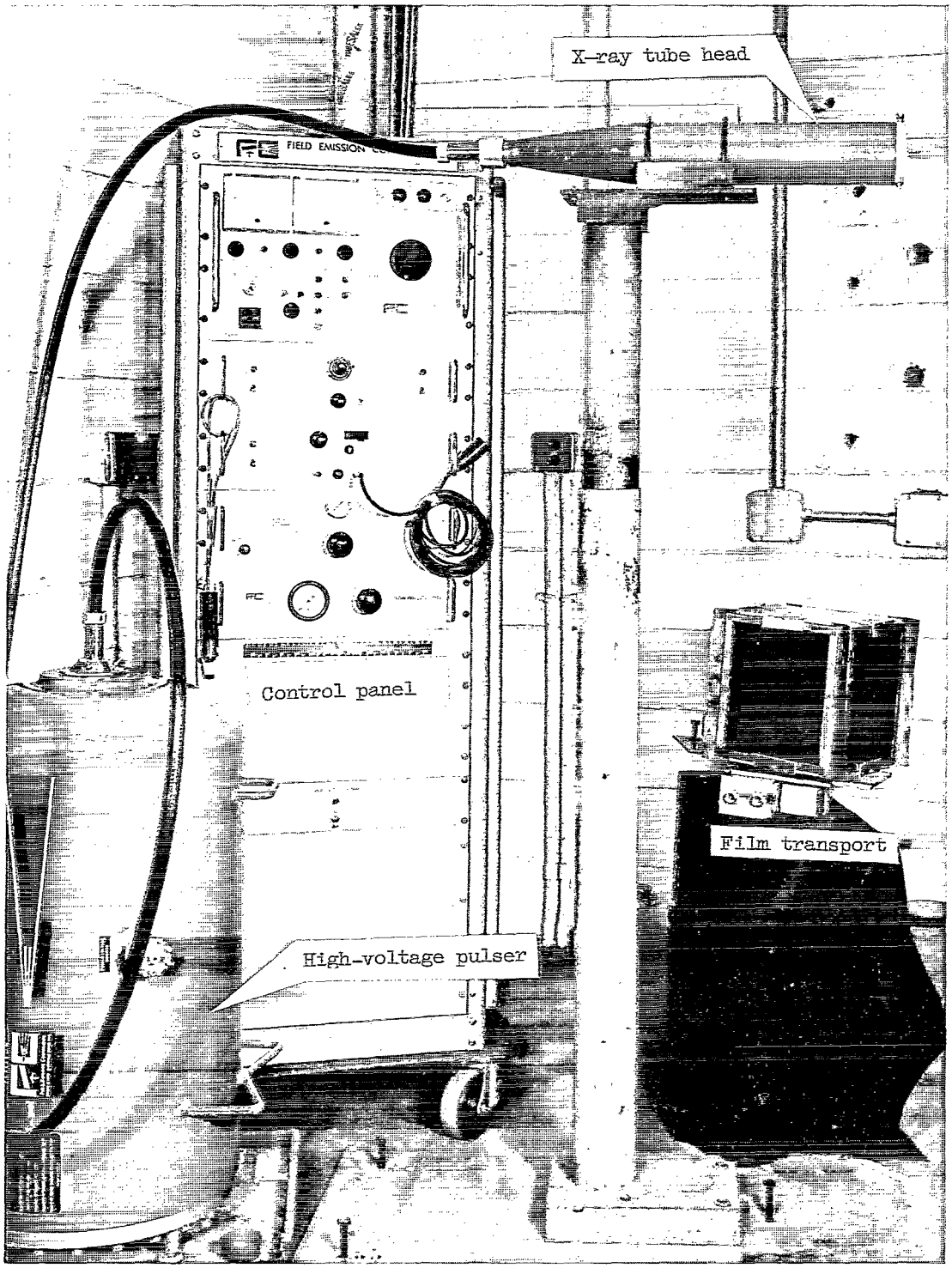


Figure 1.- Flash X-ray equipment. L-67-3809.1

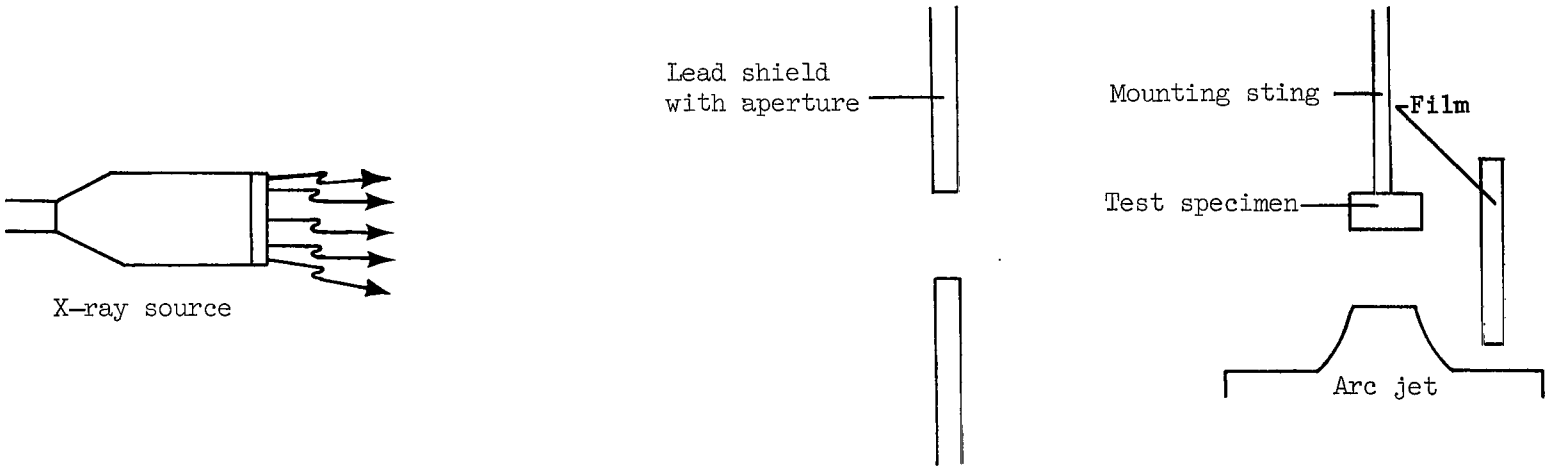


Figure 2.- Typical test setup.

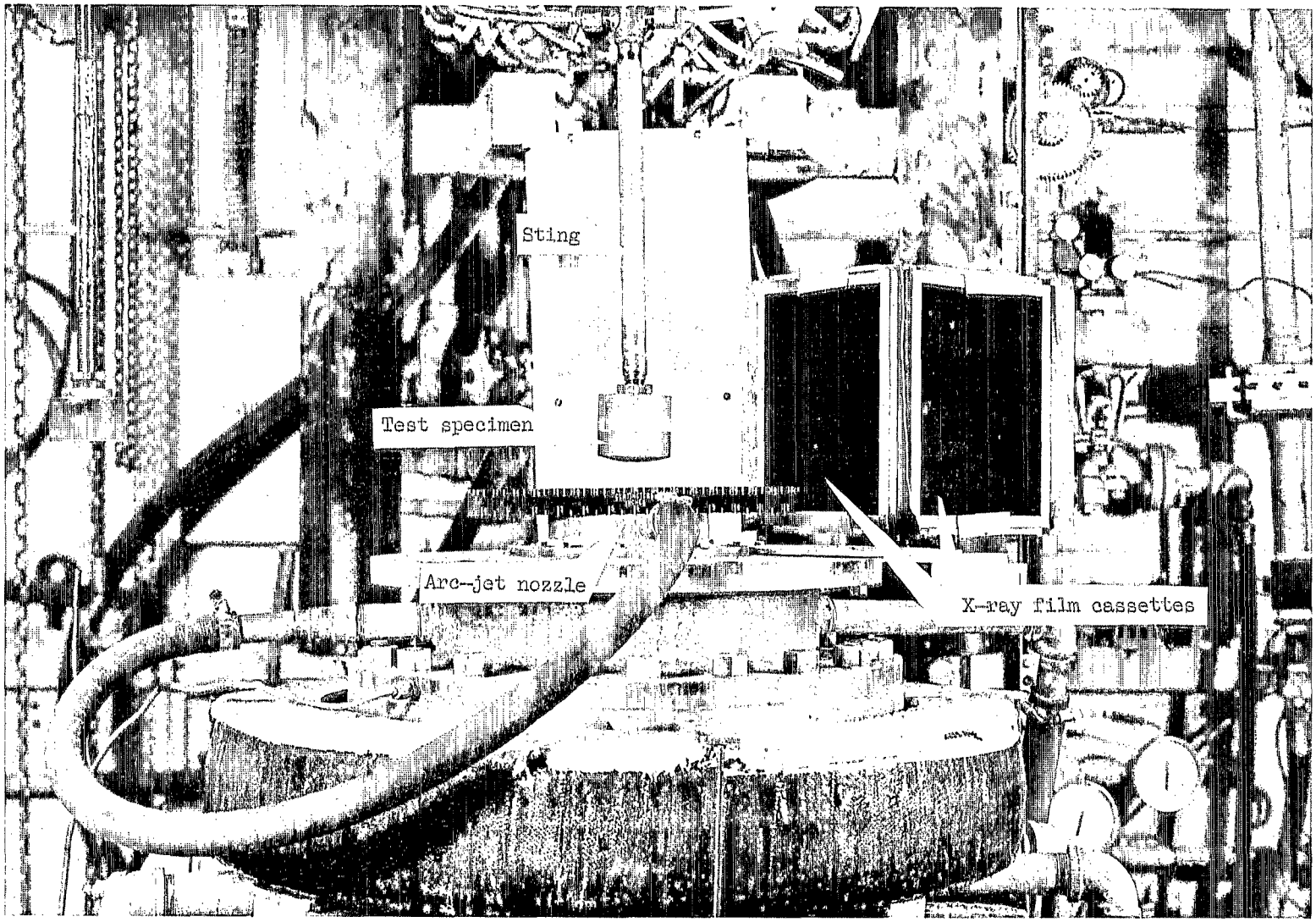
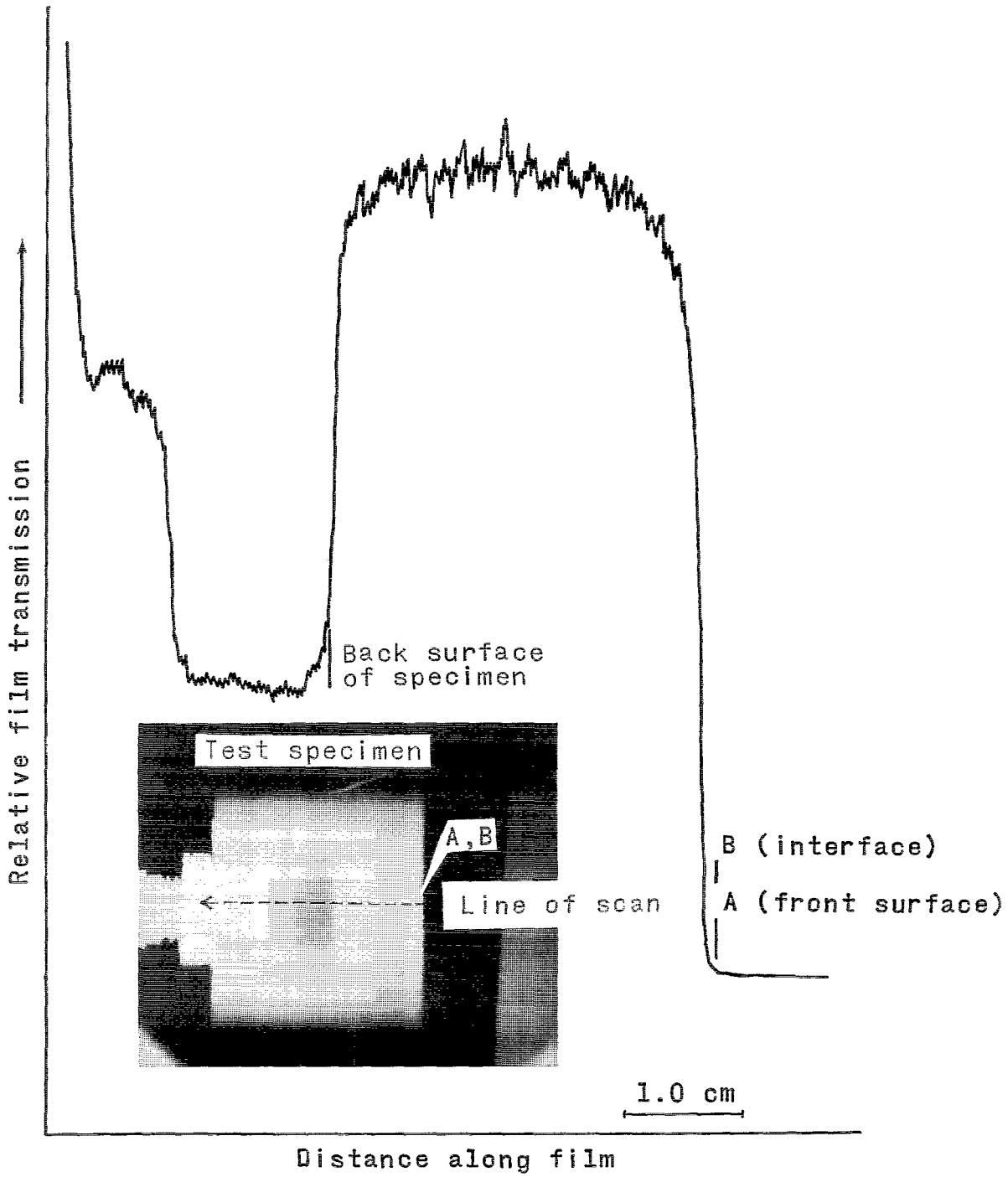


Figure 3.- Arc-jet facility with four-frame film transport system. L-67-3810.1



(a) Before test.

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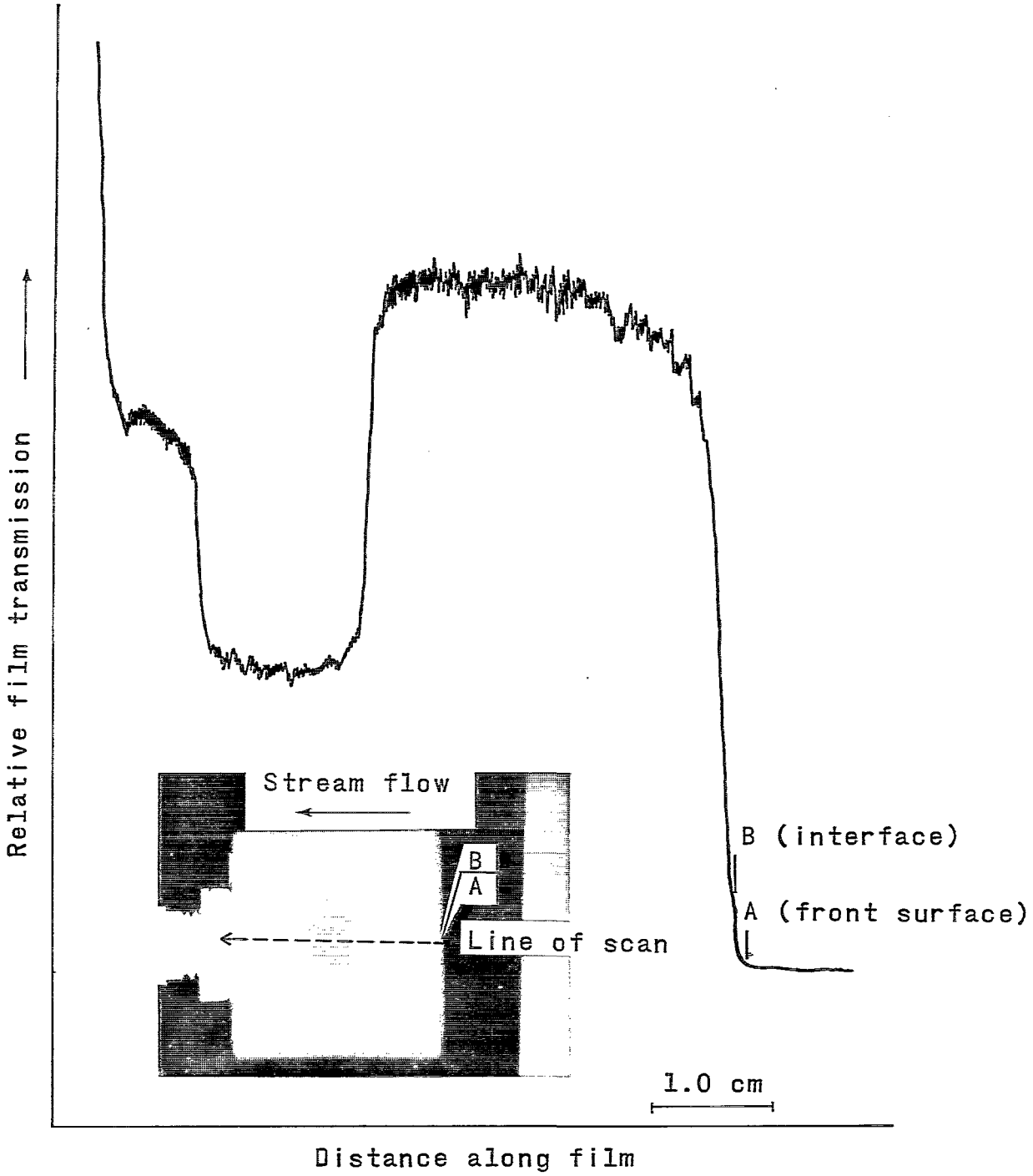
Figure 4.- Radiograph and corresponding microphotometer trace of high-density phenolic-nylon specimen.



(b) Test time, 8 seconds.

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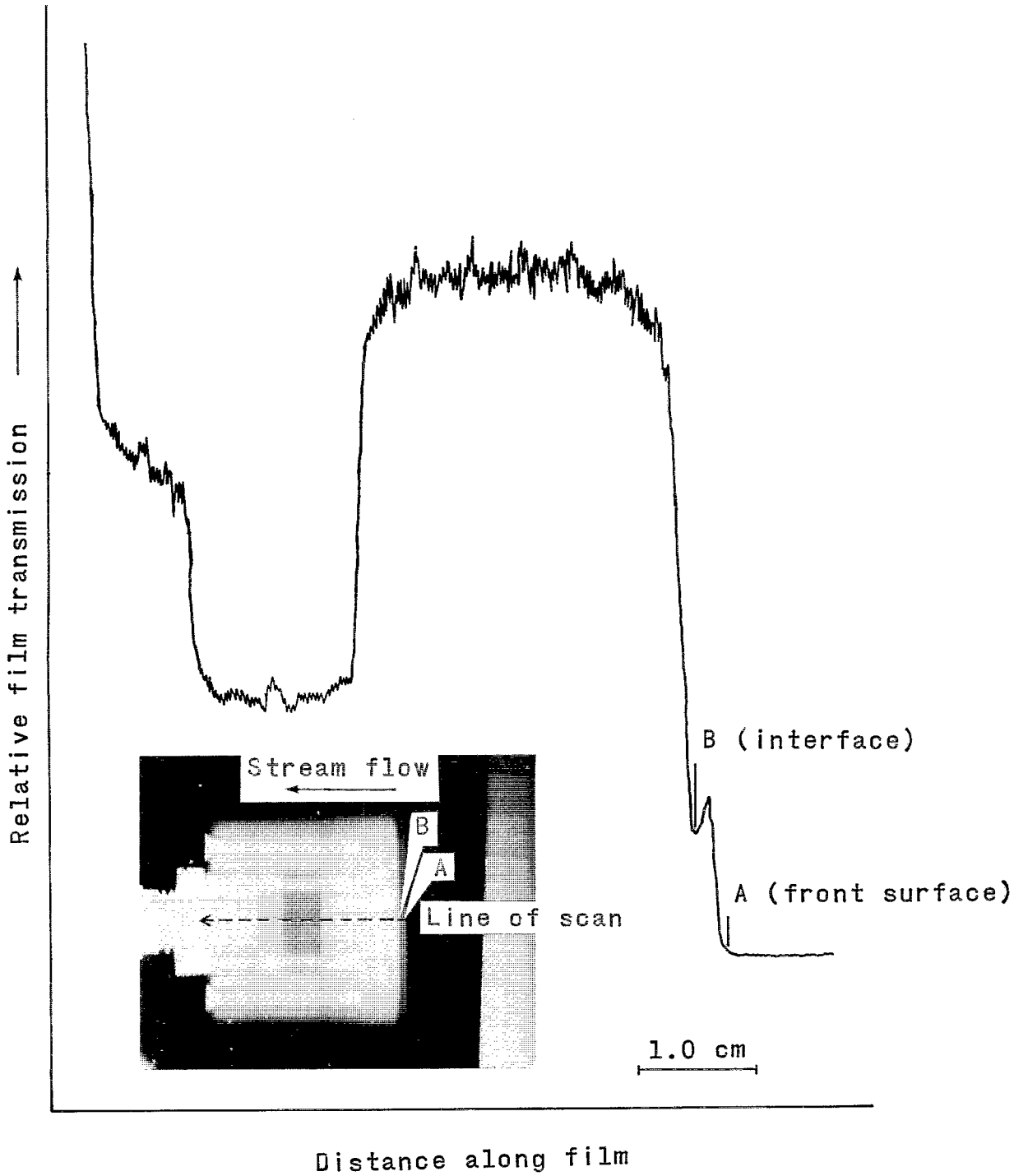
Figure 4.- Continued.



(c) Test time, 30 seconds.

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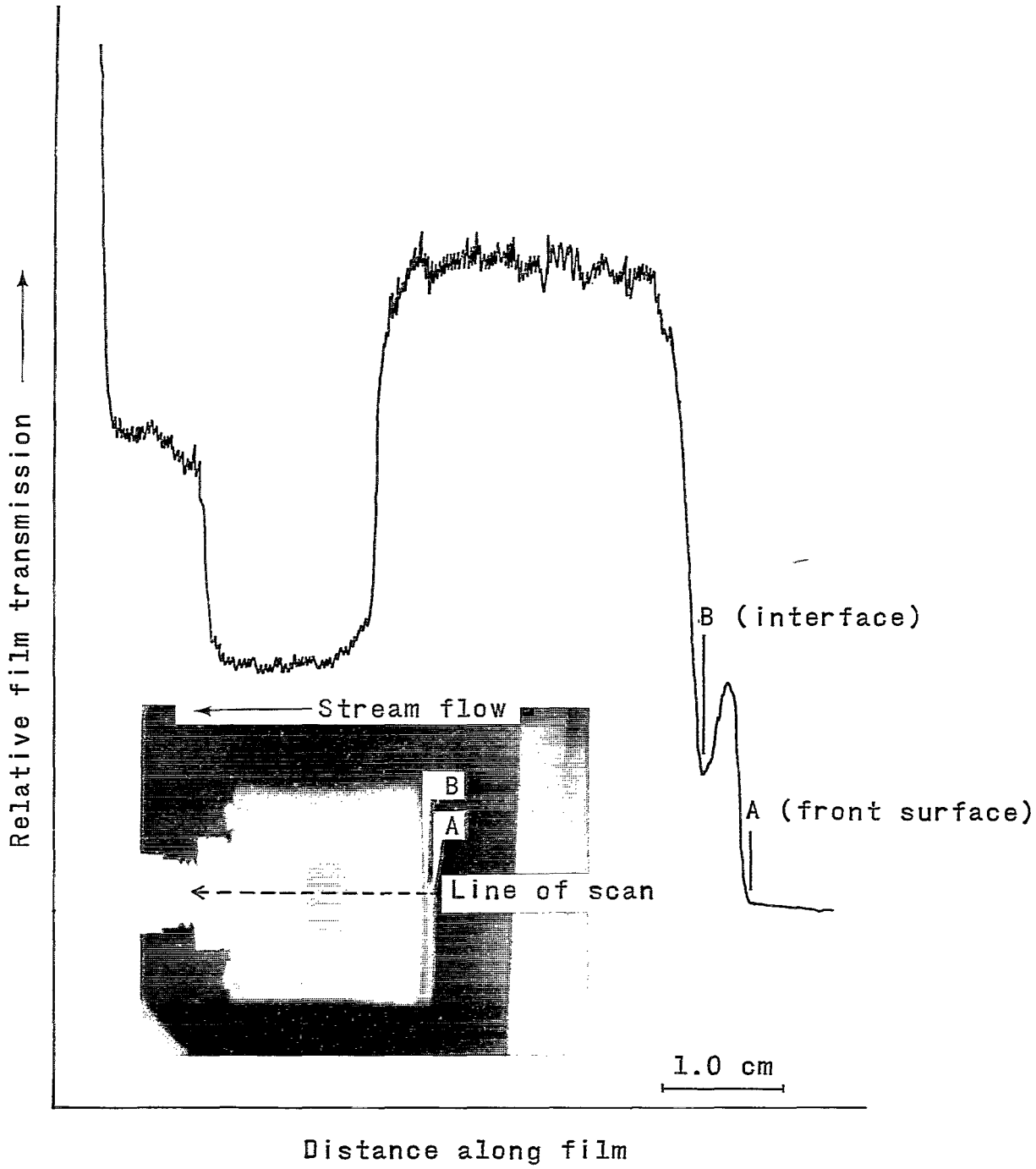
Figure 4.- Continued.



(d) Test time, 57 seconds.

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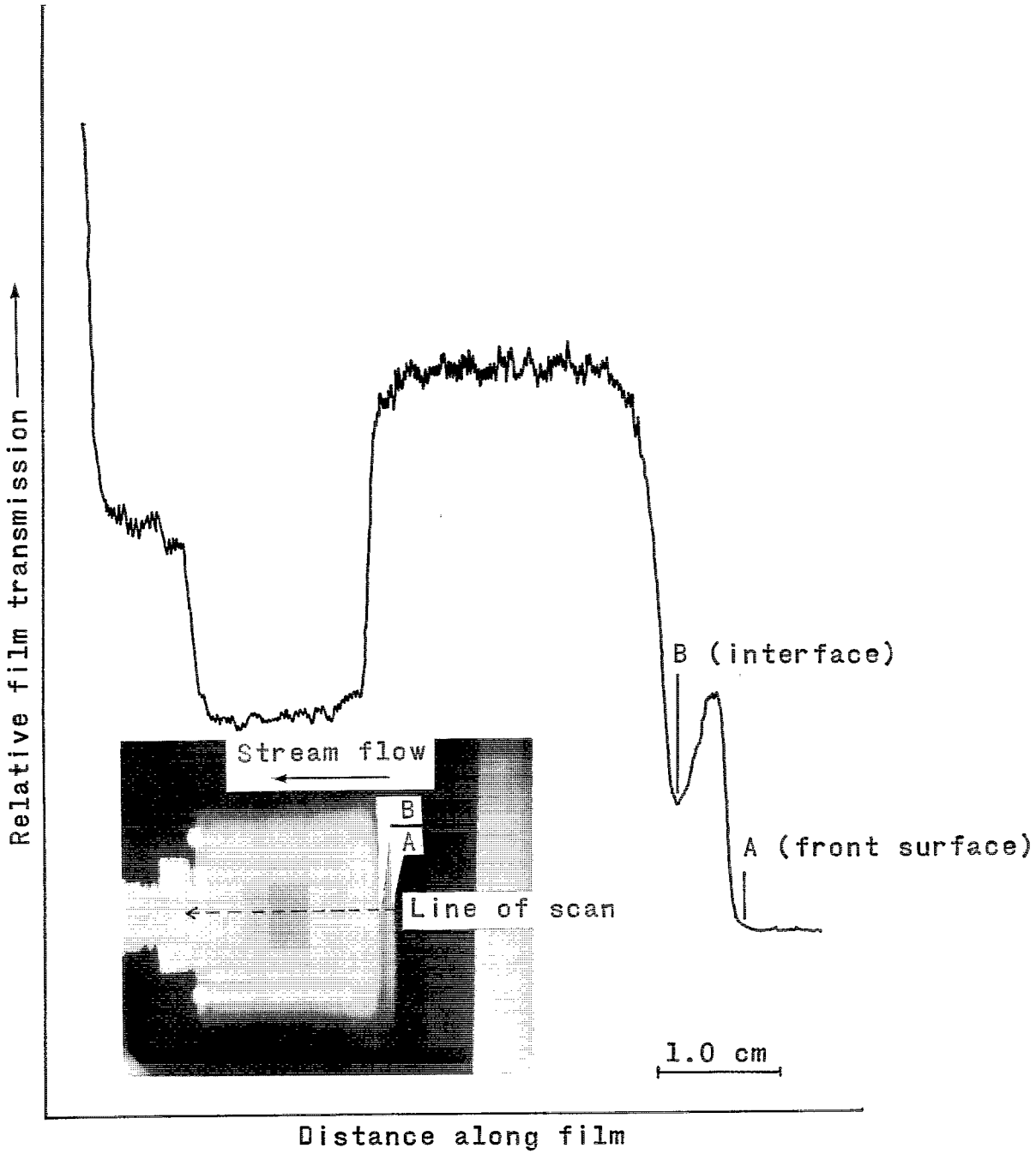
Figure 4.- Continued.



(e) Test time, 85 seconds.

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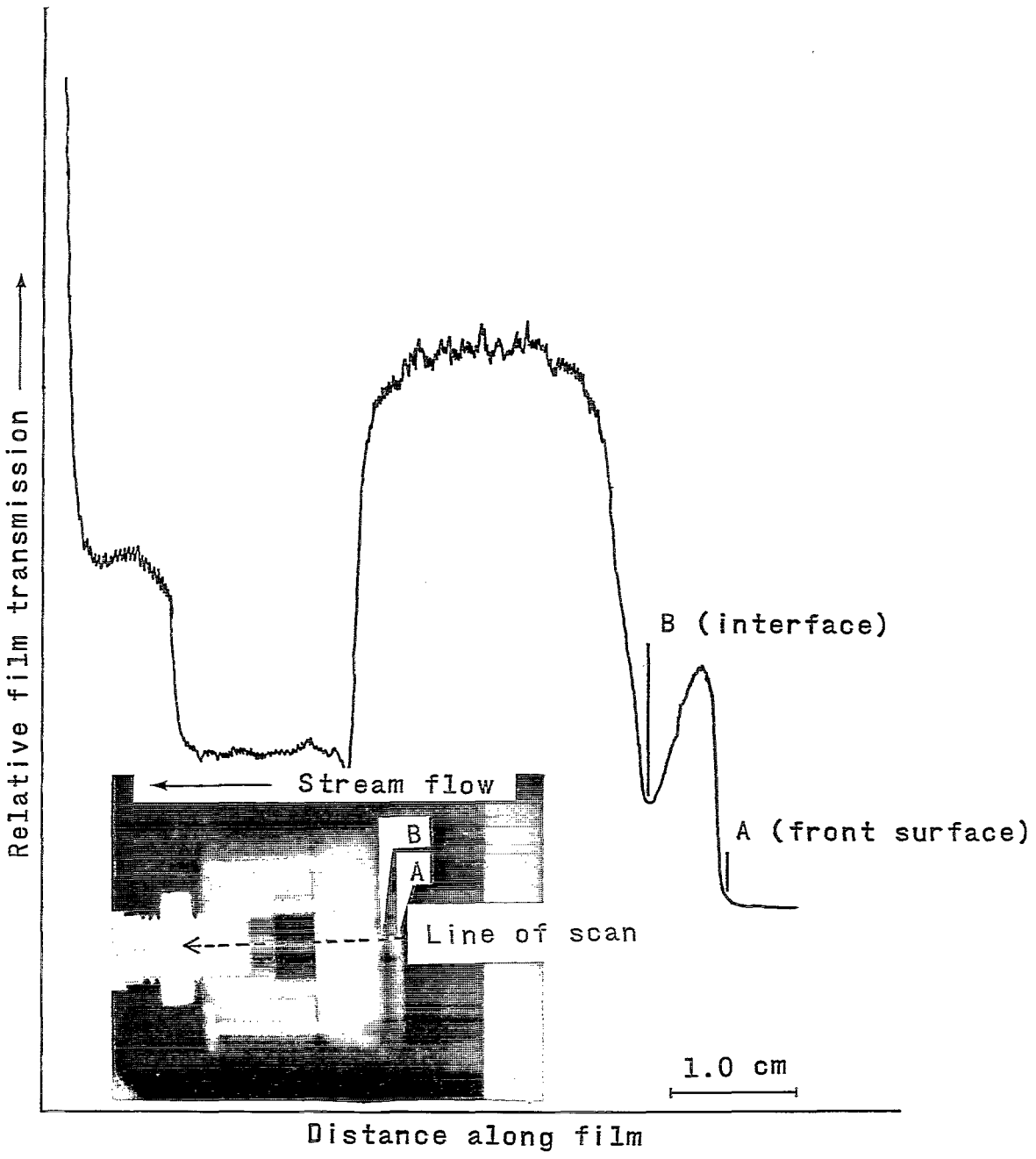
Figure 4.- Continued.



(f) 300 seconds after test.

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Figure 4.- Concluded.

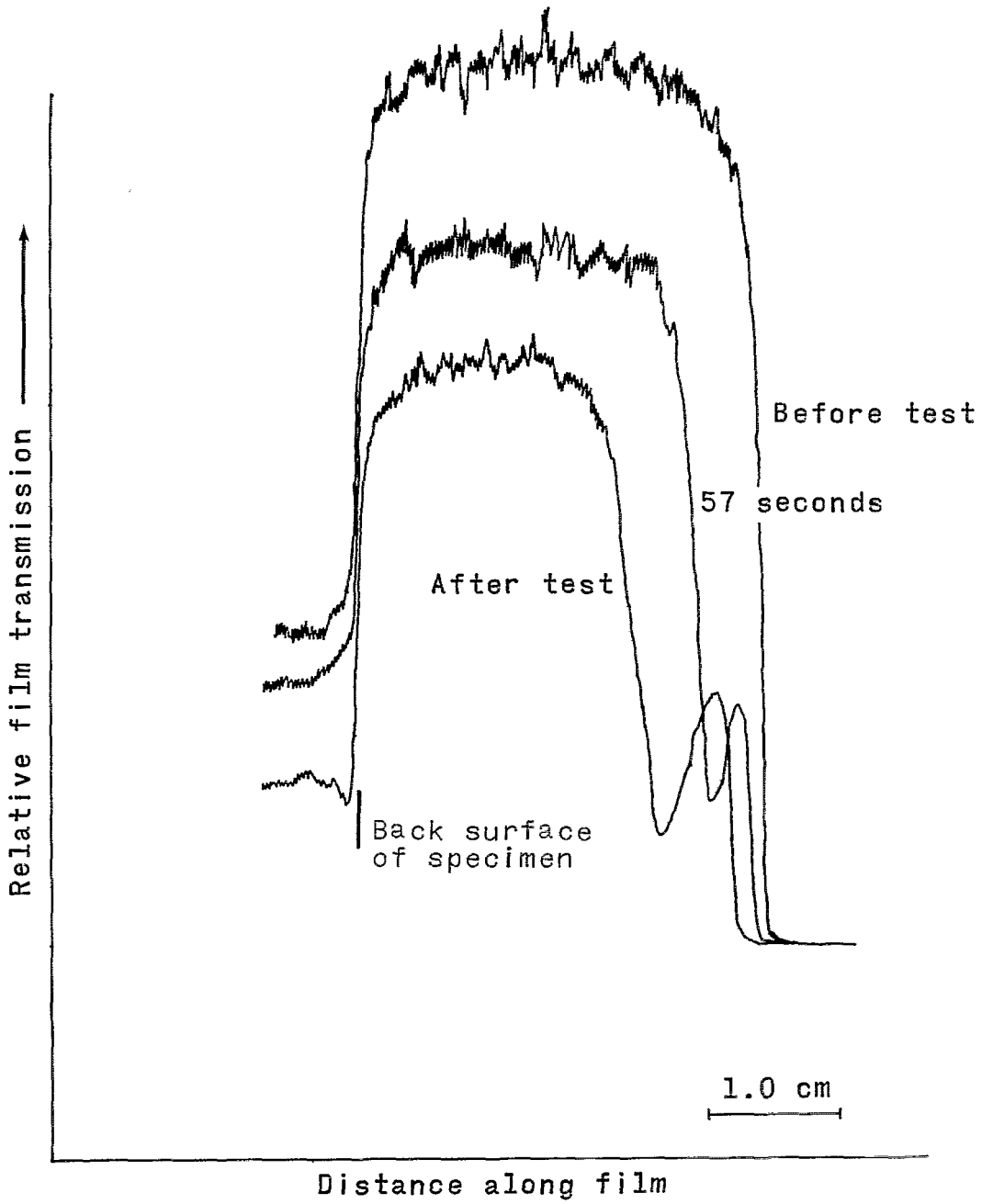

Figure 5.- Superposition of microphotometer traces of three radiographs of high-density phenolic-nylon specimen at test times indicated.

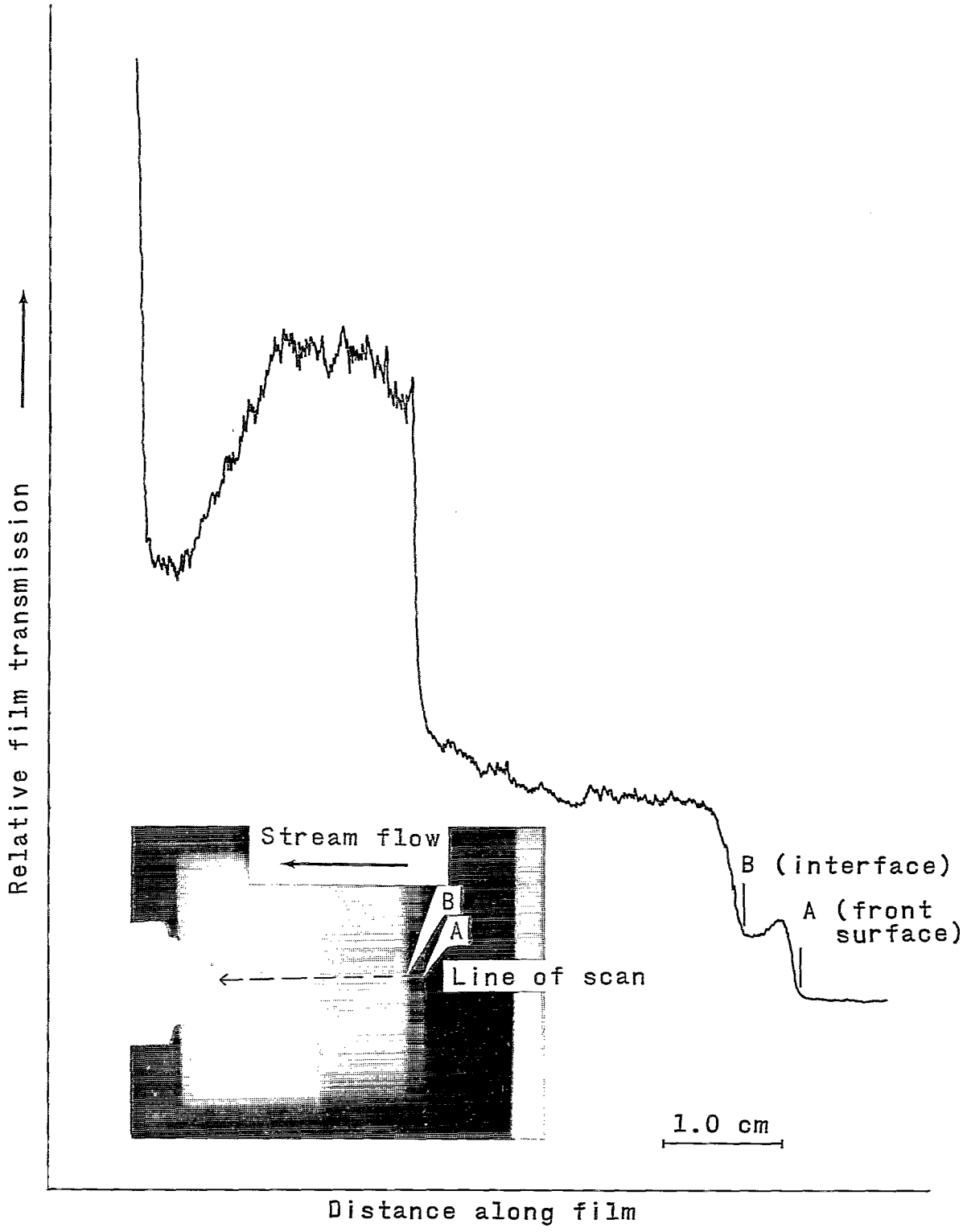

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Figure 6.- Radiograph and corresponding microphotometer trace of low-density phenolic-nylon specimen. Test time, 58 seconds.

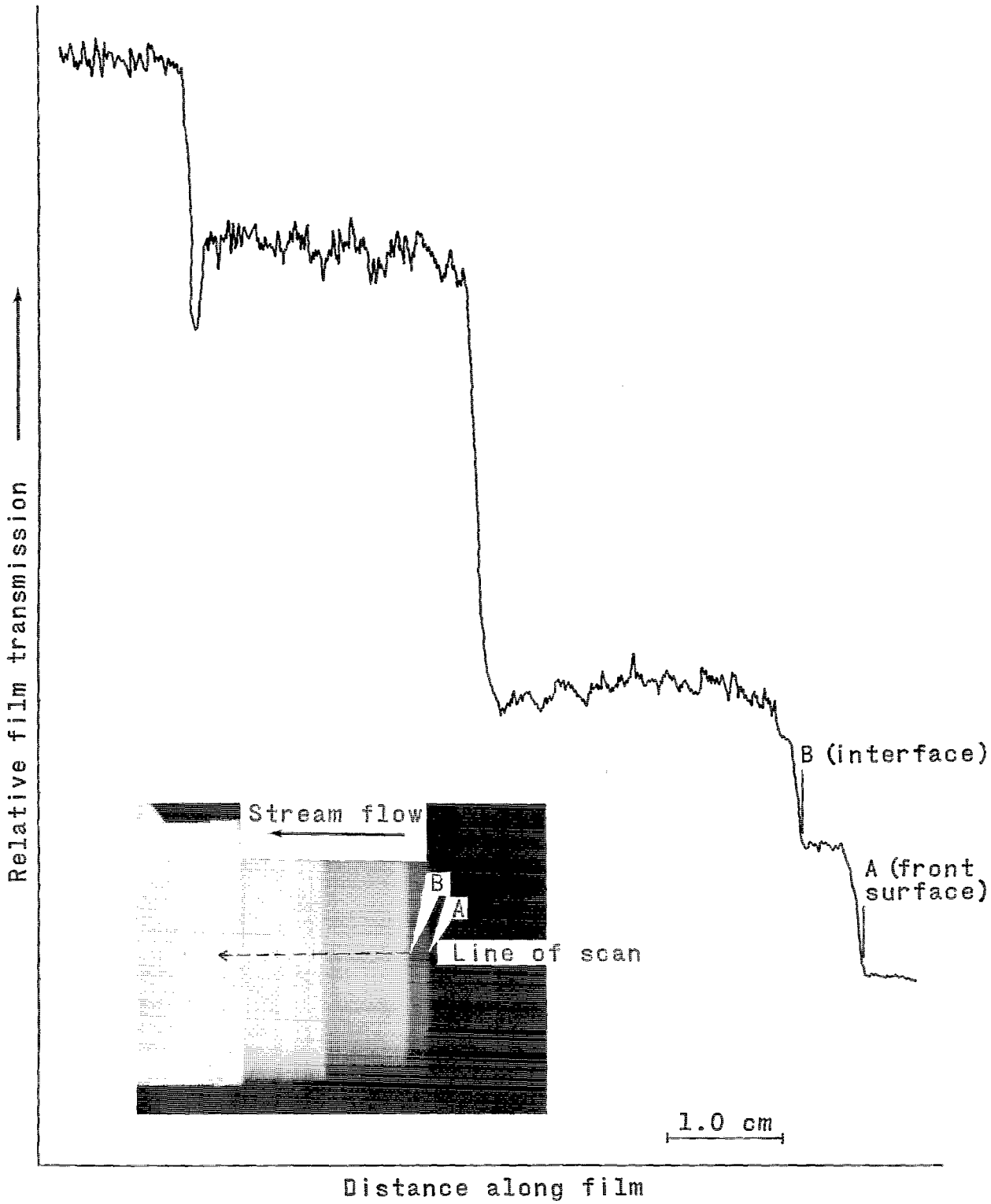

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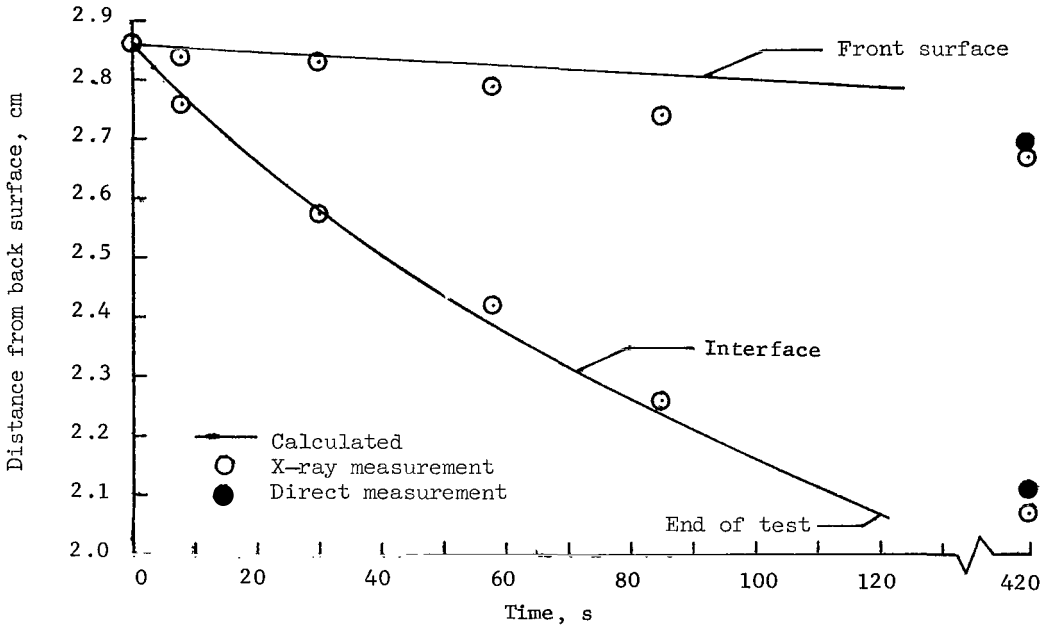
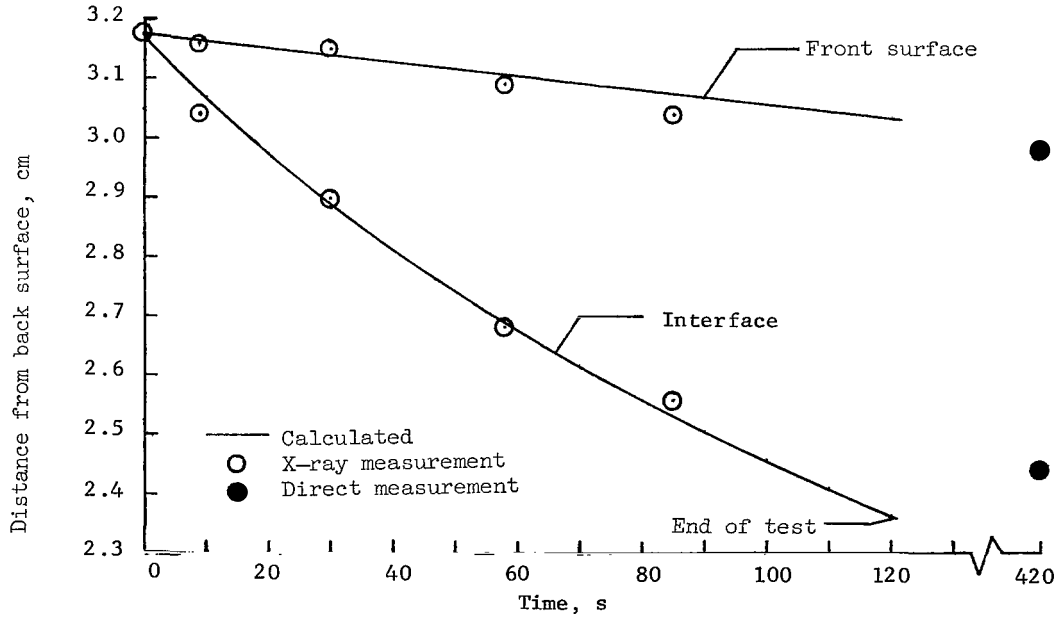
Figure 7.- Radiograph and corresponding microphotometer trace of epoxy-base material in fiber-glass honeycomb at 300 seconds after test.

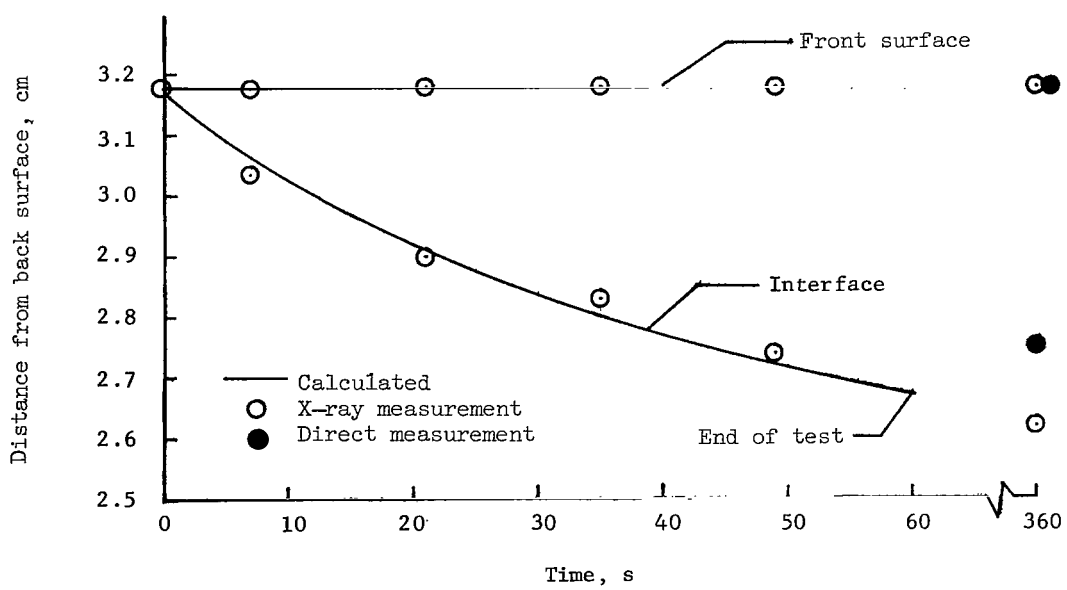


(a) High-density phenolic-nylon.



(b) Low-density phenolic-nylon.

Figure 8.- Comparison of experimental and calculated surface and interface recessions.



(c) Epoxy-base material.

Figure 8.- Concluded.

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