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Fabrication and Characteristics of Experimental Radiographic Amplifier Screens

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SUMMARY

This report describes the problems met during the fabrication of radiographic amplifier screens (RAS) which had to satisfy some special requirements. The main problems were: 1) Improvement of the image quality. 2) Fabrication of a RAS with inverse ordering of the photoconductor-electroluminescent (PC-EL) layers. This construction serves to eliminate the strong absorption of low energy X-ray photons by the substrate glass and also makes the panel applicable for neutron detection. 3) Fabrication of a RAS on beryllium substrate. Since the beryllium has a much lower absorption coefficient for low energy X-ray photons than the glass substrate, this construction is advantageous for soft X-ray imaging.

The RAS panels were developed for use in nondestructive evaluation (NDE) operations requiring large format (over 500 cm^2) radiographic images with contrast and resolution capabilities unavailable with conventional fluoroscopic screens. The work reported herein was directed toward RAS panels usable for in-motion, on-line radiographic inspection by means of closed-circuit television.

The requirements set forth by the contract were only partly satisfied: 1) Improved image quality was demonstrated on one of the delivered standard construction panels. 2) Inverse ordering of the PC-EL layers was tried in two different construction: (a) Using an evaporated EL film (thin film EL) and sintered PC; (b) Starting with a

plastic embedded EL layer, followed by a plastic embedded PC layer. The first construction was very promising, but could not be fully developed due to lack of time. A panel in the second construction was delivered. Its main shortcoming is the very low contrast. 3) One panel on a beryllium substrate was delivered, however its image quality is still not satisfactory.

1. INTRODUCTION

The work reported here was done under Contract NAS3-20742 from March 1 to July 31, 1977. It was the continuation of Contract NAS3-19902, described in NASA Contractor Report CR-2822 [1].* The goal of this work was to fabricate and deliver four radiographic amplifier screens (RAS) with somewhat modified characteristics to those delivered in the previous contract.

The work in this program was performed at the Westinghouse Research & Development Center in Pittsburgh, Pa. by D. Leksell and Z. Szepesi as principal investigator. Managerial supervision was provided by T. P. Brody and D. H. Davies.

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*See References.

2. OBJECTIVES AND RESULTS

The goal of this work was to fabricate and deliver four RAS's in three different construction groups as listed in Table I.

TABLE I. CONSTRUCTION OF RAS PANELS

GROUP	CONSTRUCTION	SUBSTRATE	SIZE-cm^2	QUANTITY
1	Standard(PC-EL)	Glass	25.4x25.4	2
2	Inverse(EL-PC)	Glass	25.4x25.4	1
3	Standard(PC-EL)	Beryllium	12.7x12.7	1

The characteristics of the delivered panels should be similar to those of RAS-1542, RAS-1543 or RAS-1666 delivered previously and approach the specification listed in Table II.

TABLE II. GOAL CHARACTERISTICS OF RAS PANELS

ITEM	CHARACTERISTICS	OBJECTIVES	UNITS
1	X-ray range	5-150	kV
2	Output spectrum	500-600	nm
3	Resolution	6-8 (300-400)	lines per mm (lines per inch)
4	Driving voltage	100-400	VRMS
5	Driving frequency	60-1000	Hz
6	Maximum current	775 (5)	$\mu A/cm^2$ (mA/in^2)
7	Sensitivity threshold	10	mR/min at 70 kV
8	Contrast(gamma)	6	----
9	Time constants at max. gain	0.1	s
10	Mottle, grain and spot contrast density at max. gain.	0.3	----

The list of delivered panels is given in Table III.

TABLE III. DELIVERED RAS PANELS

ITEM	GROUP	RAS No.	SIZE cm^2	 (in^2)	MAXIMUM DRIVING VOLTAGE
1	1	1645	25.4x25.4	10x10	300
2	1	1682	25.4x25.4	10x10	300
3	1	1681	7.62x7.62	3x3	150
4	2	PL-4	10.16x10.16	4x4	120
5	3	1679	7.62x7.62	3x3	130

The amplifier screen RAS-1681 of group 1 approached most closely the specifications of Table II. However its size and its contrast ($\gamma = 3.3$) were somewhat smaller than specified. Its rise time at maximum gain (at about 1.3R/min) was several hundred ms, its decay time longer. Its transfer characteristics are shown in Figure 3.

The other two panels of group 1 (RAS-1645 and RAS-1682) satisfied the size specifications and also items 1 to 6 of Table II. Figures 1 and 2 show the transfer characteristics of the panels RAS-1645 and RAS-1682 respectively. As it is seen there, the sensitivity threshold is around 100 mR/min for both amplifier screens, and the maximum contrasts are $\gamma_{max} = 1.5$ and 2.17 respectively. The lower contrast was made for widening the working X-ray intensity range similar to that of RAS-1666 (Fig. 6 in Ref. 1). It can be seen in Figure 3 that the panel RAS-1681 has only a one decade input working range, meanwhile the RAS-1645 and RAS-1682 have about three decades of working range. But for the wider working range one had to sacrifice the contrast.

However higher contrast can be obtained by using a higher frequency driving voltage. The response times of the panel RAS-1645 are somewhat faster and those of RAS-1682 quite slower than those of RAS-1681. The image quality of the two amplifier screens are not as good as that of RAS-1681. Further discussion of this fact is given in Section 3A.

The amplifier screen with inverse ordering of the PC-EL layers (RAS-PL-4) has a very low contrast (See Figure 4) and shows a fine grainy structure. Its top electrode (on the PC layer) is a thin film of gadolinium (about 0.03 μm) and gold. Its main use could be the detection of neutron radiation. If the thickness of the Gd film is not enough, the Gd sheet (delivered with it) could be placed on top of the gold electrode and the detection efficiency could be increased. Similar arrangements with group 1 screens, placing the Gd sheet on the glass substrate, could probably improve the neutron sensitivity of these panels also.

The Be substrate amplifier screen (RAS-1679) has a contrast of nearly unity. Its image quality at maximum gain is good, but at lower excitation some spotty structure is visible. However, it should give better sensitivity for low energy photons than the glass substrate amplifier screens, and sandwiched to the Gd sheet it could be useful for neutron detection.

3. CONSTRUCTION PROBLEMS

The fabrication procedure of the amplifier screens in group 1 and 3 was principally the same as described in pages 10 to 14 of Reference 1. However, some problems arose during this work and also the construction of the inverse order PC-EL panel presented some problems. In the following these problems will be discussed.

A. Standard Glass Substrate Panels

A1. Image quality

The main goal in the standard construction was to improve the picture quality by eliminating non-uniformities, grains, spots and mottled structures on the output image. It was found in previous work that non-uniformities and pinholes in the tin-oxide coating of the glass substrate cause non-uniformities and spots in the image. Also a bad tin-oxide coating can result in a grainy and mottled image structure.

Coating the tin-oxide film with a thin inactive insulating film as Al_2O_3 or SiO can eliminate most of the above deficiencies. We deposited by electron beam gun evaporation about 1 μm thick SiO film on smaller (7.6 cm x 7.6 cm) substrates and observed the improvement in image quality. The delivered panel RAS-1681 was built on such a substrate.

Since the size of the substrate in the EB-gun unit was limited to about 16 cm x 16 cm, another vacuum system was used for the large substrates, where the SiO was evaporated by thermal heating. In these experiments the image quality was not as good as on the smaller panels. The shortness of time did not permit finding out the cause of this difference and the delivered large panels were built without the SiO coating film. Consequently more deficiencies are visible on the output image especially at low intensity input.

Beside the tin-oxide coating, any small dust particle fallen on the PC layer can cause bright or dark spots in the image. Most critical is the time until the end of the sensitizing, because a very small particle during the high temperature baking can be diffused as a donor or acceptor and results in a much larger and disturbing spot. To eliminate this extremely careful handling of the panel in a clean atmosphere is essential. Most of the preparation of the panel was made in a super-clean laminar-flow bench, however some steps had to be done in a much less clean atmosphere.

A2. High dark current

From the beginning of this work program for quite a long time a disturbing problem was a high dark current in the PC layer, which caused high background brightness and low contrast on the output image. Many series of experiments were carried out for finding the cause of the high dark current. It was thought that an excess $CdCl_2$ remaining in the sintered layer is the culprit. This feeling seemed to be confirmed by the fact that a water wash of the PC layer largely decreased the dark current. However intensifier panels built on such PC panels showed some patterns caused by the washing, therefore the washing did not solve the problem. Several experiments were tried after this to compensate or eliminate the high $CdCl_2$ or donor concentration, as:

1. Additional copper evaporation and subsequent baking of the high dark current PC layer
2. New CdSe-S powder mixtures prepared with higher copper concentration.
3. Using newly made very pure $CdCl_2$ and new solution of $CuCl_2$.
4. Cleaning the sensitizing furnace, which could have a large quantity of $CdCl_2$ deposited on its walls.
5. Changing the Xylene used in the settling of the PC powder.

Though most of these steps resulted in some decrease of the dark current, they did not bring about the expected change.

At the end two observations cleared up the high dark current problem:

1. The temperature controller of the furnace was not working correctly. This resulted in a 100°C higher baking temperature (1060°C instead of 960°C) of the pure CdSe-S mixture, and increased grain size. After the doping of the powder it was found that the dark current was higher with the higher temperature baked powder.

2. Still a stronger effect was produced by the addition of ethylcellulose to the PC powder and to the Xylene used for the settling of the powder.

In the standard preparation process a 0.1% ethylcellulose Xylene solution was used for the settling. Without the ethylcellulose the CdSe-S layer easily separated from the substrate and several times caused the rejection of the panel. It was supposed that the ethylcellulose disappears during the subsequent high temperature (520°-540°C) baking.

Since the ethylcellulose does not dissolve easily in Xylene, its concentration was generally less than 0.1%. Since at the beginning of the work a failure in the adhesion of the PC layer was experienced special care was taken in dissolving the ethylcellulose powder. Even occasionally a small amount of ethylcellulose was mixed to the PC powder for the ball milling operation. It was finally proved that this increase of the ethylcellulose caused a strong increase in the dark current and an instability of the PC layer. Settling in pure Xylene resulted in a very low dark current and good stability (probably better than any previously made panels had).

B. Inverse Ordering of PC-EL Layers

The standard fabrication method does not permit changing the order of the PC-EL layer deposition, because the plastic embedded EL layer cannot stand the high temperature, needed for the sensitizing of the PC layer. Four methods were considered to solve this problem:

1. Thin film EL (TF-EL) and sintered PC layer
2. Plastic embedded EL and plastic embedded PC
3. TF-EL and TF-PC
4. Glass embedded EL + sintered PC.

Because of the limited time, experiments were conducted only according to constructions 1 and 2.

B1. TF-EL and sintered PC layer

The breakthrough in the development of high brightness, long life and high contrast TF-EL cells [2] offered the possibility not only to fabricate an amplifier screen in inverse ordering of the PC-EL layers, but also to increase the resolution of the image appreciably, mostly by using an evaporated PC film with the TF-EL. The main problem in this construction is to eliminate interdiffusion of impurities between the EL and PC layers. Due to the limited time, only very few experiments were carried out for solving this problem.

Experiments were made by depositing first the TF-EL on a NESA[†] coated glass, followed by the deposition of the PC powder. During the high temperature sensitizing of the PC layer, both the EL and the PC layers were poisoned.

Depositing a 0.7 μm thick SiO film between the EL and PC layers saved the PC sensitivity, but did not protect the EL film.

A thicker,(about 1.2 μm) SiO film was tried next, but still the EL brightness was lost.

It was planned to use about 2 μm thick SiO or Al_2O_3 film for the separation, thereby providing a good protection of the EL film, but there was not enough time to do this.

[†] Transparent tin oxide.

B2. Plastic embedded EL and plastic embedded PC

By using plastic embedded powders for both layers, high temperature baking is not needed and there is no danger of impurity diffusion from one to the other layer.

In a previous work program [3], the preparation of plastic embedded PC powders was developed. This technique was used in building RAS-PL-4.

The fabrication schedule of this panel was the following:

1. A standard plastic embedded EL layer was deposited on a NESA coated substrate glass by the usual spray-method.
2. The sensitized PC powder (100g) was mixed in a plastic-solvent solution of the following composition:

    0.3g ethyl cellulose
    14 ml xylene
    10 ml amylalcohol
    1.6ml di-butyl phtalate
    0.13 ml butanol

3. The PC plastic mixture was doctor-bladed above the EL layer (thickness about 150 μm)
4. Drying was done in a 135°C furnace for 30 minutes.
5. Gadolinium was evaporated on top of the PC layer.
6. A gold film was evaporated on top of the Gd since the conductivity of the Gd was too low.

A thin insulating layer of acrylic plastic (Krylon Crystal Clear Spray Coating, Type 1302) was applied in an experiment between the EL and PC layers, but since it diminished the contrast, its use was discontinued.

The main problem in this construction is the very low contrast of the output image which is caused by the high superlinearity of the voltage-current characteristic of the PC layer. Figure 5 shows the characteristics of the PC layer used in the RAS-PL-4 panel. We see that the maximum slope of these curves is about 8 ($I \sim V^8$). By adjusting properly the impedances of the two layers (PC and EL) one should be able to improve the contrast of the output image, but there was not enough time to do this.

C. Beryllium Substrate

The fabrication process of the image converter on beryllium substrates was exactly the same as that on glass substrates.

The main problem here was the non-uniform oxidization of the Be surface, as discussed on pages 3 and 4 of the previous report [1]. The same method, which eliminated the imperfections of the NESA coating on the glass substrate, should solve this problem also.

Several panels on Be substrates were fabricated by:

1) Etch cleaning the Be substrate in a 10% oxalic acid during 30 minutes.

2) Depositing a SiO film on the Be plate

3) Continuing with the deposition of the PC powder etc. exactly the same way as in the glass substrate standard fabrication process.

The delivered panel RAS-1679 had an about 1 μm thick SiO coating which still was not enough (or the adhesion to the Be was not perfect) and the panel shows some grainy structure. It is thought that thicker SiO or Al_2O_3 should eliminate this spottiness. The shortness of time did not permit further investigation of this.

4. DISCUSSION AND RECOMMENDATIONS

The work of this program proceeded along three lines as follows:

(a) Fabricate two large area (25.4 cm x 25.4 cm) solid state RAS-s with improved image quality and with specified other characteristics.

(b) Fabricate a RAS with inverse ordering of the PC-EL layers.

(c) Fabricate a RAS on Be substrate.

Several problems emerged during the short work period of this program and the time did not permit the thorough solving of all problems. Consequently the delivered panels do not represent the best that could have been achieved if more time had been available.

An improved picture quality was obtained in the standard glass substrate amplifier screen by the deposition of a SiO film on the NESA coated substrate. A smaller (7.6 cm x 7.6 cm) panel was delivered to NASA to demonstrate this improvement.

Two approaches were tried in the fabrication of the inverse ordering EL-PC panels. The ideal solution, building the panel with evaporated thin films of EL and PC layers, could not be planned in this short program. However, some experiments were made with evaporated TF-EL and sintered PC layer, but the time was not enough to make a working panel in this construction.

The delivered EL-PC panel was built of completely plastic embedded layers. Its low contrast and a fine grainy structure are the most objectionable characteristics of this construction.

The Be substrate panel did not have a satisfactory image quality. It is strongly felt that with a small effort both the large size amplifier screen and the Be substrate screen could be fabricated with the desired improved picture quality.

Somewhat higher effort would be needed for the fabrication of an acceptable TF-EL and sintered PC construction amplifier screen. However, the ideal construction with TF-EL and TF-PC, which could bring a large improvement in resolution and image quality, would need a major effort.

5. REFERENCES

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3. Z. Szepesi et al, Solid State Image Intensifiers, Final Report on Contract N61339-1440, NAVTRADEVCEN 1440-2, Nov. 1966, AD-645590.

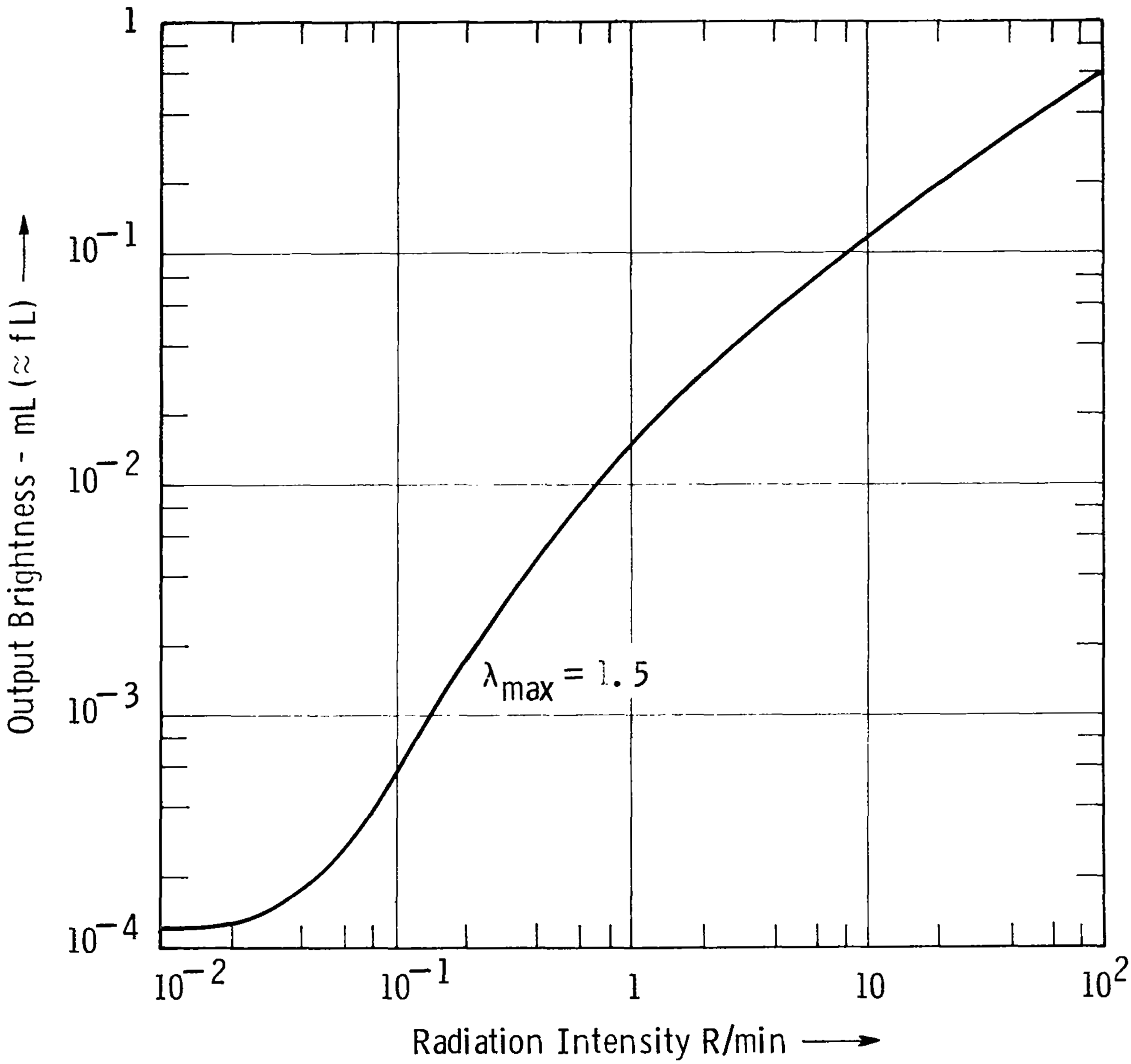

Fig. 1 — Transfer characteristics of RAS - 1645. Driving voltage: 250 volts ; frequency: 60 Hz; X-ray tube voltage: 70 kVP.

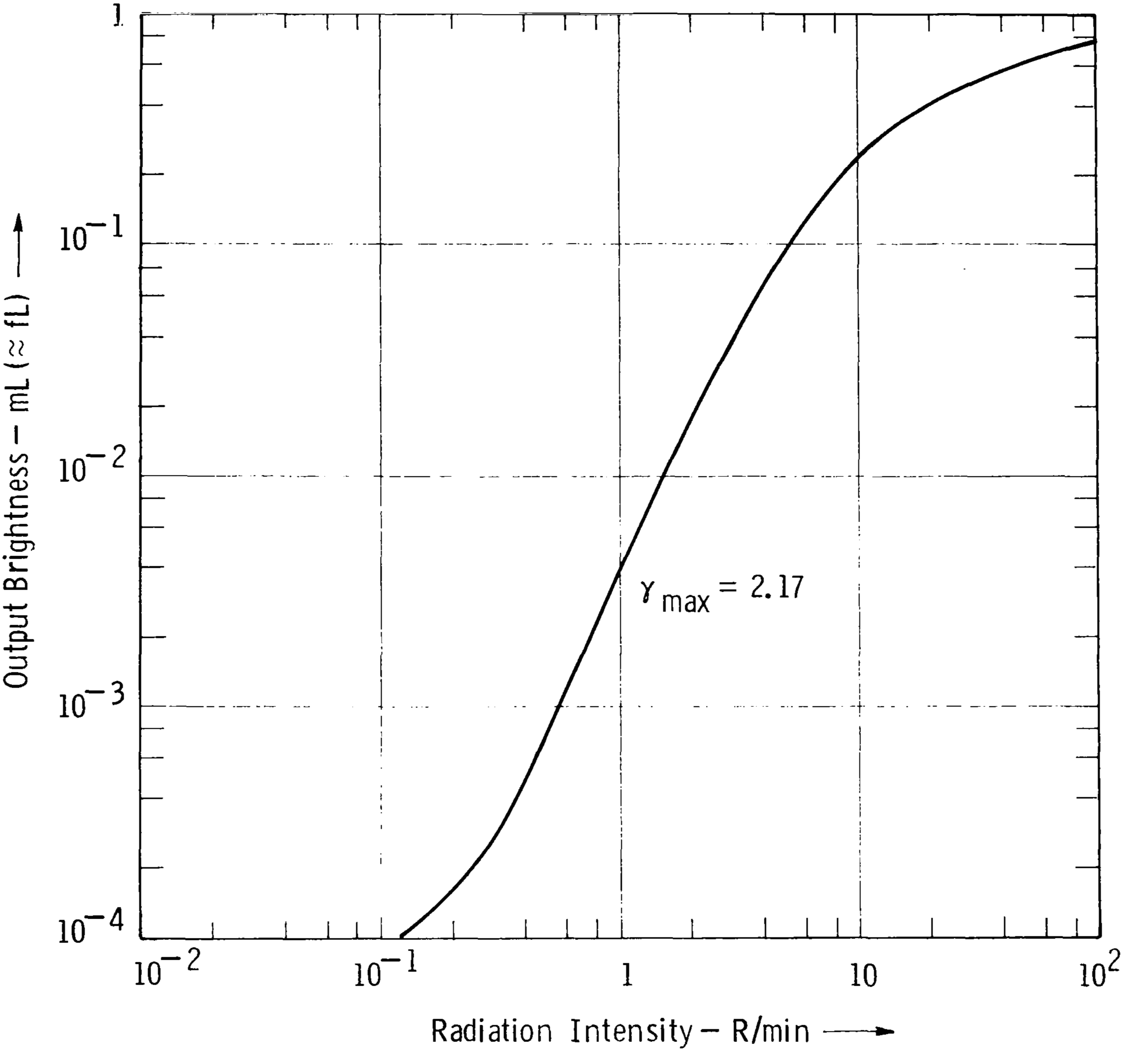

Fig. 2 — Transfer characteristics of RAS-1682. Driving voltage: 250 volts; frequency: 60 Hz; X-ray tube voltage: 70 kVP.

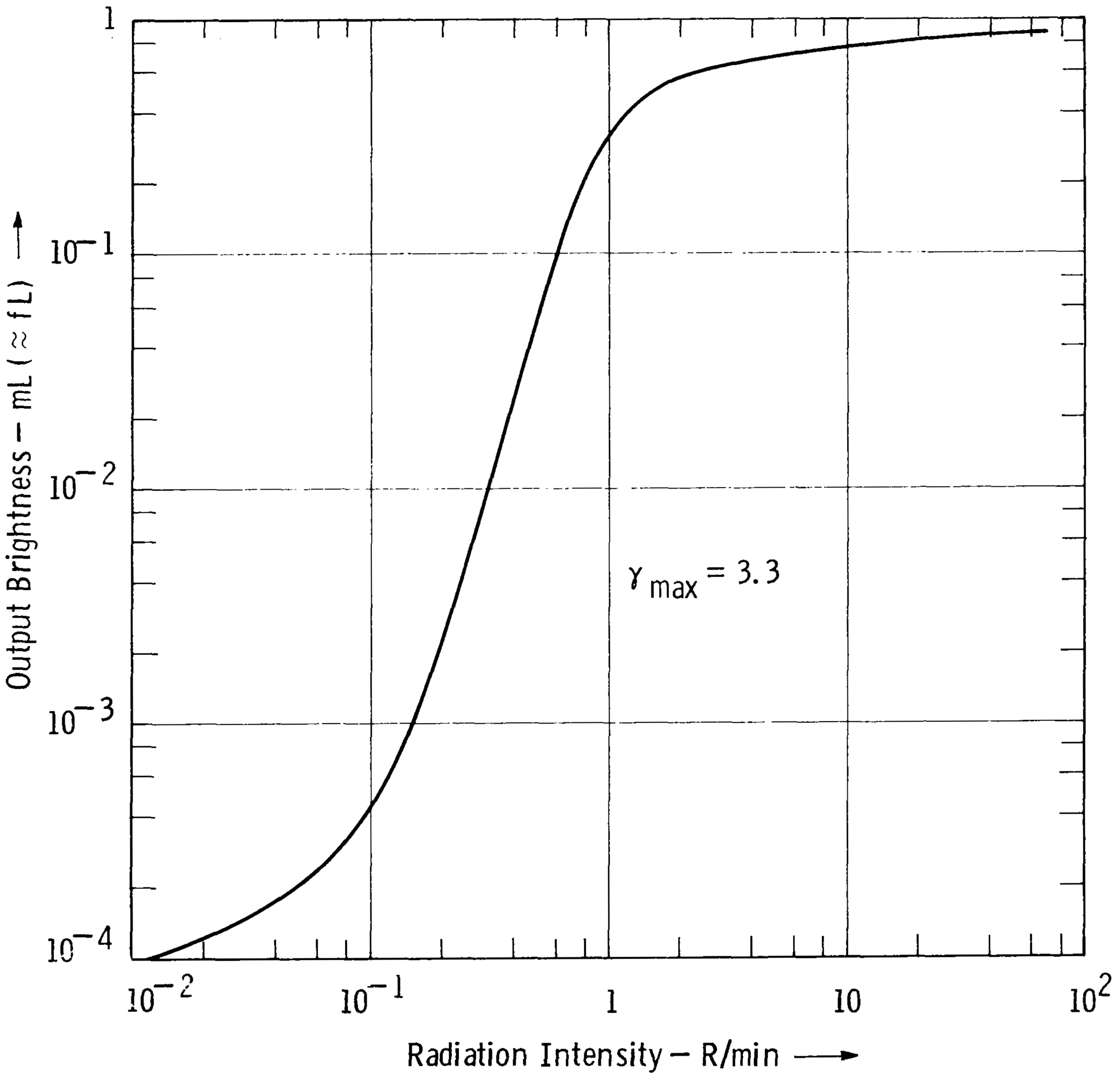

Fig. 3 – Transfer characteristics of RAS-1681. Driving voltage: 150 volts; frequency: 60 Hz; X-ray tube voltage: 70 kVP.

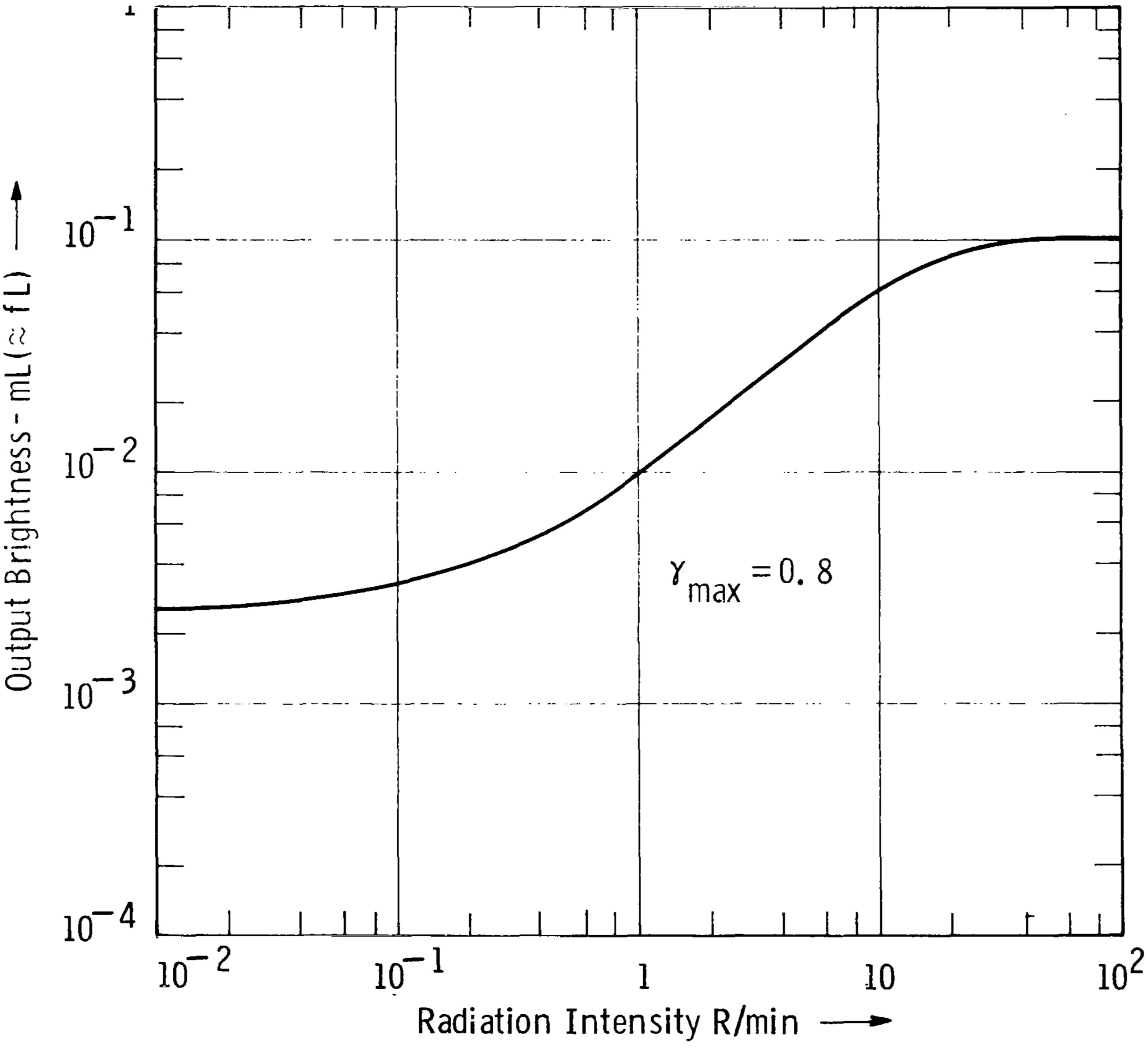

Fig. 4 — Transfer characteristics of RAS-PL-4. Driving voltage: 120 volts; frequency: 60 Hz; X-ray tube voltage: 70 kVP.

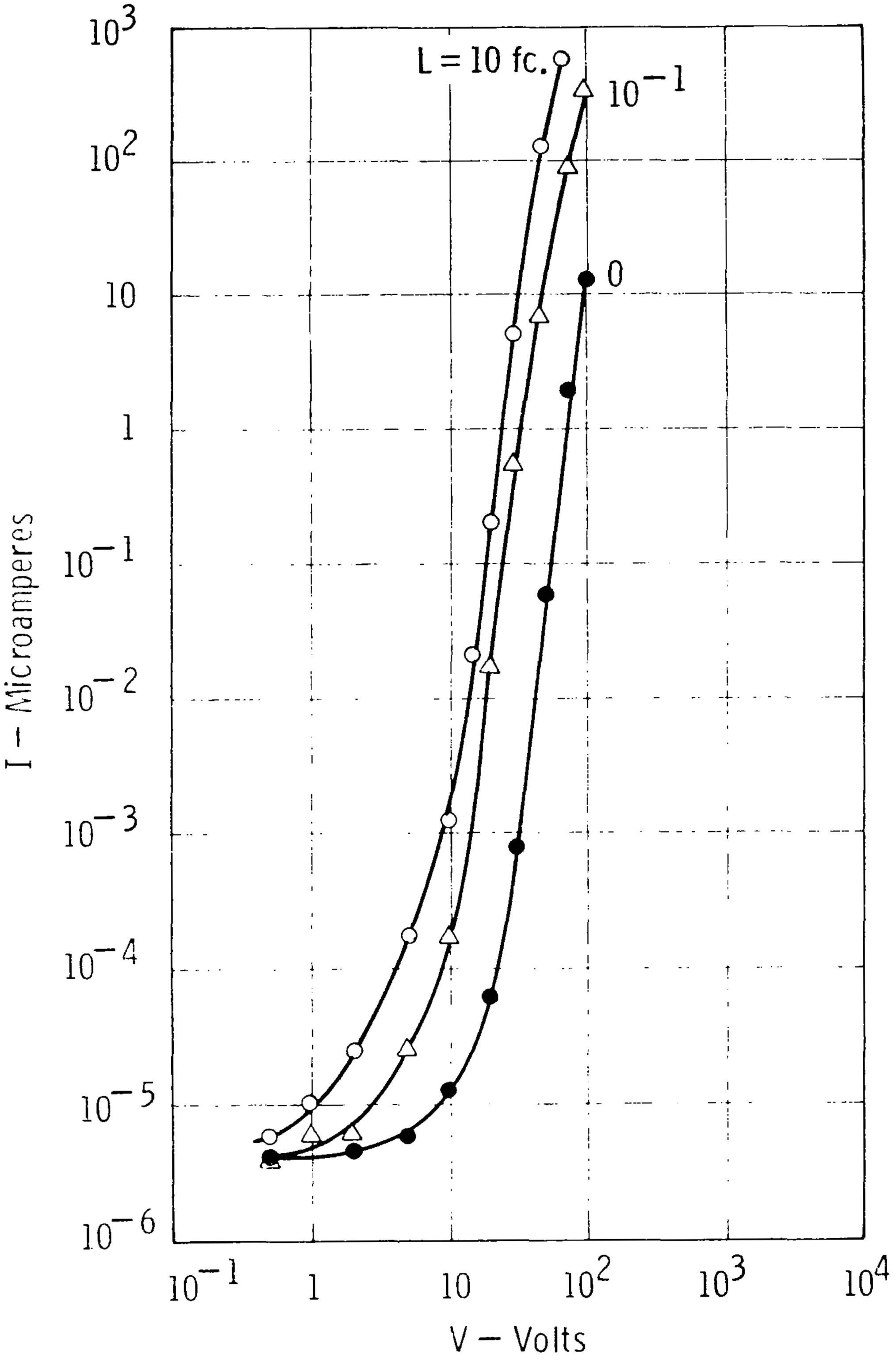

Fig. 5 — Current vs. voltage characteristics of plastic embedded PC cells