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THE EFFECT OF TERMINATION ALLOYING ELEMENTS ON THE RESISTANCE OF VACUUM EVAPORATED ALUMINUM THIN FILMS

by

Walter W. Powell

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CERTIFICATE OF APPROVAL

This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science.

23 May 1966
Date

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TABLE OF CONTENTS

TABLE OF CONTENTS (cont.)

Page

Resistance Behavior During Annealing (cont.)

LIST OF FIGURES

LIST OF FIGURES (cont.)

LIST OF TABLES

ABSTRACT

A study was conducted to investigate the reasons for changes in the effective series resistance of aluminized capacitors with time. The elements of a quarternary alloy, zinc, tin, cadmium and indium were chosen for the study. Each element was deposited onto 1500 Å aluminum films by vacuum evaporation. Zinc produced a decrease in resistance upon deposition and a subsequent increase during annealing. Tin had no effect on resistance upon deposition, but produced a decrease in early stages of annealing. Cadmium and indium did not affect the film resistance significantly. The magnitude of resistance changes was distinctly dependent on the equilibrium state (annealed or unannealed) of the aluminum films. The polarity of dc current flowing through the specimens during annealing had no appreciable effect on the resistance behavior.

INTRODUCTION

Aluminized Mylar capacitors terminated with various alloys have exhibited distinctive changes in ESR (effective series resistance) during a 1000 hour constant temperature (86^0C) and humidity (95%) test with charge voltage applied. One group of capacitors terminated with Zamak, an alloy of high zinc content, revealed a sharp initial increase followed by a gradual decrease. A second group terminated with Babbit, an alloy of high tin content, produced a distinct decrease followed by a shallow increase. The third group, terminated with Shinkalloy, an alloy of zinc, tin, cadmium and indium, exhibited a continuous increase.

It is the purpose of this paper to study in greater detail the effects of a termination alloy and its individual components on the resistance behavior of aluminum thin films. Since Shinkalloy contains some of the primary elements of the other alloys of interest, it was chosen for the termination studies.

PRELIMINARY INVESTIGATION

Under the same test conditions described above, except for a dry atmosphere to eliminate any galvanic action, the resistance of Shinkalloy terminated capacitors and fuses behaved similarly but with some distinct deviations. The resistance versus time plots of recorded data are shown in Figures 1 and 2 for the capacitors and fuses, respectively. The deviations observed were as follows:

1. The resistance of units with greater as terminated values decreased after 220 hours.
2. The rate of increase was generally more rapid for units with larger initial values.
3. The resistance of units with low initial values did not change appreciabily after 220 hours.

The flat wound test units were made from 162 turns of aluminized Mylar. Termination alloy was applied to each end with a pot spray and leads attached thereto. All units were encased to prevent damage from handling.

Two conclusions were made in light of the resulting data. One, since these units were tested in a dry atmosphere, the observed resistance behavior was due to phenomena other than galvanic action. And two, diffusion of the termination alloying elements into the aluminum film was not a likely factor in the behavior because of the low temperature (86^{o}C) and relatively short annealing time.

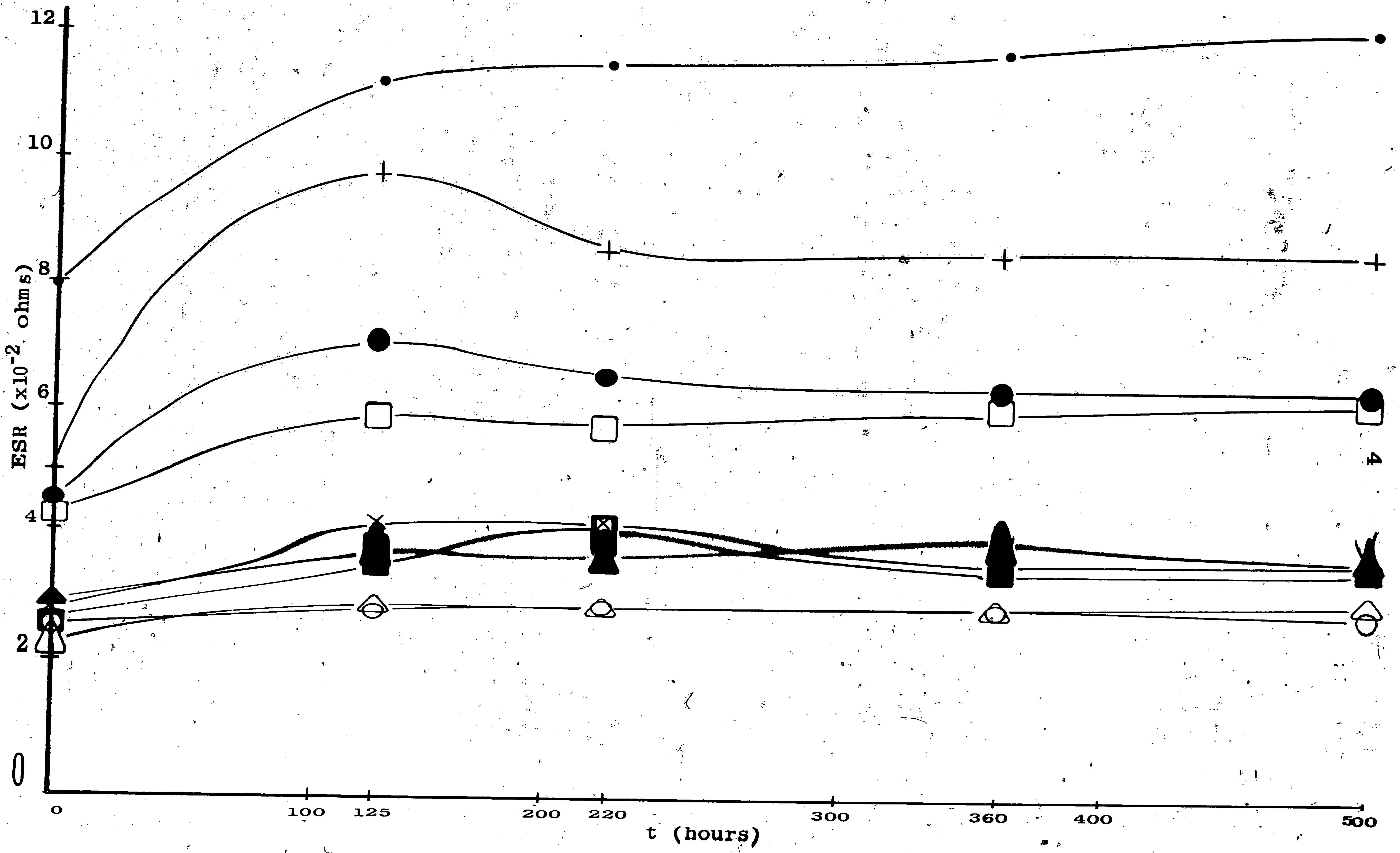

Figure 1. Effective series resistance (ESR) vs. annealing time for Aluminized Mylar Capacitors. (9 units)

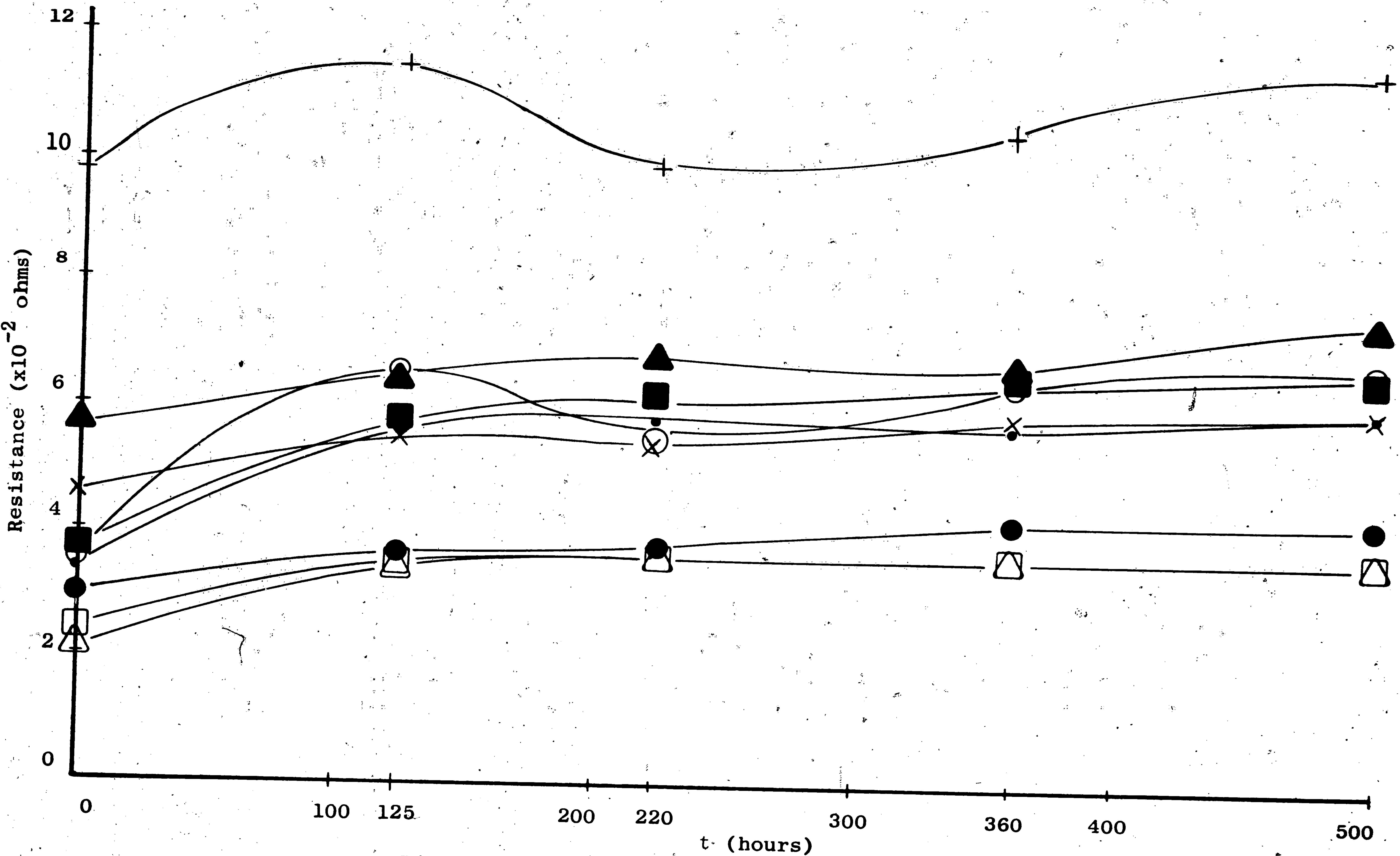

Figure 2. Resistance vs. annealing time for Aluminized Mylar Fuses. (9 units)

EXPERIMENTAL DESIGN

To initiate the investigation into the respective ESR and resistance behavior during temperature test of aluminum thin film Mylar capacitors and fuses terminated with Shinkalloy, an experiment was designed with the following objectives:

To determine

1— The effect each termination alloying element has on the resistance of thin aluminum films during an anneal.

2— If the equilibrium state of the films (annealed or unannealed) has any effect on the resistance behavior.

3— And if the polarity of dc current flowing through the film during the anneal contributes to termination resistance effects.

EXPERIMENTAL PROCEDURE

Ten aluminum thin film strips .1750" x 1.9375" approximately 1500 Å thick were simultaneously deposited by vacuum evaporation on ten 2" x 3", 1.27 mm thick glass microscope slides of which nine were precut into .3" x 2" segments. The non-segmented substrate was utilized for thickness measurements. Electrical leads in the form of .035" diameter high purity aluminum wire were attached to the ends of all remaining film strips by ultrasonic resistance heated welds. This type of bond was chosen because of its small contact resistance and elimination of a termination strip. Fig. 3 shows the preparation sequence from glass substrate to terminated strips. See Appendix I for equipment and detailed procedures used in these preparations.

Following termination the resistance of each specimen was measured on a standard Kelvin bridge setup providing values to 10.000 international ohms without interpolation. These and all subsequent measurements were made with 20 milliamperes of current at 1 volt.

The nine substrates were divided into groups of three, distributing resistance variation randomly among each. Also at this point, one strip from each substrate was set aside for metallographic studies. Group 1 was retained as deposited while groups 2 and 3 were annealed in a prepurified nitrogen atmosphere at 105°C for 10 and 35 hours, respectively. On completion of anneal and return to room temperature, the resistance of each specimen was again measured.

The elements of the termination alloy, tin, zinc, cadmium and indium, were applied to the aluminum strips by vacuum evaporation.

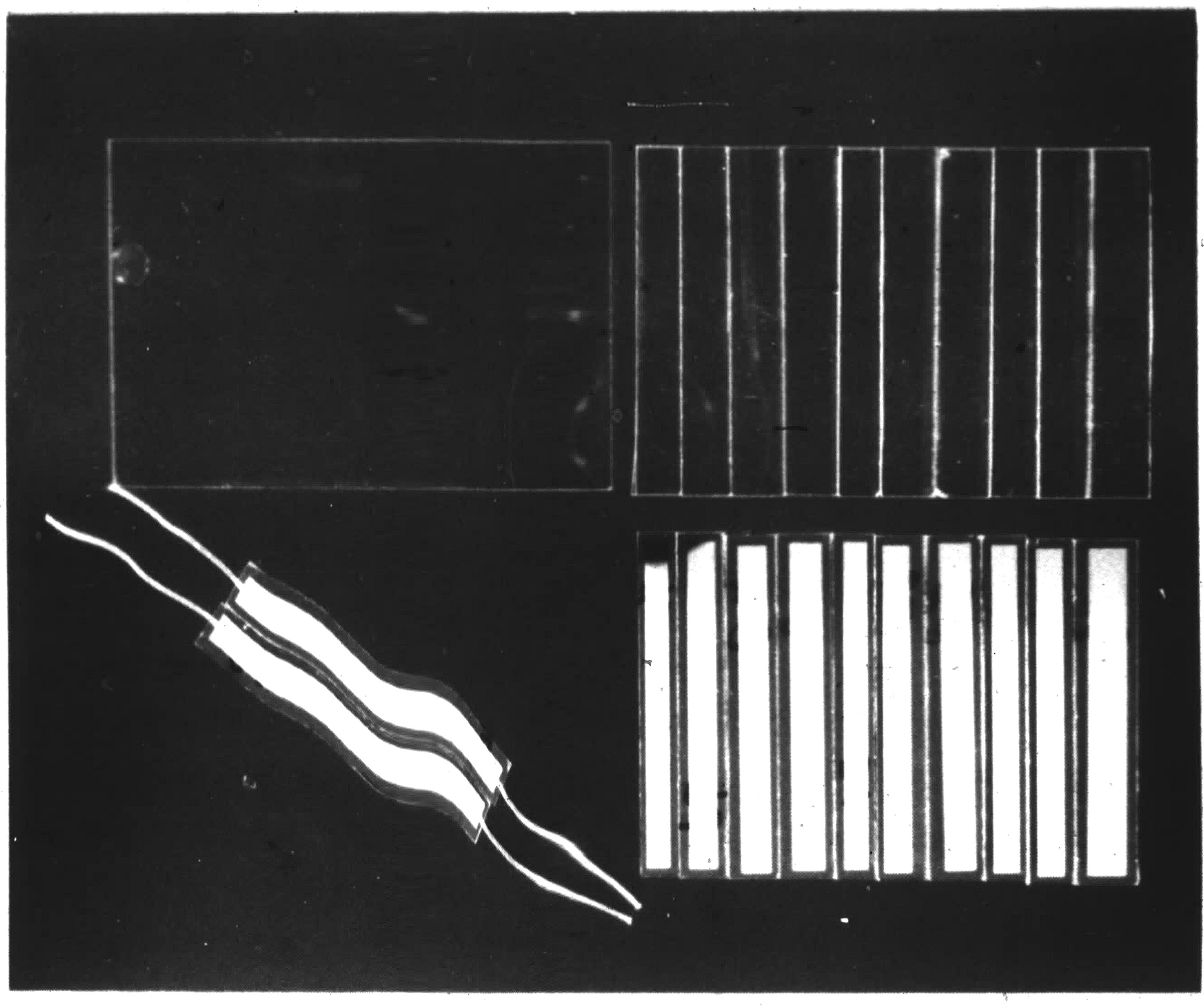

Figure 3 - Four stages in the preparation of the aluminum thin film specimens.

This method was chosen instead of flame or pot spraying to eliminate any annealing of the films during deposition. From 2000-3000 Å of one element was evaporated over 3/4" of the aluminum strip at the end of two segments, thus utilizing eight segments of each substrate. See Appendix I for details and deposition parameters. The ninth one was left as terminated for a control reference during subsequent annealing. Following deposition, the resistance of each unit was measured to establish reference values and to detect any change resulting therefrom.

The specimens were then placed on nine specially designed plugboards with each accepting the nine segments of respective substrates. The boards and their mated forced-convection oven were electrically wired to permit the passage of dc current through each unit during anneal. A current of 11 milliamperes at 260 volts was provided by a current regulated dc power supply. The arrangement on each board was such that the current would pass in different directions through the two specimens coated with the same element.

Prepurified nitrogen fed to the oven at a rate of 2 cu. ft./hr. provided a dry annealing atmosphere. Although the oven was preheated to the annealing temperature of 86°C, heat loss on loading specimens required an additional 15 to 30 minutes for temperature stabilization and nitrogen purge. Voltage was applied at this point and the temperature maintained within ± 1°F throughout the anneal. The annealing equipment and electrical circuits are fully described in Appendix III.

At intervals of 4 hours for the first 16 hours, specimens were removed from the oven, cooled to room temperature and their resistance measured on the Kelvin bridge. The interval was increased to 8 hours at 16 hours, to 16 at 48 and 40 at 144 until termination of the anneal at 224 hours.

Throughout the experiment, no attempt was made to prevent oxidation of the specimens in order to simulate conditions present during termination and testing of aluminized Mylar capacitors and fuses. Special precautions were taken to prevent the collection of moisture which might promote galvanic action and thus bias the experimental results. These precautions, which also limited dust collection on the aluminum strips prior to deposition of alloying elements, were realized through the use of desicators, a dry cooling box and a nitrogen annealing atmosphere.

EXPERIMENTAL RESULTS AND DISCUSSION

Deposition

The resistance behavior of specimens upon deposition of the four alloy elements was quite varied. Those coated with zinc experienced a distinctive drop in resistance, whereas those with cadmium suffered no significant change. The annealed specimens coated with indium and tin underwent no appreciable change, but considerable resistance drift occurred when measuring several of the unannealed indium specimens.

The decrease in specimen resistance immediately following deposition of zinc suggested the formation of a parallel resistance by the two films. This observation was supported by calculations of zinc film resistance from the resistance data. Values from specimen to specimen were reasonably consistent except for the four that experienced deposition difficulties (#12, 15, 18 and 27). A fifth specimen could not be measured because of a broken lead. Distribution of the thirteen values considered valid are given in Figure 4. See Appendix IV for actual values and method of calculation.

The average value of 11.619 ohms was considerably greater than the value of .5916 calculated from the polycrystalline resistivity[1] (5.916 microhm cm) with the equation

$$R = \frac{\rho l}{A} \qquad (1)$$

where ρ is the bulk resistivity, l the film length and A the cross-sectional area. Films were estimated to have an average thickness of 2500 Å, justifying the use of bulk resistivity.[2] The additional

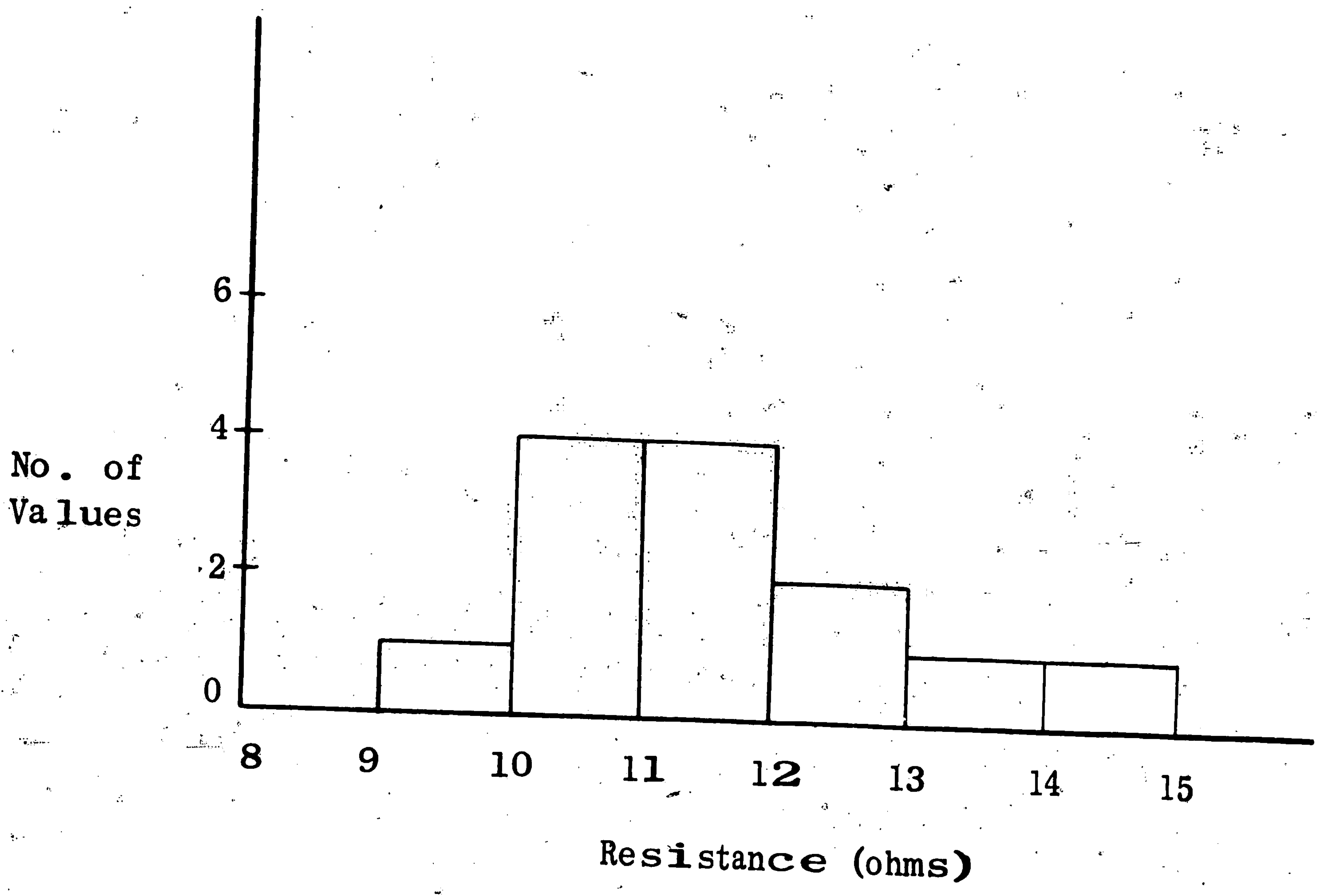

Figure 4. Distribution of resistance values for thirteen zinc films calculated from specimen resistance data.

resistance and conduction path between the films are thought to be the result of semiconduction in the oxide coating of the aluminum strips.

It is proposed that the semiconductive layer was formed as zinc atoms diffused into the oxide to provide conduction electrons. Since evaporated films are generally small grained[3] and contain numerous dislocations,[4] sources of enhanced diffusion[5] would exist in the oxide. Also, aluminum oxide is considered to be a metal-excess semiconductor in which excess cations and an equal number of electrons available for conduction are located in interstitial position;[6,7] consequently, one might expect that zinc atoms present in the oxide would similarly provide electrons for conduction. The possibility of aluminum atoms diffusion into the oxide to provide conduction was rejected, and no decrease in specimen resistance was observed upon deposition of the other three elements. Some increase in oxide thickness was expected for specimen with larger pre-anneals; however, there was no apparent reflection of this in the combined resistance of zinc film and oxide layer. It is possible that such a dependence could be concealed in the distribution of calculated values.

Anneal

Since the experiment was designed to determine the effects of termination alloying elements on the aluminum film resistance, it was necessary to extract as completely as possible the effects of self-annealing. This was accomplished by dividing each recording of specimen resistance by the appropriate control value (i.e., $(R/R_c)_t$, where t is the time of anneal when the readings were made). The resistance measurements made at designated intervals during the 224 hour anneal and the ratios obtained therefrom are tabulated in Appendix IV.

A plot of R/R_c versus annealing time was made from the data for each alloying element,* see Figure 5 (a), (b), (c), and (d) for zinc, tin, cadmium, and indium, respectively. Values of R/R_c were averaged for specimens with the same pre-anneal and are given in Table 6. In brief, the individual resistance behavior was as follows:

1. In zinc specimens it increased with annealing time approaching a maximum near 48 hours and leveling off beyond that point.
2. In tin specimens it decreased with time to a minimum near 8 hours becoming quite erratic beyond that point. The latter behavior believed to be the result resistance drift during measurement.
3. In cadmium specimens it did not change with time.
4. In indium specimens, except for four isolated cases, it also did not change with time.

Zinc Specimens

Data from zinc specimens produced smooth curves which increased non-linearly with annealing time through 48 hours. Little change occurred between this point and anneal termination except for a few large inflections resulting from peeling of the zinc film. The films

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* No data was available for #28 because of broken lead and #22 was not plotted because of severe peel during first four hours of the anneal. The data for #18 and #24 were not included because of a broken lead at 48 hours and no change in resistance during annealing, respectively.

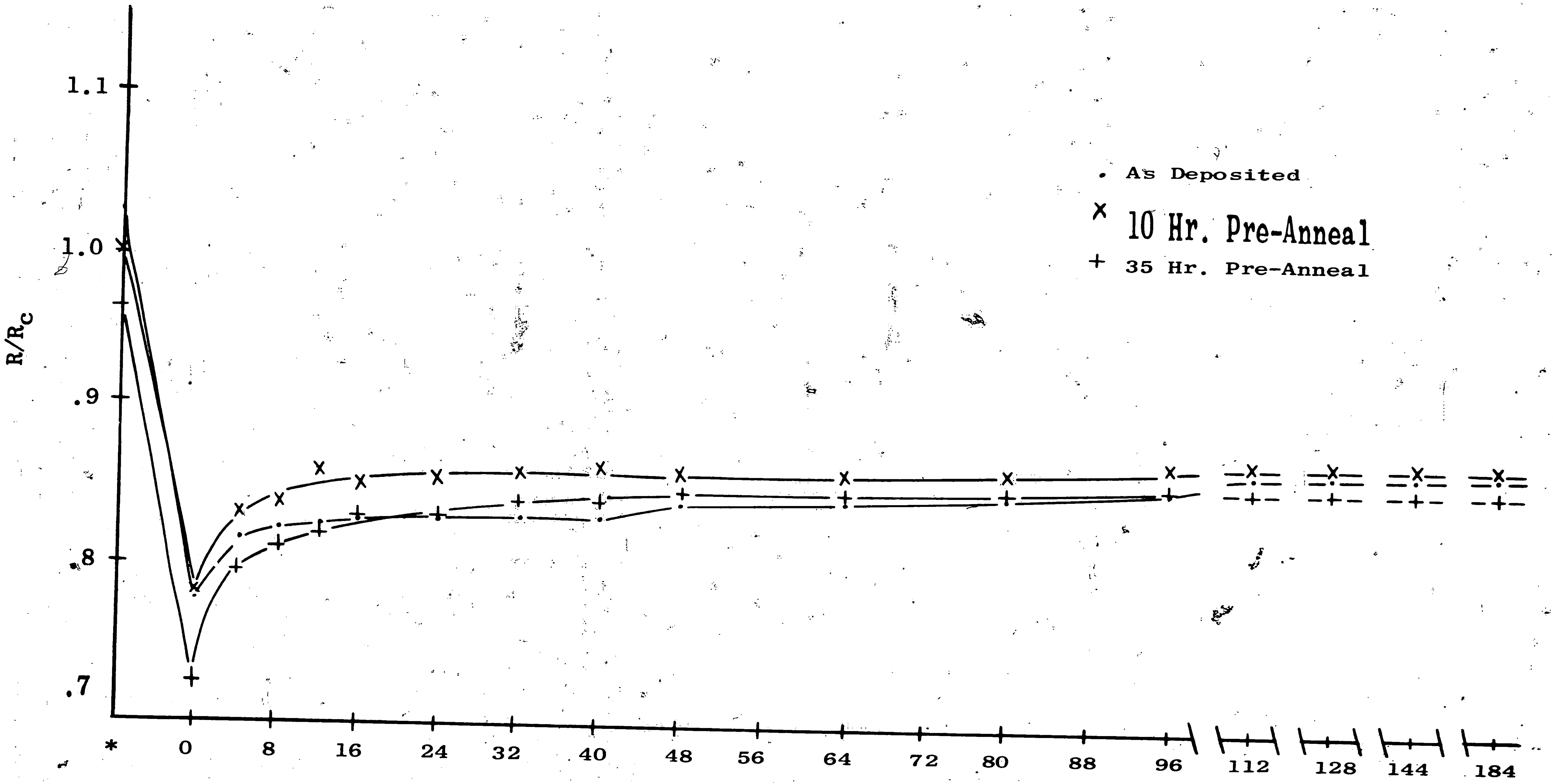

Figure 5(a). R/R_c (Average) vs. annealing time for zinc specimens; (* - average R/R_c value of aluminum strips before deposit of alloying elements).

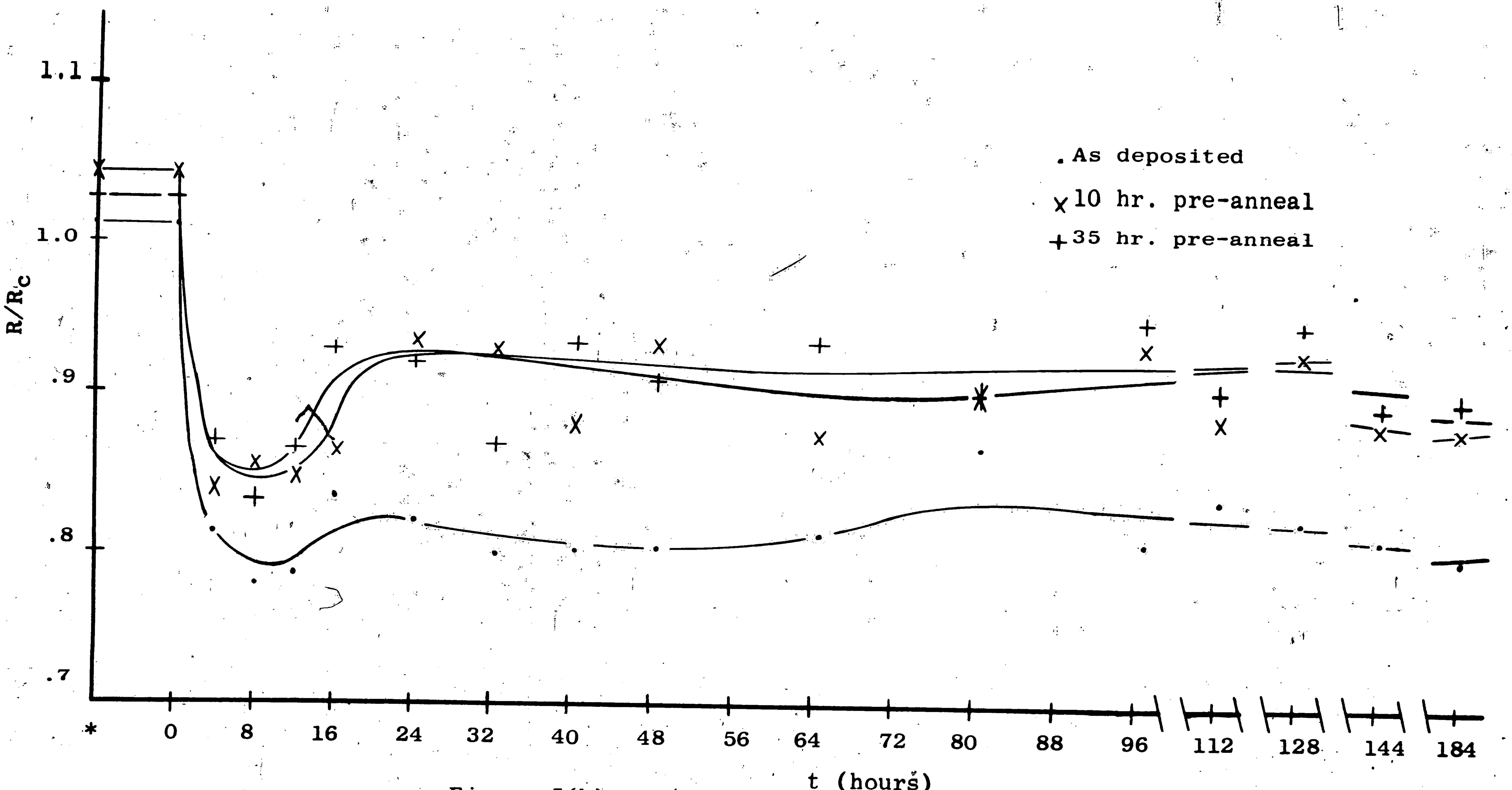

Figure 5(b). R/R$_C$ (Average) vs. annealing time for tin specimens (* - See Figure 5(a)).

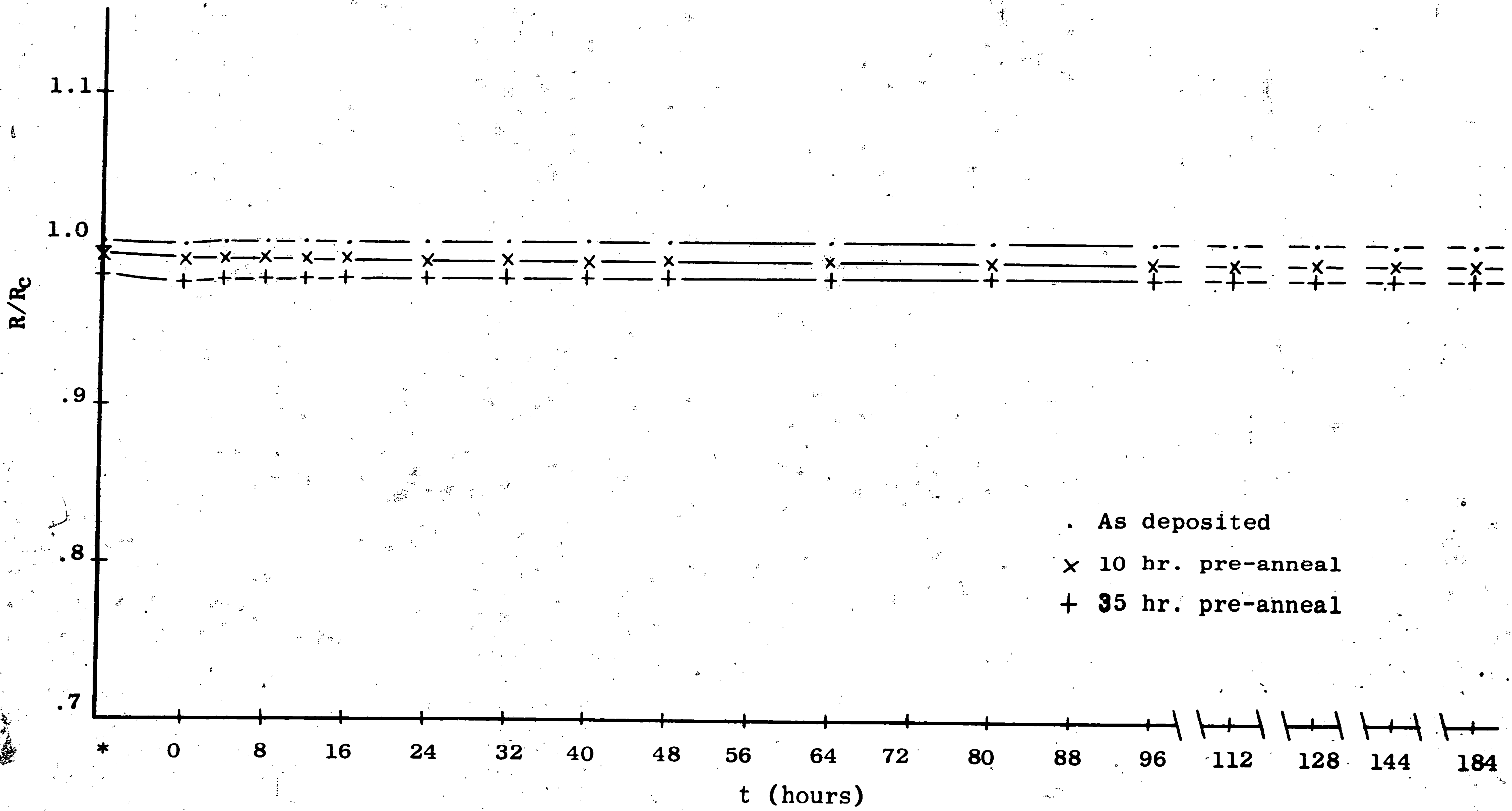

Figure 5(c). R/R$_c$ (Average) vs. annealing time for cadmium specimens (* - See Figure 5(a)).

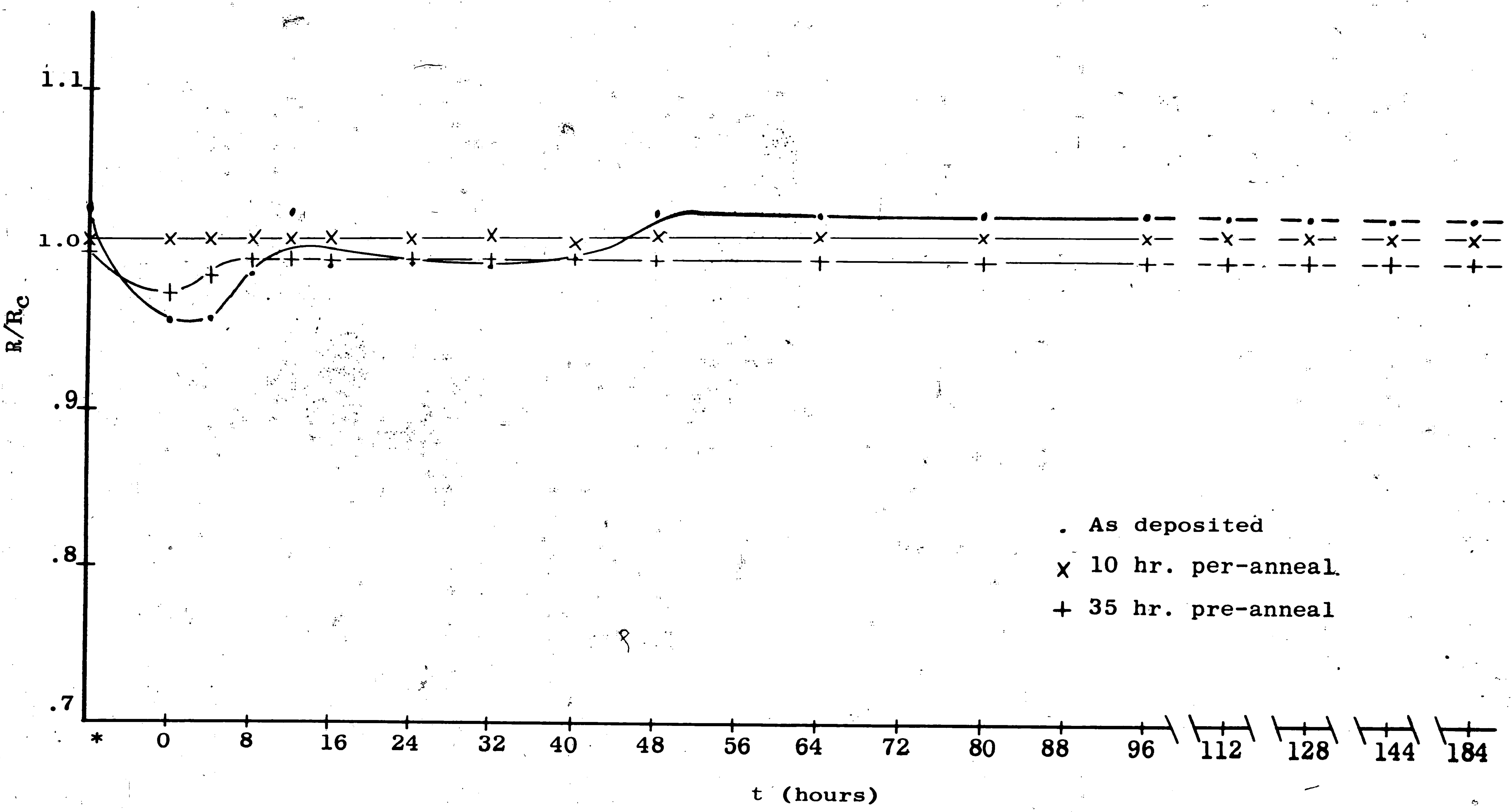

Figure 5(d). R/R_c (Average) vs. annealing time for indium specimens (* - See Figure 5(a)).

curled as they peeled from the end nearest the center of the aluminum strip and on most specimens sheared along each strip edge (see illustration in Figure 6). The curling action appeared to result from the relief of a compressive stress in the films at the aluminum strip interface.

The stress effects and shape of the curves suggested a possible relationship to creep. Recalling that at low temperatures and low stresses, the strain, ϵ , is a logarithmic function of annealing time and expressed by the equation

$$\epsilon = \alpha \log t \tag{2}$$

where α is the straight line slope on a semi-logarithmic plot.[8] Considering the fact that the resistance of a thin film is a function of its strained state,[2] equation (2) can be written

$$R = \alpha' \log t + \beta \tag{3}$$

where β is the intercept and equal to R at t = 1 and where α' is the new slope. Dividing both sides of the equation by R_c and redefining constants the equation becomes

$$R/R_c = A \log t + B \tag{4}$$

and is in the desired form.

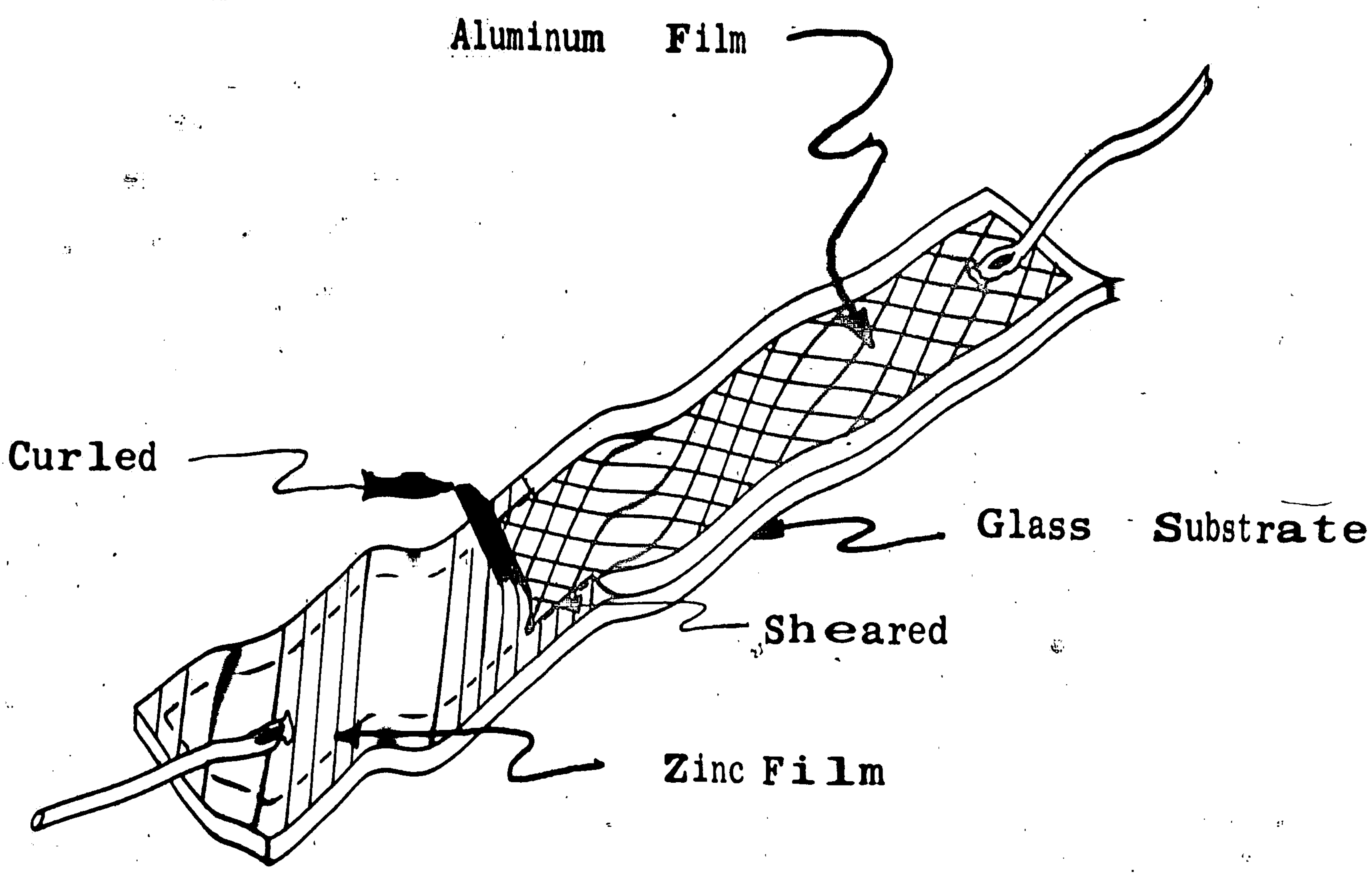

Figure 6. Illustration of zinc film peeling from aluminum strip.

The data for each zinc specimen was plotted on semi-log paper in three groups according to the equilibrium state of the aluminum strips (as deposited, 10 hour and 35 hour pre—anneal). The respective plots are shown in Figure 7 (a), (b), (c). Because the resistance of the aluminum strips originally varied between three and six ohms, a floating ordinate was utilized for compactness. Most of the data points for each specimen had straight line fits through 48 hours of anneal; however, beyond this point R/R_c became a linear function of annealing time excluding the deviation for specimens #13, 23, and 17 which resulted from peeling. The corresponding R/R_c values for specimens on each plot were arithmetically averaged, corrected to the same value of .774 at t = 0 and plotted on semi-logarithmic paper (see Figure 8). A computer regression program was used to obtain the straight line fits through 48 hours for two groups and 40 hours for the other. The specific values obtained are as follows:

Pre-anneal	Slope	Intercept	Correlation
Unannealed	6.11×10^{-3}	.8054	98.3%
10 hours	1.328×10^{-2}	.8147	98.6%
35 hours	1.962×10^{-2}	.8123	99.6%

Data were excluded from the averages for specimen #18 because of a broken lead at 48 hours and #24 which for no apparent reason did not change in resistance.

The increase in slope with pre-anneal time supports the theory of increasing resistance with increasing strain of the zinc films.

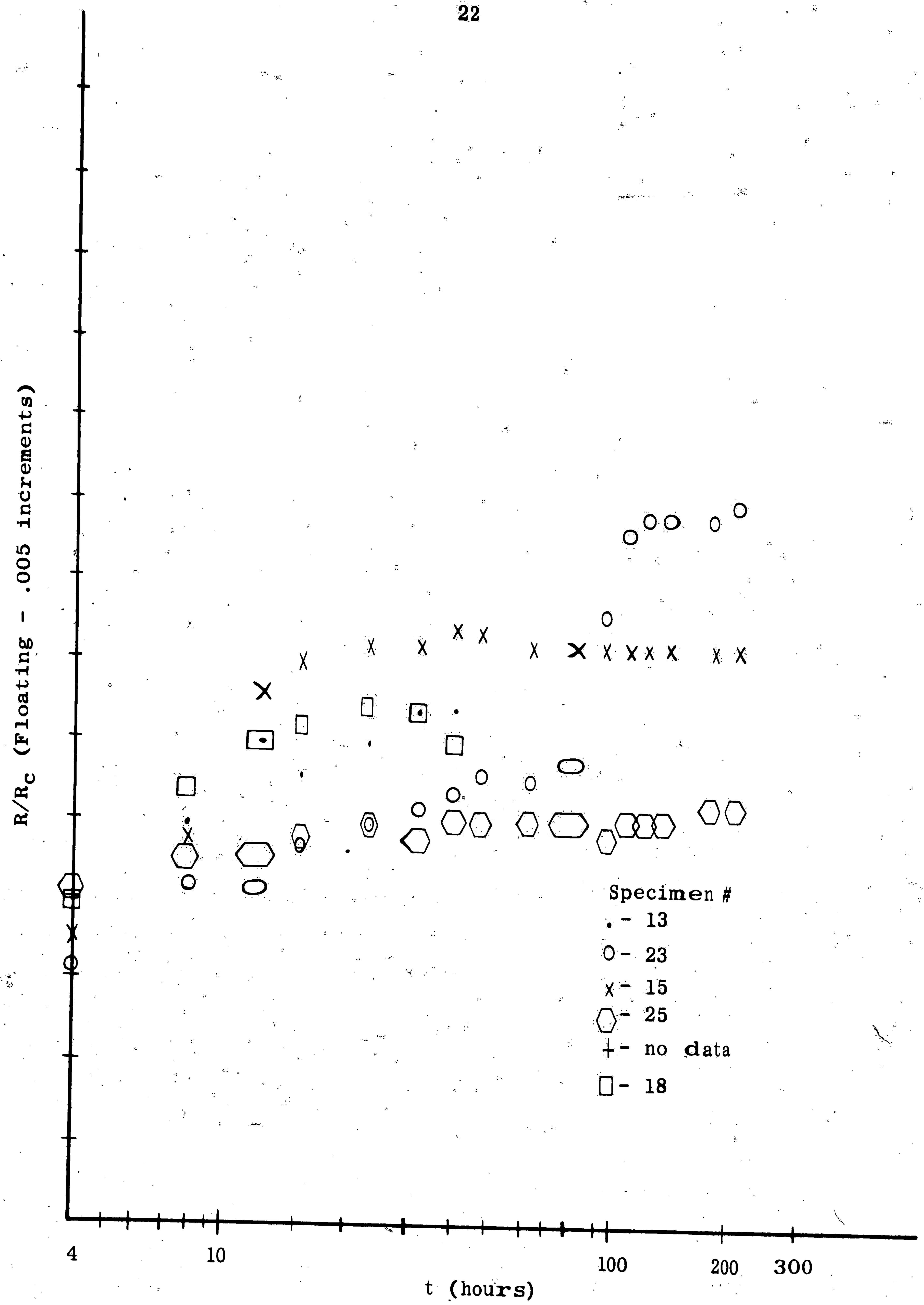

Figure 7(a). R/R$_C$ vs. logarithm of annealing time for zinc specimens with as deposited aluminum strips.

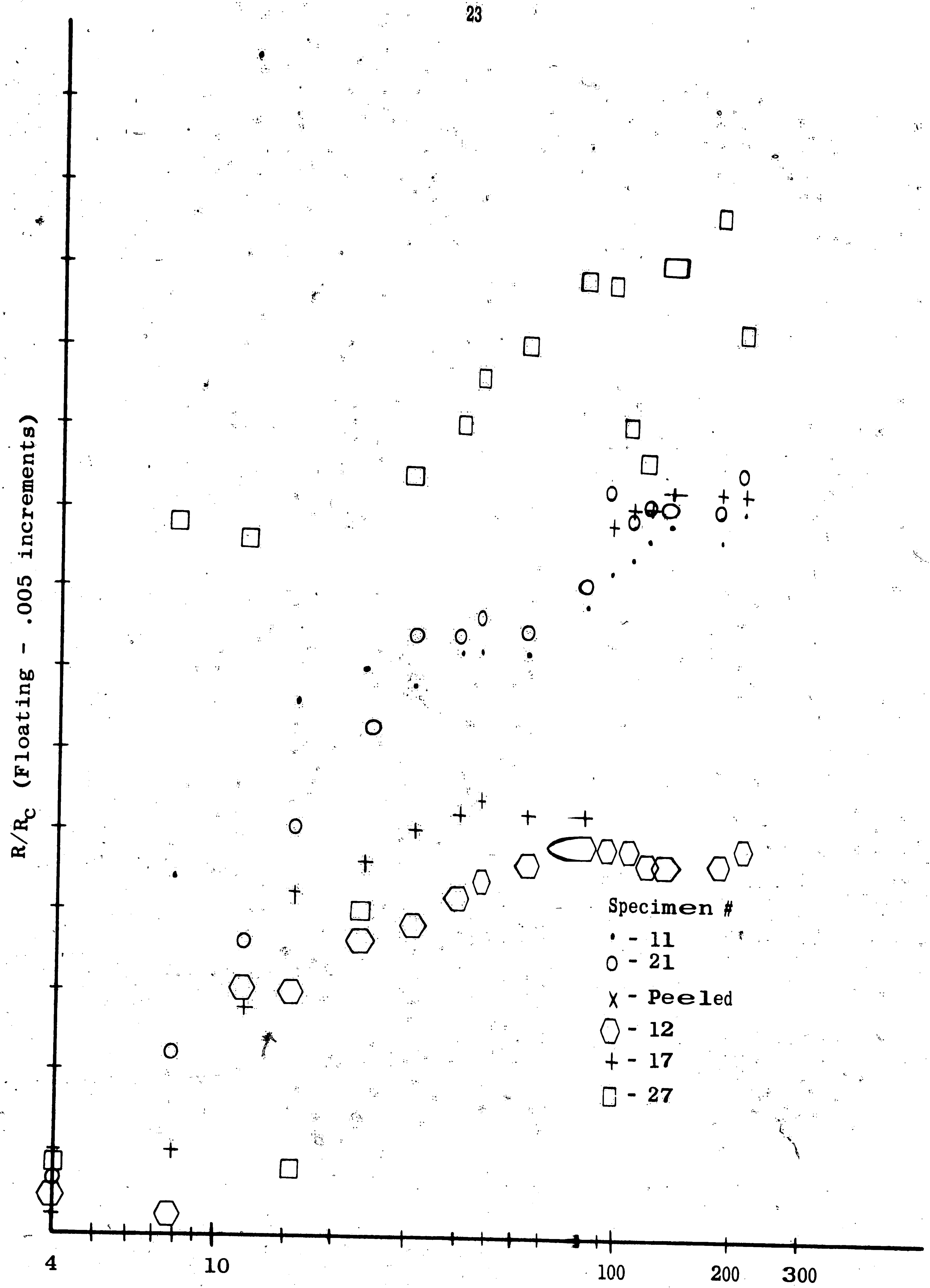

Figure 7(b). R/R$_C$ vs. logarithm of annealing time for zinc specimens with 10 hour pre-annealed aluminum strips.

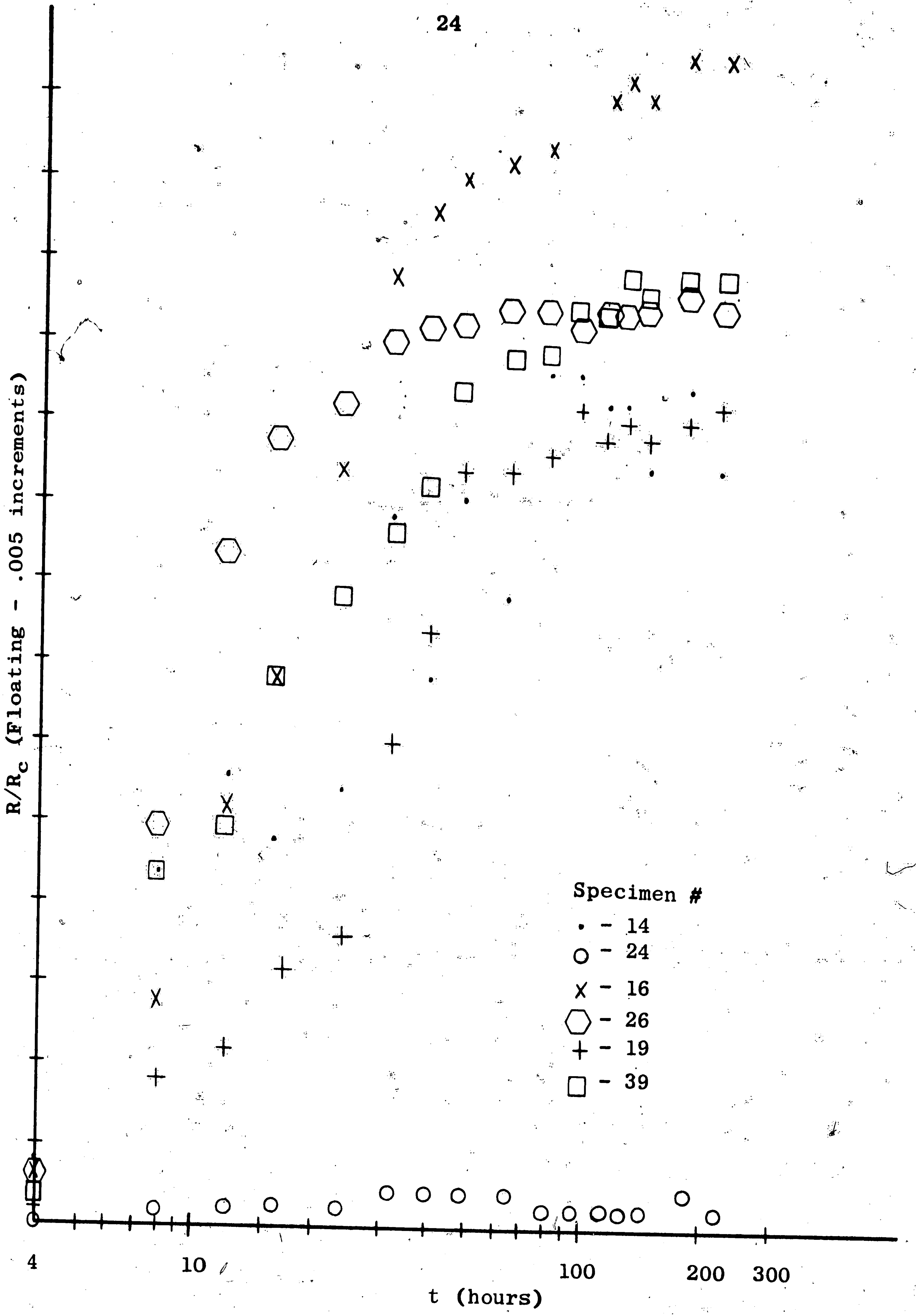

Figure 7(c). R/R_c vs. logarithm of annealing time for zinc specimens with 35 hour pre-annealed aluminum strips.

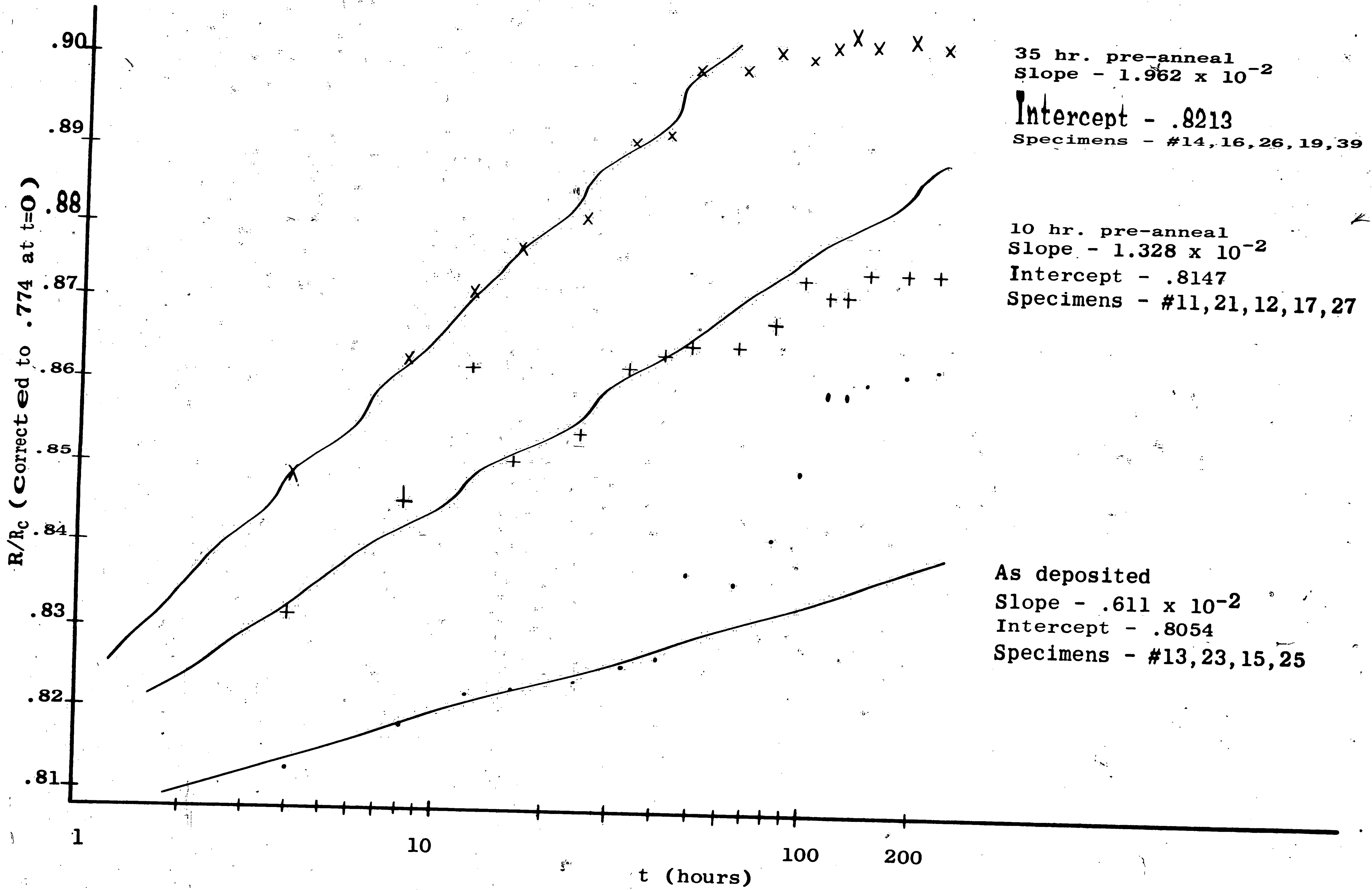

Figure 8. R/R$_C$ (Average) vs. logarithm of annealing time for zinc specimens.

First, consider the specimens with the zinc film deposited over an unannealed aluminum film. Both films will undergo recovery during an anneal, but zinc which recovers more quickly under normal conditions will be restricted by the aluminum, thus producing a small compressive stress in the zinc at the interface. The resulting strain would account for the small increase in resistance of these specimens, keeping in mind that both films if annealed separately would decrease in resistance. Now, consider the specimens with the zinc film over a partially annealed aluminum one. In this case, the restriction on the zinc film recovery begins earlier and the stress becomes greater over a given length of time. Consequently, the induced strain and its contribution to the resistance would increase more rapidly and to a greater value than the previous specimen. This corresponds to the observed increase in the slope of the semi-logarithmic plot with the annealed state of the aluminum films. Since the rate of annealing decreases with annealing time, the decrease in amount of slope change for the longer pre-anneals was expected. A graphic comparison of these two rates is shown in Figure 9. The slope values for each set of specimens are plotted against annealing time as are the average fractional resistance changes of the same specimens recorded during pre-anneals.

X-ray diffraction patterns were taken on four samples from each of the three groups using a low angle cylindrical camera. No apparent broadening of diffraction lines with increasing pre-anneal could be detected, thus indicating that the small strains present were elastic in nature.

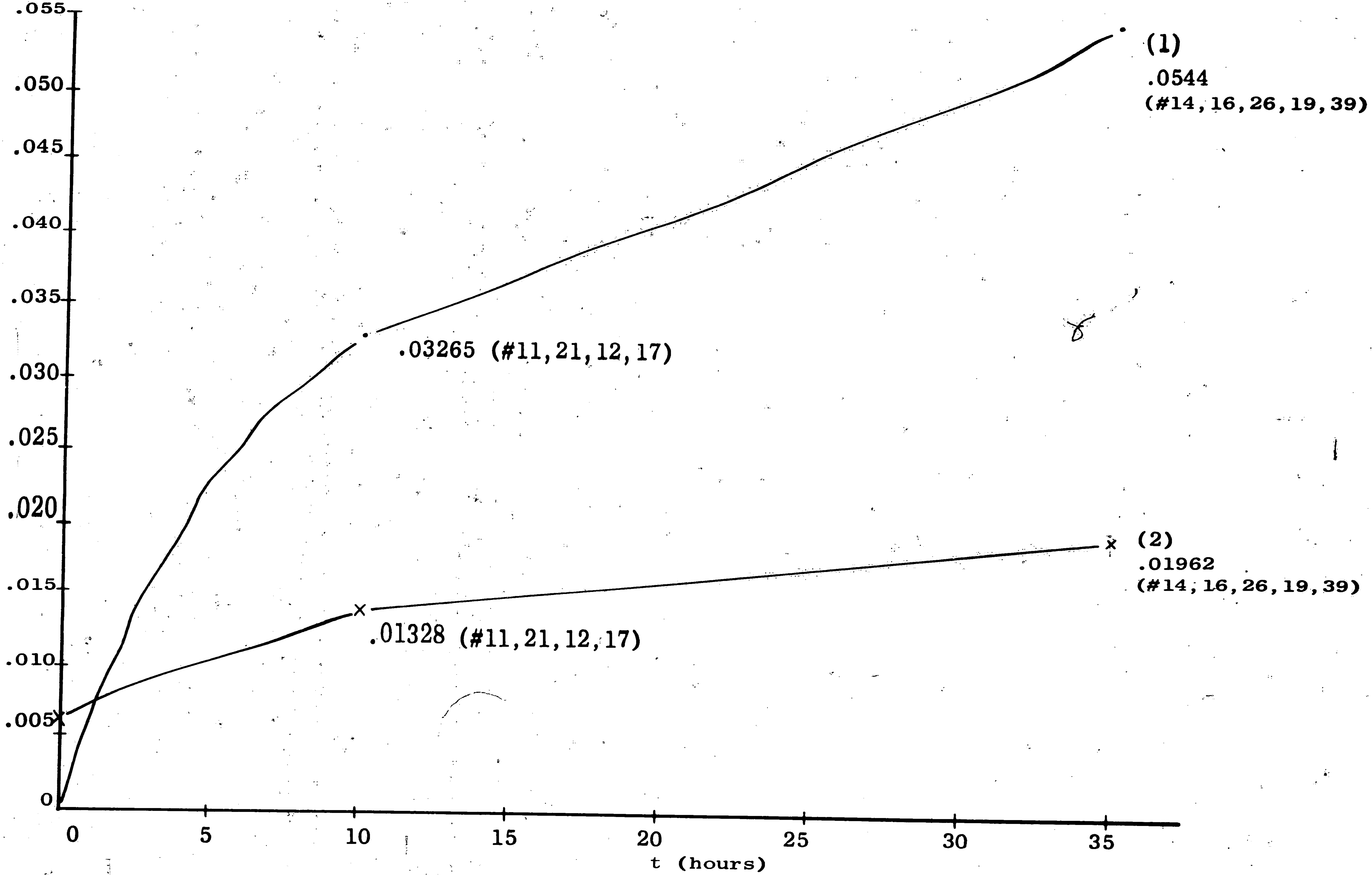

Figure 9. (1) Average fractional resistance change, $\Delta R/R$, in aluminum films vs. time of pre-anneal. (2) Slope values from Figure 8 vs. time of pre-anneal.

To evaluate the effect of current polarity on resistance behavior, an arithmetic average of corresponding data was calculated for specimens annealed under a positive dc current and another for those under a negative current. Only pairs were selected from each group to eliminate any effects due to the annealed state of the specimens. The positive average values were corrected to obtain the same R/R_c value for both at t = 0. The straight line data again obtained from a regression program is shown in the semi-logarithmic plot of Figure 10. The slope values differ only slightly, indicating that the current polarity has little effect on the resistance behavior. The distinct separation of the two lines when both were corrected to the same value at t = 0 is due to individual variation in zinc film resistance and departure from straight line correlation on the semi-logarithmic plots (i.e., specimen #15 in Figure 7(a)).

Tin Specimens

Although specimen resistance underwent no change as the direct result of tin deposition, a substantial decrease occurred during the first 8 hours of anneal. As for zinc, it is believed that this initial decrease was the result of conduction through the oxide by electrons of diffused tin atoms. The relative larger atomic size (i.e., atomic, ionic and covalent radii) of tin would support a decreased diffusion rate and an explanation for the delayed resistance decrease. An additional distinction was apparent in the magnitude of average resistance changes for specimens with different pre-anneals.

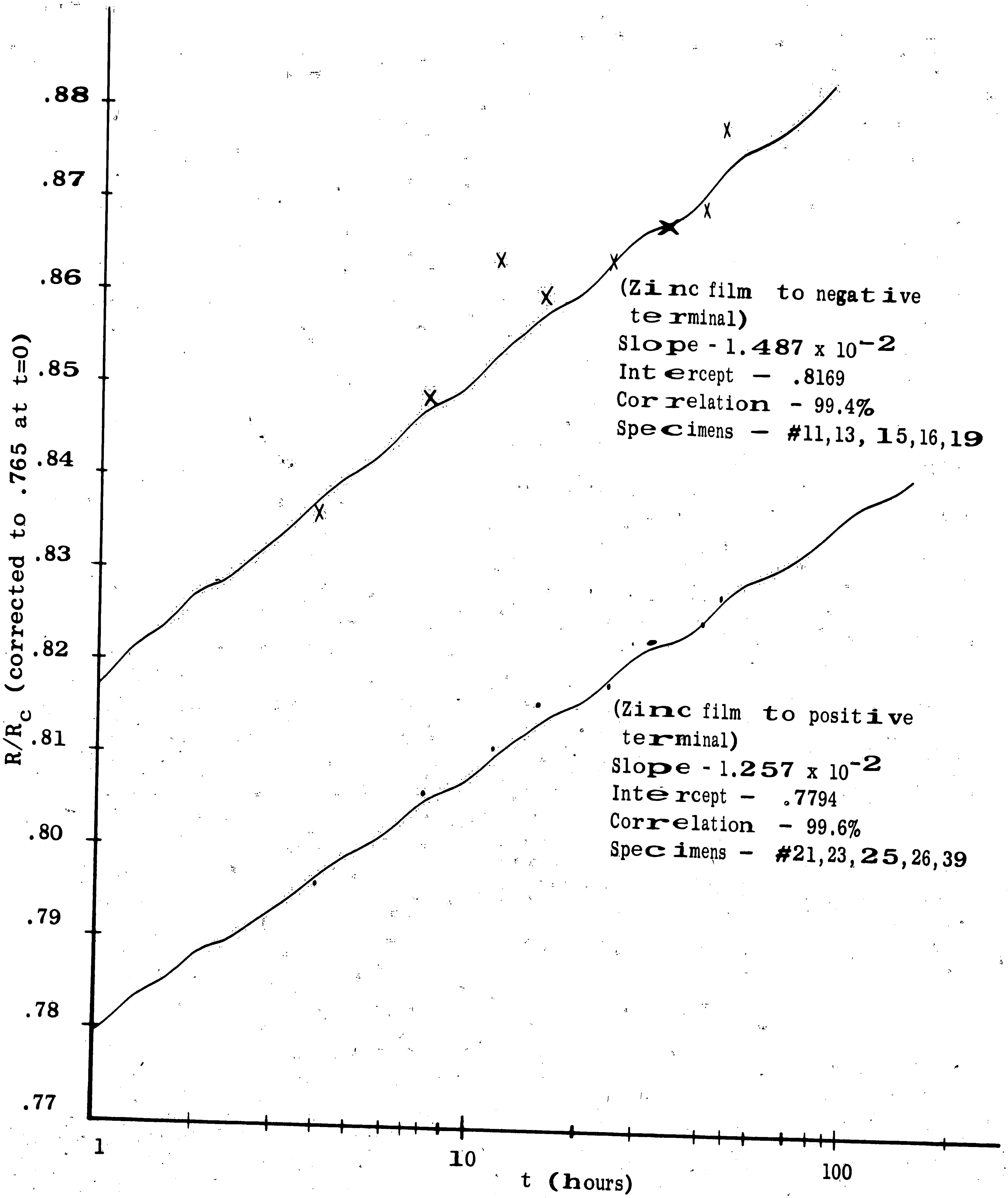

Figure 10. Average R/R_c values for zinc specimens under positive and negative dc current vs. annealing time.

Unlike zinc, where the average total decrease in resistance was approximately constant, tin specimens decreased with increased pre-anneal time. This would tend to verify the existence of a thicker oxide layer on aluminum strips with longer pre-anneals. Consequently, at any given time during the anneal the percent of tin atoms and conduction electrons in the total volume of oxide would be smallest in the specimens pre-annealed for 35 hours. Therefore, the combined resistance of the tin film and semiconducting oxide would be greatest for these specimens and result in the smallest decrease on paralleling with the aluminum strip.

An accurate interpretation of the resistance behavior of tin specimens between the twelfth hour and anneal termination was impossible because of erratic resistance readings from most specimens. The inconsistencies were due to resistance drift during measurement and since the amount of drift ranged from zero to better than twenty-five percent for most specimens, a logical explanation could not be found. It was noted, however, that the specimens previously unannealed experienced the least drift and consequently produced the most consistent data (see Figure 11). This would point to the oxide as a possible source of the drift. An increase in resistance with time beginning at 8 hours into the anneal was also apparent from the data. A difference in the data for each pre-annealed state was evident, but a comparison like that for zinc was not attempted for reasons given above.

The polarity of dc current flowing through the tin specimens during anneal also had little effect on their resistance behavior

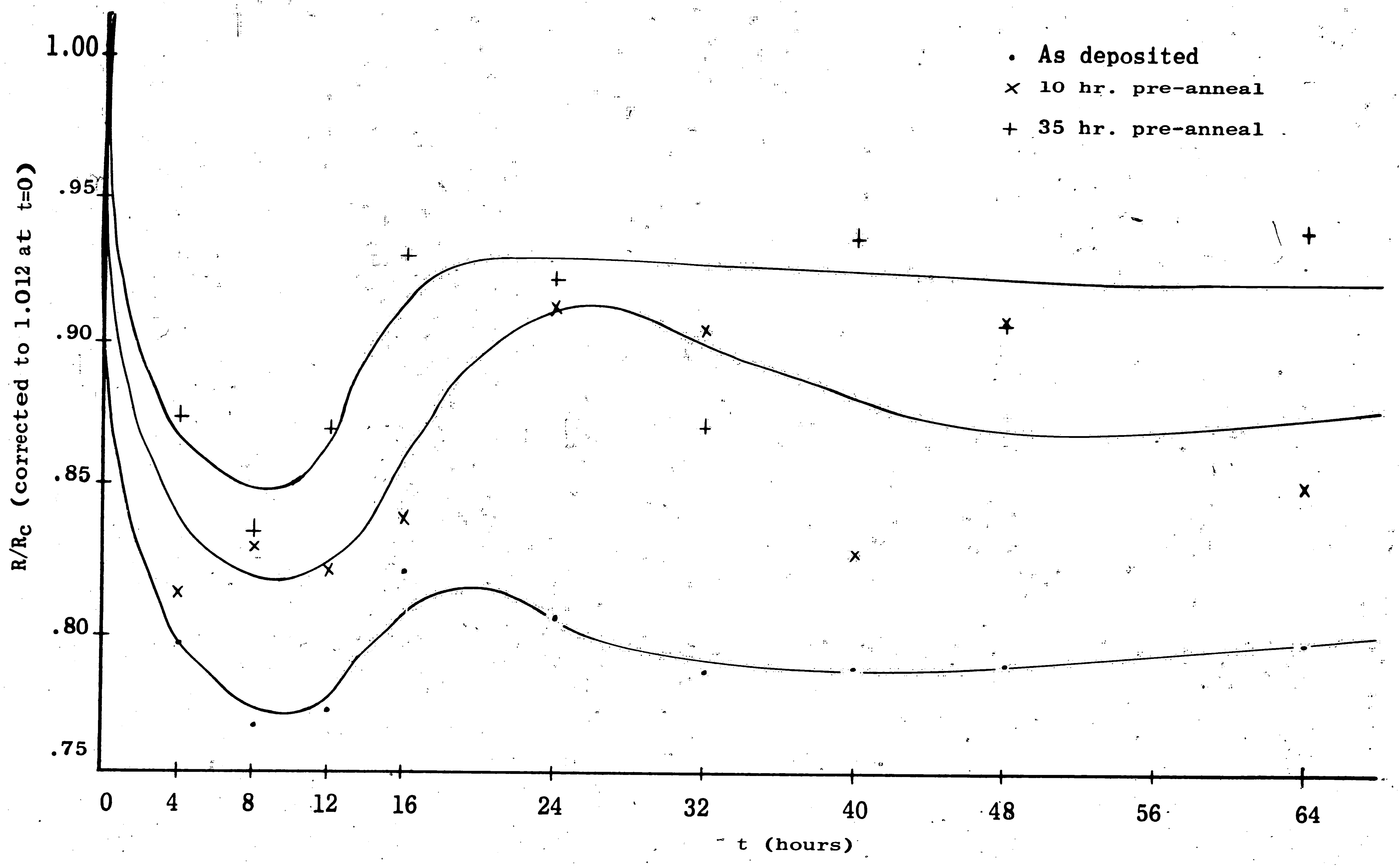

Figure 11. R/R_C (Average) vs. annealing time for tin specimens.

(see Figure 12). Average R/R_c values, Table 6, for each current polarity were obtained in the same manner as those for zinc; however, a standard plot was used because of the nature of the data. Here again, the results may be questionable because of resistance drift during measurement.

Cadmium Specimens

Since the resistance of specimens coated with cadmium did not change with time except for self-annealing of the aluminum strips, it is apparent that a conduction path was not established between the two films. Assuming here as was done for zinc and tin that the aluminum oxide layer containing sufficient current carriers would provide such a path, two possible explanations are proposed. One, cadmium atoms did not diffuse into the oxide in sufficient quantities during the anneal; or two, sufficient cadmium atoms were present but their electrons were not available for electrical conduction. Since the ionization properties of zinc and cadmium are similar, the second proposal would appear unlikely. However, the rate of diffusion of cadmium into the oxide is expected to be less than zinc because of atomic size differences. The atomic, covalent and ionic radii of cadmium are all greater than those of zinc. The respective values[9] are 1.54, 1.48 and .97 Å for cadmium as compared to 1.38, 1.31 and .74 Å for zinc. Except for an atomic radius of 1.62 these values are also greater than those of tin. If the atomic radii were a major controlling factor of diffusion in the oxide, tin might be expected to behave like cadmium; but it must be remembered that each tin atom would provide twice as many valence electrons.

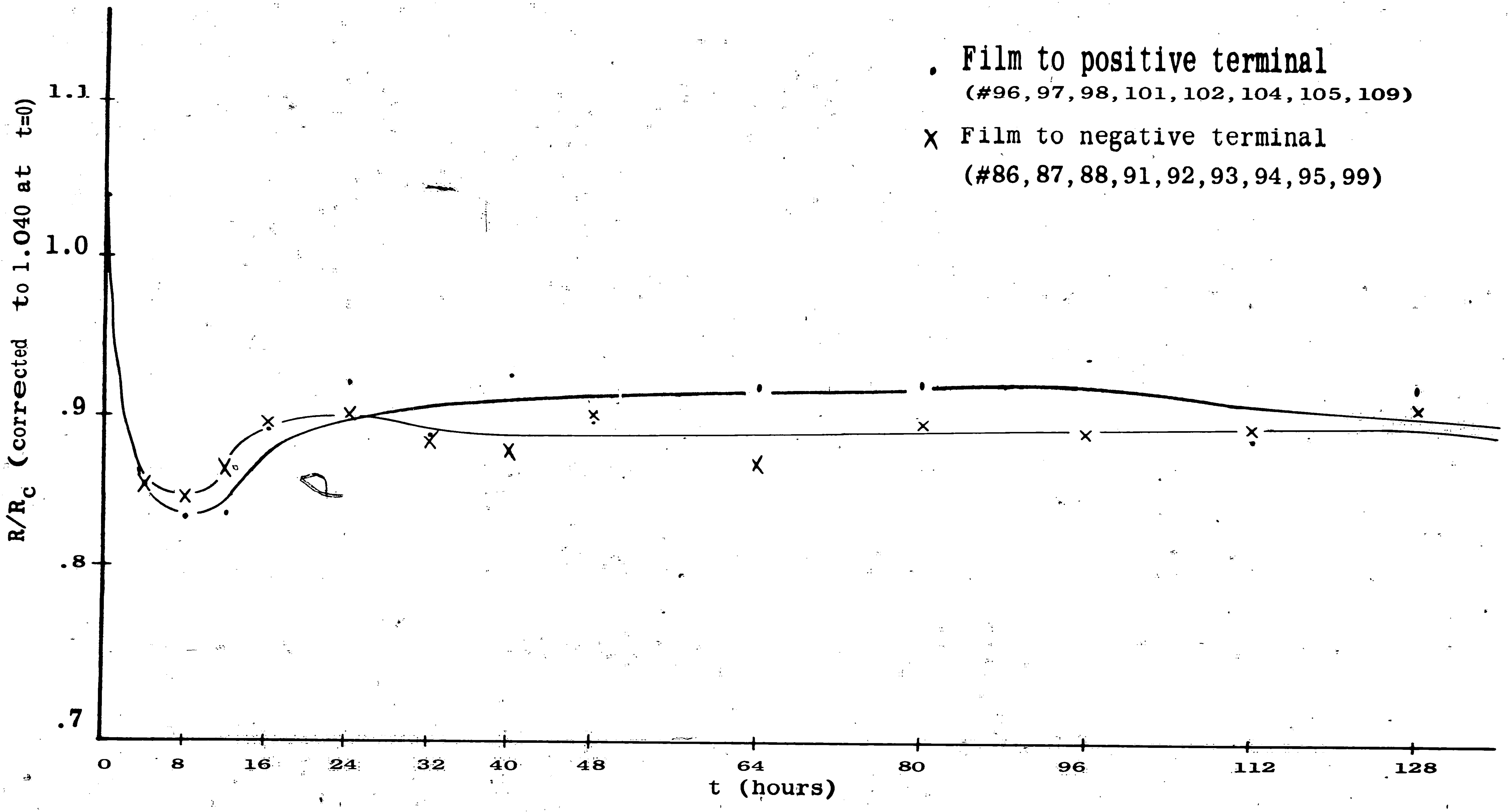

Figure 12. Average R/R_c values for tin specimens under positive and negative dc current flow vs. annealing time.

Indium Specimens

With some expansion, the same explanation is proposed for the resistance behavior of the indium specimens where a similar pattern was observed exclusive of four samples. For each exception drift like that for tin was observed during resistance measurements. Also, three of the four specimens were of the "as deposited" group where the aluminum oxide layer was believed to be thinner. The atomic sizes of indium are slightly greater than those of tin (i.e., 1.66 to 1.62 Å for atomic radii, 1.44 to 1.41 Å for covalent radii and .81 to .71 Å for ionic radii) and indium has one less valence electron per atom. Consequently, these property differences would provide a possible explanation for conduction in only those specimens where thinner oxides were expected.

CONCLUSIONS

The conclusions from this investigation are summarized as follows:

1. Effect of the four alloying elements of Shinkalloy on the resistance of aluminum thin films during annealing.
    - a. Zinc when deposited on aluminum films produces a decrease immediately on deposition and a subsequent increase on annealing. The increase follows a logarithm of time function through the initial 48 hours.
    - b. Tin has no effect upon deposition but produces a distinct decrease followed by a lesser increase on annealing. Tin also produces resistance drift during measurement.
    - c. Cadmium has no apparent effect either upon deposition or during annealing.
    - d. Indium produces an intermediate decrease with resistance drift only on as deposited aluminum films upon deposition and during early stages of annealing.
2. Effect of equilibrium state of aluminum film on the resistance behavior.
    - a. The rate and magnitude of the resistance increase (slope of semi-logarithm plot of resistance vs. time) for zinc on aluminum increases with increased aluminum film pre-anneal.
    - b. The rate and magnitude of the resistance decrease for tin on aluminum decreases with increased pre-anneal.

   c. Since no resistance change occurred in the case of cadmium no evaluation could be made.

   d. Indium produces resistance changes only on unannealed films.

3. The polarity of dc current through the films during the anneal has no effect on the resistance behavior.

SUGGESTIONS FOR FURTHER STUDY

A quantative evaluation of the apparent strain effects between zinc on aluminum films could be made using electron diffraction and metallographic techniques. A study of the effects of aluminum on the resistance of aluminum would be helpful. Also the temperature dependence of the resistance behavior with these elements (zinc, tin, cadmium and indium) on aluminum could be determined by varying the annealing temperature.

The oxide layer apparently plays an important part in the observed resistance behavior of the combined films, therefore, a similar study utilizing controlled oxide thickness and tracer elements should provide quantative information on diffusion in and around the aluminum oxide.

APPENDIX I

Preparation of Aluminum Thin Film Specimens

Substrate Preparation

The 3" x 2" glass substrates required by design were obtained by cutting commercially available 2" x 3", 1.27 mm thick glass microscope slides (Corning 7059) into ten segments. This was accomplished with minimum breakage using a specially designed fixture and a diamond scribe lubricated with kerosene.

The substrates were cleaned in a detergent solution agitated by ultrasonics as detailed below :

1. Washed in a solution of Alconox in de-ionized (DI) water with ultrasonics for 1 minute (to remove kerosene used as lubricant in cutting glass).
2. Rinsed in running DI water.
3. Repeated (1) in new solution for 2 minutes.
4. Rinsed in Running DI water.
5. Immersed in boiling DI water for 5 minutes.
6. Rinsed in boiling DI water and removed slowly.
7. Dried in a stream of dry nitrogen gas.

To insure the best possible film adherence a final cleaning was administered with a glow discharge in the vacuum chamber during pumpdown for evaporation.

Evaporation of Aluminum

Equipment and Fixtures

A commercial 18" bell jar oil diffusion vacuum unit fitted with an MRC ring and safety features for high voltage glow discharge was used as the evaporation system (see Figure 13).

Evaporation fixtures (stands, holders, etc.) required for the simultaneous deposition of aluminum equally on ten substrates were designed of aluminum sheet. The holders were made up to accept 2" x 3", 1.27 mm thick glass substrates or segments thereof and a .015" thick stainless steel deposition mask. Ten of these holders could then be positioned on the stand, each at a distance of 12" from the point evaporation source. The masks restricted deposition on each substrate to ten .175" x 1.9375" strips. All fixtures described here are shown in Figure 14.

A resistance heated tungsten filament of four strand .030" diameter wire was found to be the most effective point source for evaporation with these fixtures (see Figure 15). A similar filament of spiral design provided the heat for degassing the fixtures prior to evaporation.

Evaporation Procedures[10]

Ten slide segments were placed in each of nine substrate holders directly after cleaning and the deposition mask fixed in position. The tenth holder was similarly readied with a 2" x 3" slide and all units stored in a desiccator until mounted on the evaporation stand inside the chamber. The following detailed procedure was then used to deposit

Figure 13 - Bell jar vacuum system and high voltage power supply used for evaporation of aluminum in preparation of thin film specimens.

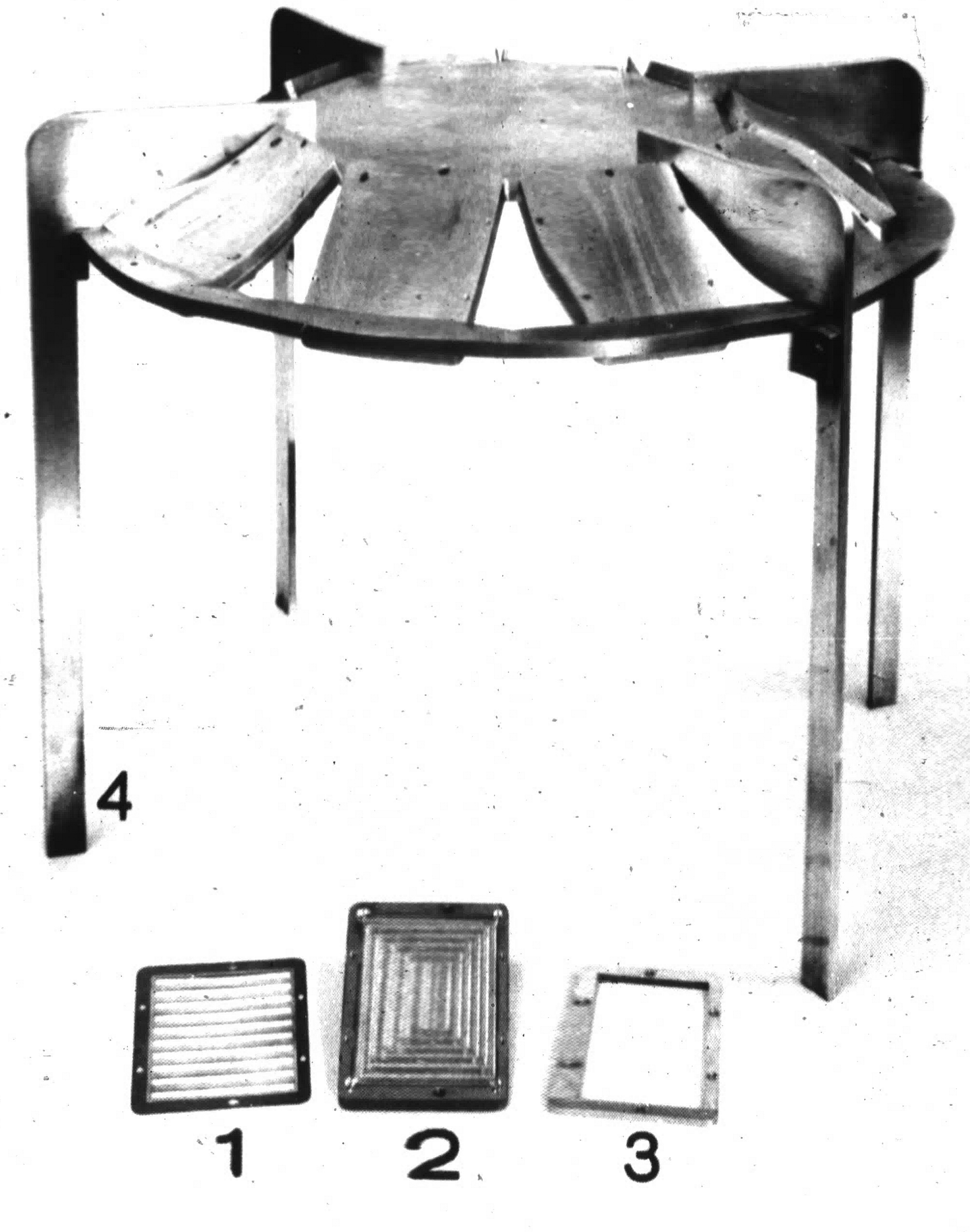

Figure 14 – Evaporation fixtures used with vacuum system for preparation of thin film strips. (1) deposition mask, (2) and (3) glass substrate holder and frame, (4) positioning stand for 10 holders.

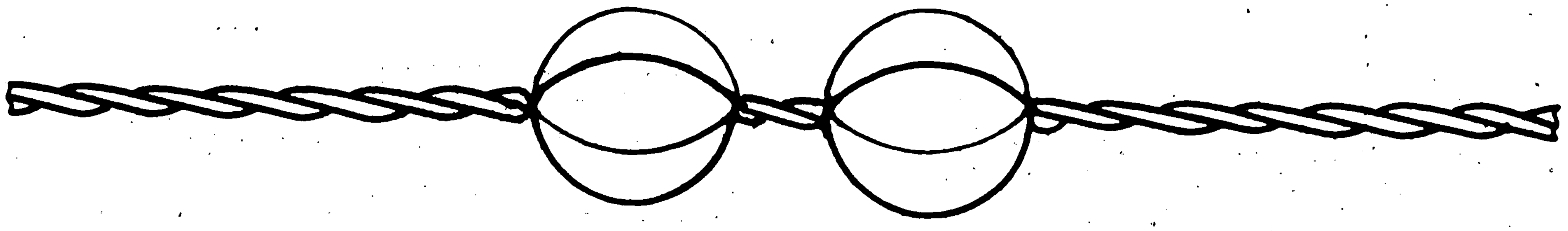

Figure 15. Point source evaporation filament of tungsten wire used for deposition of aluminum.

approximately 1500 angstroms of aluminum on each substrate:

1. Placed four 3/8" pieces of .075" 99.9% pure aluminum wire shaped as "horseshoes" over filament. Pieces were ultrasonically cleaned in reagent alcohol.
2. Removed any dust that may have collected on substrate during loading with Effa Duster.
3. The bell jar was closed and roughed down to 100 microns of Hg.
4. Closed roughing valve, opened foreline valve and cracked high vacuum valve until pressure stabilized at 90 microns.
5. Set glow discharge power supply at 1.1 Kv. with a current of 40 milliamperes.
6. Allowed 15 minutes for cleaning of substrates by ionized residual gases and then opened high vacuum valve fully.
7. Set heater current at 54 amperes (AC) and allowed substrate temperature to reach 150°F and degas fixtures (approximately 1 hour).
8. Turned heater off and allowed substrate temperature to cool to 105°F (approximately 2½ hours). Pressure at 6 x 10^{-7} mm of Hg.
9. Set filament current at 50 amperes (AC) until aluminum melted. Raised current to 100 amperes and opened shutter.
10. Evaporated until hot filament could no longer be seen through test glass slide hanging on rim of fixture (1.5 min.). Pressure increased to 2 x 10^{-5} mm of Hg.
11. Closed shutter and turned off filament.
12. Closed high vacuum valve and cracked air vent.

Since the temperature of each substrate during evaporation would have been difficult to obtain, a representative valve was obtained by placing a chromel/alumel thermocouple in contact with the back of one substrate through a hole in the holder. Although no actual checks were made, this value should be close to the true temperature for two reasons. One, all holders were tied together as a unit by the aluminum fixture which provided good thermal conduction, especially in the vacuum, and two, the cooling time of 2½ hours was sufficient for this unit to reach thermal equilibrium. The thermocouple was calibrated at 0 and 100°C and its millivolt potentials measured with a potentiometer.

Film Thickness Measurements

All thickness measurements were made with a multiple beam optical interferometer using standard techniques and an opaque aluminum film to provide the reflecting surface. The thickness of the test specimens was estimated by measuring the tenth substrate which was not segmented. Following deposition of the strips a second continuous opaque aluminum film was applied over the entire slide. Measurements were then made at three positions (top, center, bottom) of strips #2, 5 and 9. These values are given in Table 1. To determine the uniformity of thickness obtained from deposits at various points around the evaporation fixture, strips were simultaneously deposited at five positions (2, 4, 6, 8, and 10) with position 10 perpendicular to the filament. A postcoat was applied to each substrate and the thickness measured at the center of strips 2, 5 and 9 or ten. The average of four readings taken at the same point are given in Table 2.

TABLE 1

Film Thickness (Å) at Several Points on Single Substrate

Strip #	Top	Center	Bottom
2	1480	1516	1460
5	1652	1625	1636
9	1536	1576	1528

TABLE 2

Film Thickness (Å) on Substrates at Various Positions on Evaporation Stand

Strip #	2	4	6	8	10
2	1036	972	1072	1088	1096
5	1068	984	1128	1060	1160
9 or 10	1156	996	1068	1088	1128

Electrical Lead Attachment

Electrical leads for resistance measurements were attached to the aluminum thin film strips by resistance heated ultrasonic welds. This method permitted the attachment of .035" diameter high purity aluminum wire to each end of the strips, thus providing a low resistance contact and eliminating the need for an additional termination element, (See Figure 16).

The equipment used was a commercial 100 watt 60 KC ultrasonic welder equipped to preheat the leads with ac current to the hot working temperature. Parameters which gave the desired bonding properties were experimentally determined and are listed below:

Welder Parameters	Heating Parameters
1. Power setting - 10 low	1. Variac setting - 50
2. Weld time setting — 1	2. Heat time - .7 sec.
3. Horn pressure setting - 7	

Small variations in power setting (7-12) and heat time (.5-.7 sec.) were required to obtain optimum bond properties on each strip.

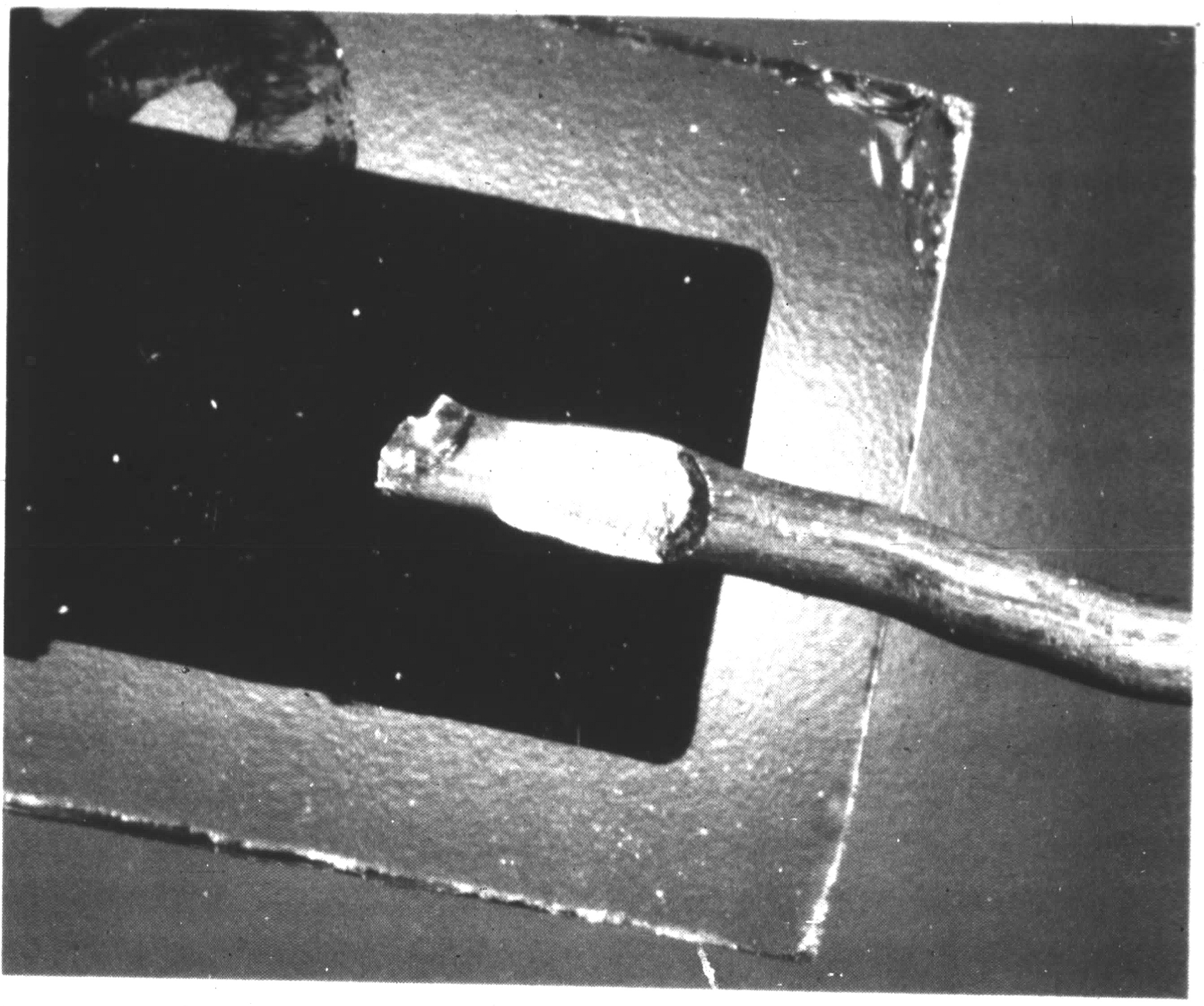

Figure 16- Aluminum wire lead attached to aluminum thin film strip with ultrasonic weld.

APPENDIX II

Vacuum Evaporation of Sn, Zn, Cd and In[10]

The bell jar vacuum evaporation system described in Appendix I was used to evaporate a 2000-3000 Å film of tin, zinc, cadmium or indium onto specified aluminum film strips. A fixture capable of accepting a single 2" x 3" substrate or 10 segments thereof was used to position the specimens 5" from the evaporation source. A mask of 1/16" thick aluminum sheet was used to expose 3/4" of the aluminum strips to the vapor (see Figure 17).

A total of 18 strips were coated with each element, two each from the nine substrates. Therefore, the deposition of each element was done in two evaporations on 10 and 8 segments, respectively. Consistent adherent films of Sn and In were obtained with reasonable rates. However, rates much greater than normal were required to obtain satisfactory nucleation of Zn and Cd on the aluminum strips.

The general evaporation procedure used for all elements was as follows:

1. Loaded chamber with specimens and metal to be evaporated.
2. Roughed chamber to a pressure of 60 microns Hg.
3. Opened high vacuum valve.
4. When pressure reached a value between 2×10^{-6} and 4×10^{-6} mm Hg, filament current was gradually increased until metal was molten.
5. Current was increased to desired level for evaporation, and shutter opened.

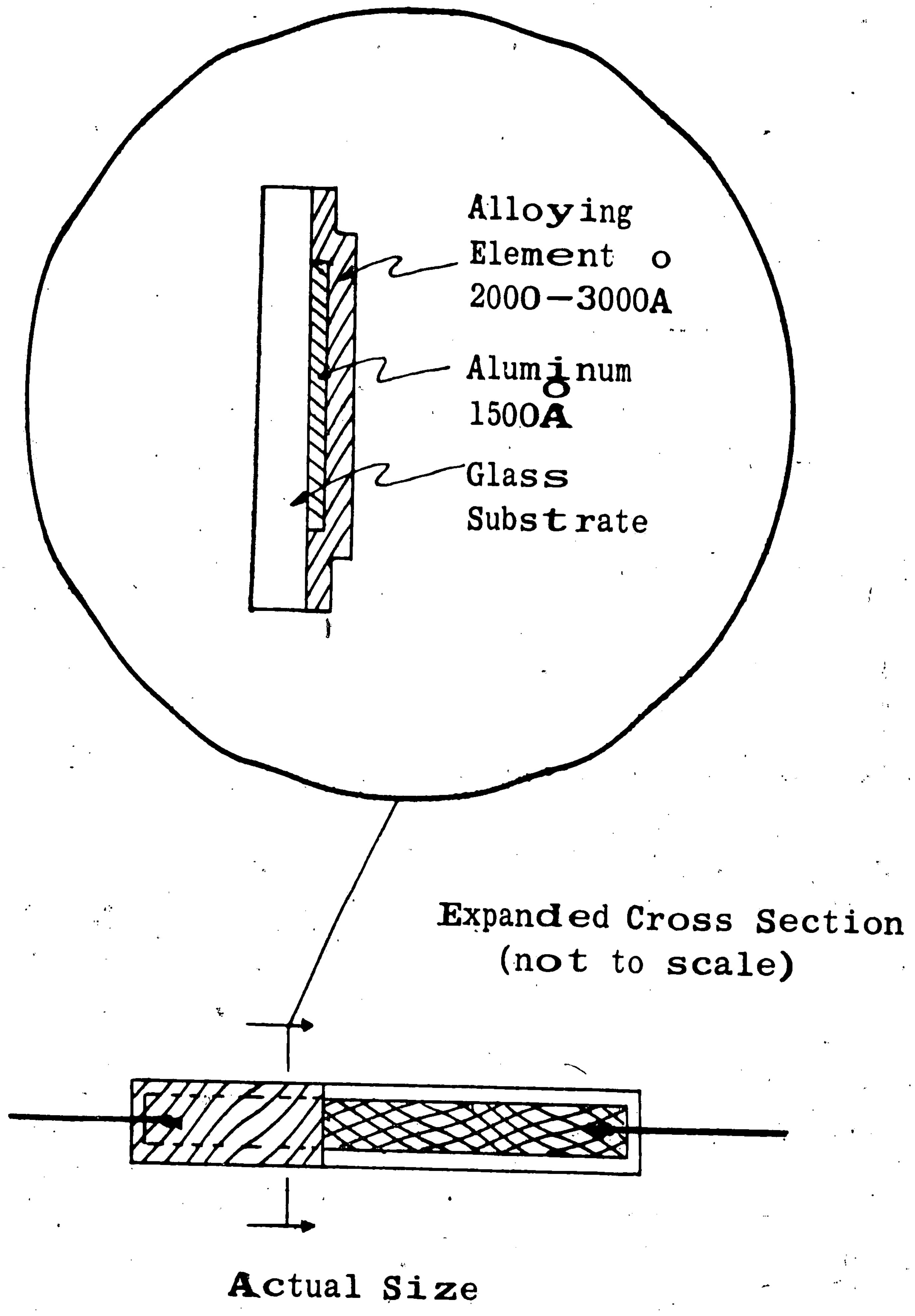

Figure 17. Illustrated appearence of specimen following deposition of alloying element.

6. Closed shutter after required exposure.

7. Turned current off, closed high vacuum valve and released vacuum.

Specific parameters and other information are tabulated below:

Metal	Purity	Source	Pressure mm Hg.	Evaporation Parameters Current AC AMPS.	Time Seconds
Tin	99.999	Tungsten Boat	3×10^{-6}	180	45
Indium	99.999	Tantalum Boat	2×10^{-6}	120	90
Zinc	99.999	Alumina Crucible	3×10^{-6}	40	75
Cadmium	99.999	Covered Tungsten Boat	4×10^{-6}	150	60

The specimens were not cleaned prior to deposition except for dust removal with an Effa Duster. This was to preserve the aluminum oxide coat for reasons given in experimental procedure.

A supplement to the procedure was required for zinc due to circumstances. The zinc film deposited on four specimens (#'s 12, 15, 27 and 18) was spotty from apparently poor nucleation. Consequently, to obtain a continuous film the same procedures were used to deposit a second coat over the first on three of the specimens. The fourth one (#18) was left as deposited for observation during annealing.

APPENDIX III

Annealing Equipment

Annealing of specimens prior to and following deposition of alloying elements at 105 and 86°, respectively, was carried out in a forced-convection oven. An on-off type controller with a 100 ohm platinum resistance element maintained the oven temperature at set value ± 1°F. Prepurified nitrogen fed to the oven at a rate of 2 cu. ft./hr. provided the dry annealing atmosphere. The complete annealing setup is shown in Figure 18.

A circuit was designed to pass a current of 10 ma at 250 volts through each of 90 specimens during anneal and adapted to the oven using plugable units for ease in removing specimens for resistance measurements. For the actual experiment where 81 specimens were annealed a current of 11 ma at 260 volts was used. Each plugboard held nine specimens of the original ten segment substrates attached through their electrical leads by spring loaded clips. They were arranged on the board so that the current flow through the two specimens containing the same alloying element had opposite polarity (see Figure 19). Power was supplied by a 1.25 ampere 300 volt dc power supply with .1% current control. A block diagram of the electrical circuit is shown in Figure 20.

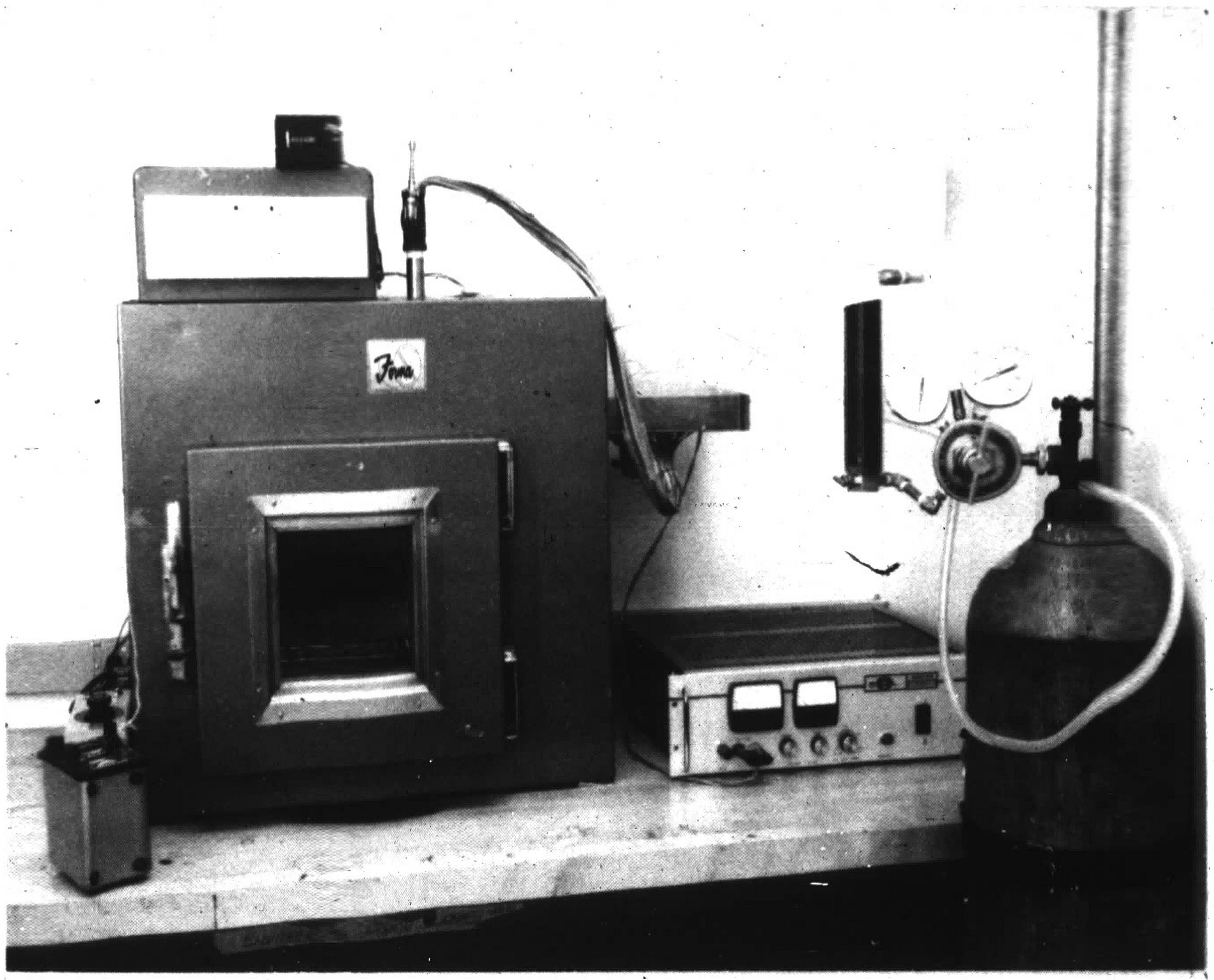

Figure 18- Annealing Equipment: oven, power supply, temperature controller and prepurified nitrogen cylinder.

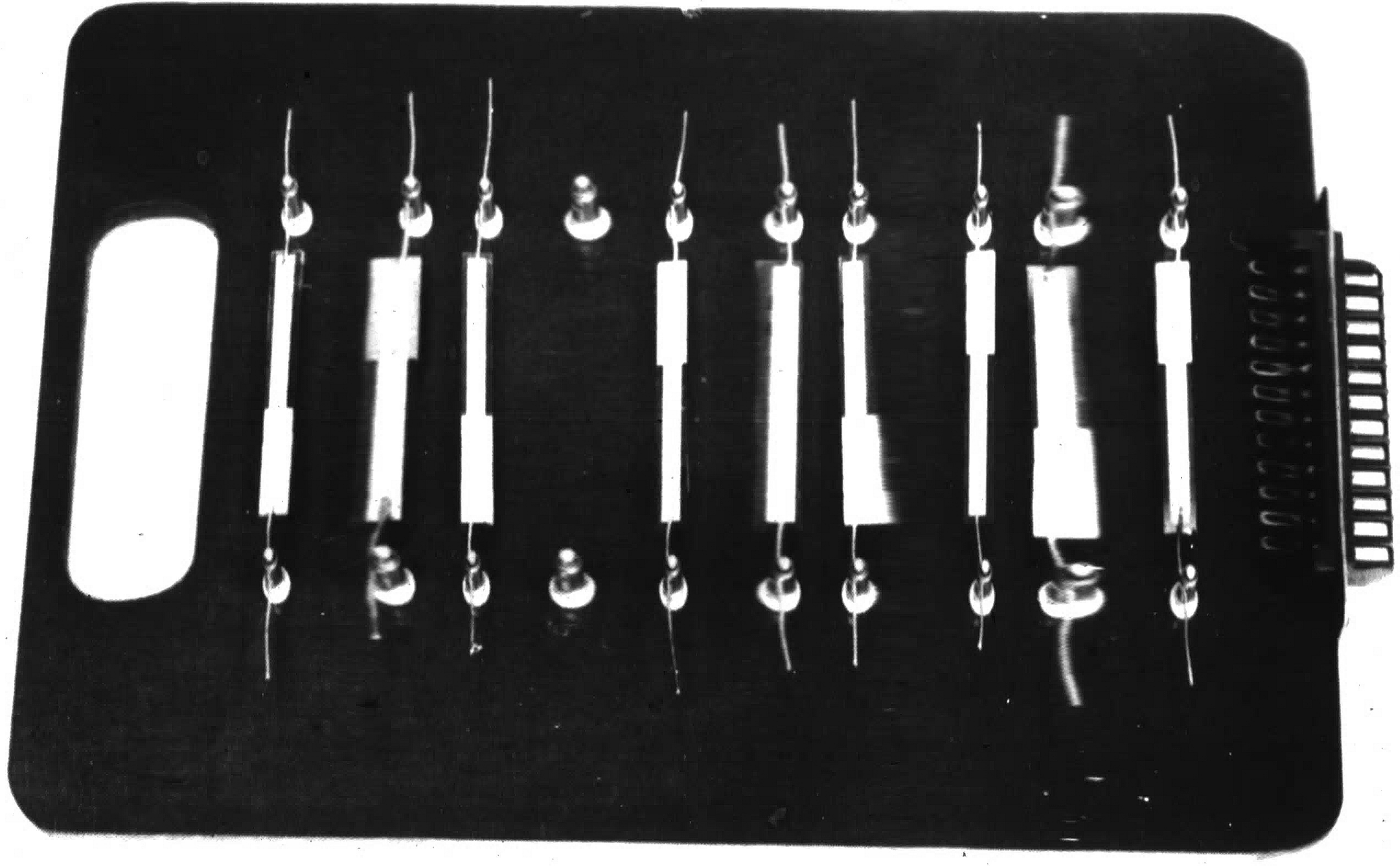

Figure 19- Arrangement of specimens on Plugboard from left to right 1, 2 - zinc; 3, 5 - cadmium; 6 - control; 7, 8 - Indium; 9, 10 - tin.

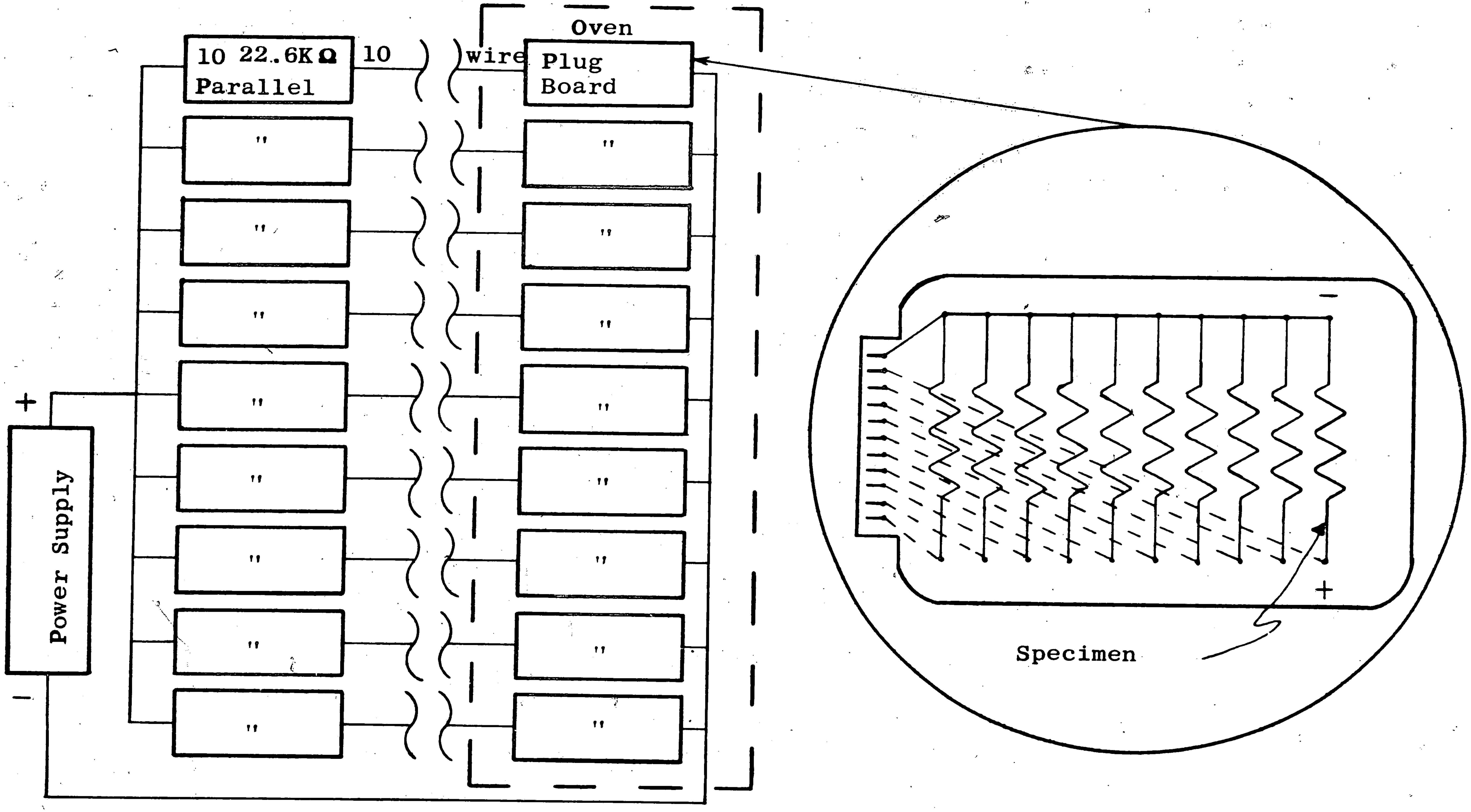

Figure 20. Block diagram of electrical circuit used to apply dc voltage to test specimen during the anneal. Ten specimens on each board each in series with a 22.6K ohm 5 watt resistor. All 90 series combinations in parallel with power supply.

APPENDIX IV

Experimental Data Tabulations and Calculations

Calculation of zinc film resistance from specimen resistance was with the equation

$$R_Z = \frac{R_B R_A}{R_B - R_A}$$

where R_B is the resistance of the aluminum strip before deposition of zinc and R_A is the resistance of the combined films following deposition. The recorded values of R_B and R_A and the calculated values of R_Z are given in Table 3.

TABLE 3

CALCULATED RESISTANCE FOR ZINC FILMS (R_Z)

(10^{-3} ohms)

Specimen Number

	11	21	12	22	13	23	14	24	15	25
R_B	4253	4176	4115	3963	4114	4060	4000	3957	4507	4309
R_A	3101	3256	3475	2895	3049	2941	3025	2768	3850	3023
R_Z	11448	14779	22343	10742	11778	10671	12410	9212	26411	10129

Specimen Number

	16	26	17	27	18	28	19	34
R_B	4535	4490	4948	5043	4886	--	4355	4351
R_A	3154	3259	3565	3945	4280	--	3277	3167
R_Z	10357	11887	12755	18119	34508	--	13239	11638

TABLE 4

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	(d)	(f)	(e)	4	8	12	16	24	32	40
11	Zn	-	4390	4253	3101	3200	3244	3447[1]	3278	3278	3273	3299
21	Zn	+	4306	4176	3256	3355	3406	3432	3447	3466	3488	3508
31	Cd	-	4244	4105	4110	4108	4117	4116	4103	4097	4096	4113
41	Cd	+	4265	4123	4126	4127	4134	4134	4122	4115	4116	4132
51	Control		4214	4082	4085	4089	4096	4095	4082	4073	4072	4093
61	In	-	4292	4142	4140	4147	4154	4154	4142	4132	4134	4147
81	In	+	4327	4168	4174	4182	4188	4188	4173	4162	4167	4185
91	Sn	-	4436	4270	4264	3600*	3945*	3500	3652*	3924*	3855*	3621*
101	Sn	+	4429	4240	4238	3700*	3749*	4043	3703	4183	4213	4230
12	Zn	-	4237	4115	3475	4032	4053	4058	4074	4088	4082	4104
22	Zn	+	4108	3963	2895	3971	3992	3961	3951	3956	3948	3958
32	Cd	-	4129	3982	3988	3991	4012	3983	3973	3977	3968	3982
52	Cd	+	4194	4037	4040	4046	4071	4048	4037	4043	4037	4045
62	Control		4094	3932	3940	3944	3969	3946	3937	3939	3930	3941
72	In	-	4264	4142	4167	4182	4201	4186	4175	4177	4172	4187
82	In	+	4170	3989	3992	4008	4024	4007	3997	4000	3995	3908
92	Sn	-	4252	4068	4076	3271*	3379*	3498*	3541*	3795*	3702*	3472*
102	Sn	+	4483	4270	4276	3404*	3469*	3245*	3555*	3728*	3782*	3524*
13	Zn	-	4114		3049	3024	3052	3054	3013	3014	3011	3015
23	Zn	+	4060		2941	2937	2956	2942	2920	2917	2931	2939
33	Cd	-	4009		3977	3941	3943	3923	3878	3870	3854	3858
43	Cd	+	4012		3997	3955	3947	3925	3879	3870	3858	3862
53	Control		4007		3995	3937	3937	3915	3874	3864	3851	3857
63	In	-	3903		3882	3834	3831	3809	3774	3763	3751	3756
73	In	+	4016		3997	3940	3937	3913	3884	3872	3860	3863
83	Sn	-	3995		4000*	3055*	3024	3079	2962*	3107*	3146*	2971
103	Sn	+	4139		4123*	3160*	3019	3105	3253*	3072*	2992	3137

TABLE 4 (cont.)

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	48	64	80	96	112	128	144	184	224
11	Zn	-	3290	3289	3287	3297	3291	3297	3301	3295	3298
21	Zn	+	3504	3496	3493	3519	3510	3510	3509	3508	3512
31	Cd	-	4102	4096	4076	4075	4068	4068	4069	4066	4063
41	Cd	+	4122	4117	4098	4100	4089	4089	4091	4090	4087
51	Control		4082	4077	4061	4063	4054	4056	4056	4053	4049
61	In	-	4140	4134	4123	4125	4114	4120	4119	4116	4113
81	In	+	4172	4169	4161	4158	4149	4153	4153	4155	4150
91	Sn	-	4057*	3429	4204*	3685	3698	3765*	4011*	3476	4060*
101	Sn	+	3967*	3718*	3830*	4056*	4189	3897*	4192	3621	3353
12	Zn	-	4097	4102	4096	4099	4086	4085	4087	4088	4083
22	Zn	+	3948	3945	3933	3936	3926	3924	3927	3926	3919
32	Cd	-	3972	3969	3957	3961	3944	3945	3945	3947	3941
52	Cd	+	4042	4038	4029	4033	4020	4021	4024	4025	4019
62	Control		3932	3932	3924	3927	3915	3915	3918	3918	3913
72	In	-	4181	4179	4170	4173	4161	4161	4163	4167	4158
82	In	+	4000	3993	3983	3988	3974	3974	3977	3975	3970
92	Sn	-	3723*	3427	3405	3704*	3768*	3804*	3441	3445	3516
102	Sn	+	3327	3954	3854	4055	3118	3932*	3056	3807*	3896
13	Zn	-	3145	3145	3207	3295	3404	3401	3424	3427	3441
23	Zn	+	2926	2925	2923	2955	2957	2963	2967	2959	2958
33	Cd	-	3837	3835	3831	3820	3808	3804	3808	3795	3795
43	Cd	+	3839	3836	3830	3819	3810	3805	3813	3802	3799
53	Control		3832	3830	3824	3816	3800	3803	3807	3797	3794
63	In	-	3735	3732	3726	3720	3704	3703	3705	3696	3692
73	In	+	3844	3840	3833	3827	3810	3809	3812	3803	3798
83	Sn	-	3054*	2952	3030	2931	2949	2888	2895	2856	2882
103	Sn	+	3082	3063	3390	2922	3035	2999	2951	3047	3104

TABLE 4 (cont.)

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	(d)	(g)	(e)	4	8	12	16	24	32	40
14	Zn	−	4220	4000	3025	3245	3323	3346	3323	3329	3405	3373
24	Zn	+	4146	3957	2768	2771	2780	2781	2772	2767	2781	2787
34	Cd	−	4069	3864	3863	3869	3879	3880	3870	3861	3877	3884
44	Cd	+	3985	3780	3785	3786	3797	3796	3785	3774	3794	3799
64	Control		4064	3840	3842	3846	3854	3856	3844	3839	3853	3859
74	In	−	4045	3806	3807	3811	3819	3818	3807	3799	3815	3820
84	In	+	4052	3812	3811	3817	3824	3822	3812	3805	3821	3826
94	Sn	−	4031	3785	3784	3098	3581*	3452*	3215	3411*	3196	3404*
104	Sn	+	4140	3886	3886	3045	3515*	3332*	3684*	3446*	3381*	3429*
15	Zn	−	4507		3850	4361	4383	4388	4376	4362	4355	4357
25	Zn	+	4309		3023	3016	3022	3001	2991	2982	2974	2977
35	Cd	−	4345		4341	4296	4299	4264	4249	4236	4229	4232
55	Cd	+	4294		4274	4225	4227	4193	4176	4163	4154	4154
65	Control		4312		4301	4243	4241	4208	4190	4170	4163	4161
75	In	−	4300		4290	4228	4229	4196	4175	4162	4153	4156
85	In	+	4579		4490	3684	3591	4470	4447	4432	4420	3654
95	Sn	−	4300		4283	3260*	3292*	3200	3336*	3161	3248	3128
105	Sn	+	4466		4457	3635	3583*	3235	4022	3354	3223	3590*
16	Zn	−	4786	4535	3154	3880	3944	4000	4032	4093	4167	4191
26	Zn	+	4738	4490	3259	3585	3706	3782	3809	3821	3848	3853
36	Cd	−	4789	4538	4543	4550	4561	4556	4544	4540	4555	4558
46	Cd	+	4901	4649	4640	4657	4681	4672	4667	4664	4679	4686
56	Control		5080	4905	4914	4923	4939	4934	4924	4922	4936	4941
66	In	−	5178	4923	4928	4937	4956	4949	4940	4936	4948	4952
76	In	+	5134	4946	4746*	4356*	4865*	4863	4851	4771	4863	4868
86	Sn	−	5160	4901	4902	4566*	3722	4492*	4670*	4546*	3963*	4493*
96	Sn	+	5090	4832	4835	3977*	3839	3961*	4002*	3981*	4188*	4687*

TABLE 4 (cont.)

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	48	64	80	96	112	128	144	184	224
14	Zn	-	3407	3382	3431	3435	3419	3419	3383	3425	3413
24	Zn	+	2777	2776	2771	2774	2767	2767	2772	2714	2772
34	Cd	-	3872	3870	3863	3867	3855	3854	3863	3862	3862
44	Cd	+	3787	3787	3781	3785	3773	3771	3778	3779	3778
64	Control		3847	3846	3843	3847	3835	3835	3844	3842	3843
74	In	-	3807	3807	3803	3807	3795	3792	3801	3801	3801
84	In	+	3814	3813	3809	3810	3799	3798	3809	3807	3808
94	Sn	-	3196	3441*	3381*	3377*	3113	3567*	3317*	3185*	3343
104	Sn	+	3091	3867	3410*	3870*	3574*	3873	3492*	3873	3246*
15	Zn	-	4349	4335	4320	4324	4305	4303	4303	4294	4287
25	Zn	+	2971	2963	2954	2951	2943	2943	2943	2939	2934
35	Cd	-	4222	4216	4220	4195	4177	4173	4172	4166	4158
55	Cd	+	4143	4134	4121	4121	4103	4101	4100	4086	4084
65	Control		4153	4144	4130	4131	4114	4112	4112	4103	4097
75	In	-	4143	4133	4120	4120	4104	4102	4100	4095	4087
85	In	+	4408	4395	4386	4387	4364	4367	4363	4355	4352
95	Sn	-	3136	3208	3322*	3313	3375*	3303	3227	3271*	3351*
105	Sn	+	3298*	3182	3734*	3293	3225	3231	3320	3180	3218
16	Zn	-	4194	4197	4204	4154	4208	4217	4216	4229	4228
26	Zn	+	3854	3860	3854	3857	3851	3852	3861	3862	3858
36	Cd	-	4545	4547	4541	4545	4534	4535	4541	4544	4543
46	Cd	+	4673	4679	4675	4680	4667	4667	4674	4676	4675
56	Control		4931	4933	4930	4934	4923	4923	4932	4934	4934
66	In	-	4942	4942	4938	4942	4928	4932	4938	4937	4939
76	In	+	4857	4859	4858	4862	4849	4852	4848	4849	4849
86	Sn	-	4298*	4132	4764*	4805*	4912	4639*	4427*	4307	3969*
96	Sn	+	4655*	4214*	4095	4181*	4315*	4139*	4423	4936	4267

TABLE 4 (cont.)

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	(d)	(f)	(e)	4	8	12	16	24	32	40
17	Zn	-	5120	4948	3565	3664	3701	3733	3764	3764	3778	3787
27	Zn	+	5219	5043	3945	4471	4695	4684	4466	4538	4689	4707
37	Cd	-	5189	5024	5020	5031	5045	5034	5026	5011	5019	5021
47	Cd	+	5279	5092	2620	5095	5110	5097	5089	5071	5076	5077
57	Control		5475	5293	5292	5299	5313	5303	5293	5281	5286	5291
67	In	-	5240	5039	5034	5043	5058	5050	5041	5031	5036	5040
77	In	+	5525	5341	5338	5361	5379	5370	5358	5355	5361	5371
87	Sn	-	5265	5040	5042	3823	3877	3899	3912	4296*	4163*	3970
97	Sn	+	5914	5684	5695	4330	4234	4170	4473	4833*	4766*	4584
18	Zn	-	4886		4280	4522	4555	4550	4537	4525	4516	4515
28	Zn	+	5313		Broken Lead							
38	Cd	-	4956		4942	4900	4910	4878	4861	4846	4837	4841
48	Cd	+	4954		4944	4897	4906	4875	4856	4840	4831	4835
58	Control		4911		4901	4861	4863	4838	4822	4806	4797	4801
68	In	-	5244		4153	4069	5023*	5133	4133	4142	4077	5096
78	In	+	5180		4304*	5177	5183	5153	5134	5117	5109	5115
88	Sn	-	5090		5085	3809*	3642*	3751	3679	3737*	3800	3804*
98	Sn	+	5128		5121	4285	3829*	4060*	4273*	4737	4095*	3924
				(g)								
19	Zn	-	4624	4355	3277	3514	3599	3614	3640	3662	3685	3702
39	Zn	+	4614	4351	3167	3377	3414	3428	3435	3443	3503	3536
49	Cd	-	4567	4377	4371	4400	4410	4416	4403	4401	4416	4422
59	Cd	+	4500	4242	4225	4256	4262	4265	4251	4249	4259	4264
69	Control		4519	4265	4264	4270	4274	4278	4262	4261	4271	4273
79	In	-	4489	4240	4157	4242	4250	4249	4238	4236	4247	4250
89	In	+	4538	4298	4285	4307	4313	4314	4301	4299	4312	4318
99	Sn	-	4643	4396	4396	3994*	3447	3962*	4136*	4004*	4006*	4016*
109	Sn	+	4770	4511	4514	4180*	3526	3504	4517	4510	3986	4528

TABLE 4 (cont.)

RECORDED RESISTANCE (x 10^{-3} ohms) OF SPECIMENS

(a)	(b)	(c)	48	64	80	96	112	128	144	184	224
17	Zn	−	3785	3777	3775	3865	3853	3855	3860	3861	3853
27	Zn	+	4716	4719	4740	4737	4666	4658	4723	4734	4696
37	Cd	−	5006	5001	4997	5000	4971	4974	4977	4975	4967
47	Cd	+	5067	5060	5051	5054	5029	5033	5035	5031	5024
57	Control		5278	5272	5269	5267	5240	5244	5247	5245	5238
67	In	−	5030	5025	5017	5020	4999	4999	5002	5000	4991
77	In	+	5362	5357	5352	5353	5337	5339	5342	5343	5338
87	Sn	−	4621*	4061*	3906*	3881	3910	3955	3902	3814	3934
97	Sn	+	5173	4534	4416	5185*	4541	4864	4584*	5104	5273
18	Zn	−	Broken	Lead							
28	Zn	+	"	"							
38	Cd	−	4826	4799	4791	4793	4780	4783	4785	4777	4780
48	Cd	+	4822	4796	4788	4788	4777	4778	4780	4766	4770
58	Control		4790	4765	4757	4757	4745	4744	4750	4740	4743
68	In	−	5080	5054	5048	5048	5035	5040	5042	5028	5032
78	In	+	5099	5078	5074	5074	5061	5063	5065	5044	5049
88	Sn	−	3941	3737*	3651	3651	3798	4113*	3715	3654	3813
98	Sn	+	4066	4664*	4493	4493*	4854	4344*	4415	4065*	4463
19	Zn	−	3723	3727	3729	3738	3727	3739	3741	3745	3748
39	Zn	+	3572	3571	3573	3587	3566	3573	3579	3583	3586
49	Cd	−	4413	4411	4410	4413	4402	4404	4409	4413	3586
59	Cd	+	4261	4261	4257	4260	4247	4252	4257	4258	4260
69	Control		4268	4265	4263	4264	4251	4254	4262	4264	4265
79	In	−	4244	4241	4241	4244	4231	4233	4238	4241	4239
89	In	+	4310	4307	4307	4308	4295	4298	4303	4305	4305
99	Sn	−	3967*	4172	3643	4000*	3868*	3959*	3854*	3768	4234*
109	Sn	+	4522	4521	4245*	4524	3765*	4374	3850*	3829	3820*

TABLE 4 (cont.)

HEADINGS:

(a) Specimen Number
(b) Alloying Element Deposited on Film
(c) Terminal of Power Supply to which coated end of specimen was connected during annealing
(d) Resistance of As Deposited Aluminum Film
(e) Resistance of Specimen After Deposition of element in column (b)
(f) Resistance of Aluminum Film After 10 Hr. Anneal
(g) Resistance of Aluminum Film After 35 Hr. Anneal;
4, 8, etc. - Elapsed Annealing Time in Hours when respective resistance measurements were made.

NOTES:

* Specimen experienced drift during resistance measurement

1 Galvanometer behaved erratically during measurement

TABLE 5

RATIOS OF SPECIMEN TO CONTROL RESISTANCE (R/R_C) ($x\ 10^{-3}$)

			Elapsed Annealing Time (Hours)						
(a)	(d)	(c)	4	8	12	16	24	32	40
11	1042	759	773	792	842	803	805	804	806
21	1023	797	821	831	838	845	851	857	857
31	1006	1006	1005	1005	1005	1005	1006	1006	1005
41	1010	1010	1010	1009	1010	1010	1010	1011	1009
61	1015	1013	1014	1014	1014	1015	1014	1015	1013
81	1021	1022	1023	1022	1023	1022	1022	1023	1022
91	1046	1044	881	963	855	895	963	947	885
101	1039	1037	905	915	987	907	1027	1035	1033
12	1046	882	1022	1035	1035	1038	1039	1041	1042
22	1008	735	1007	1006	1004	1004	1004	1005	1004
32	1013	1012	1012	1011	1009	1009	1010	1010	1010
52	1027	1025	1026	1026	1026	1025	1027	1027	1026
72	1014	1013	1016	1014	1015	1015	1016	1017	991
82	1053	1058	1060	1059	1061	1060	1061	1062	1062
92	1035	1034	829	852	886	899	964	942	881
102	1086	1085	872	874	822	903	947	963	894
	(b)								
13	1027	763	768	775	780	778	780	782	782
23	1013	736	746	751	751	754	755	761	762
33	1001	995	1001	1002	1002	1001	1002	1001	1000
43	1001	1000	1005	1003	1002	1001	1002	1002	1001
63	974	972	974	973	973	974	974	974	974
73	1002	1000	1001	1000	999	1002	1002	1002	1002
83	997	1012	776	768	786	765	804	817	770
103	1033	1032	803	767	793	840	795	777	813
	(e)								
14	1042	787	844	862	868	864	867	884	874
24	1030	721	720	721	721	721	721	722	722
34	1006	1006	1006	1007	1006	1007	1006	1006	1006
44	984	985	984	985	984	984	983	985	984
74	991	991	991	991	990	990	990	990	990
84	993	992	992	992	991	992	991	992	991
94	986	985	805	929	895	836	889	829	882
104	1012	1012	792	912	864	958	899	877	888

TABLE 5 (cont.)

RATIOS OF SPECIMEN TO CONTROL RESISTANCE (R/R_c) (x 10^{-3})

Elapsed Annealing Time (Hours)

(a)	48	64	80	96	112	128	144	184	224
11	806	806	809	811	812	813	814	813	815
21	858	857	860	866	864	865	865	865	867
31	1005	1004	1004	1003	1004	1003	1003	1003	1004
41	1009	1009	1009	1009	1009	1008	1008	1009	1009
61	1014	1014	1015	1015	1015	1016	1015	1015	1016
81	1022	1022	1024	1023	1024	1024	1024	1025	1025
91	994	841	1035	907	912	928	988	858	1003
101	972	912	943	998	1033	960	1033	893	828
12	1042	1043	1044	1044	1044	1043	1043	1043	1044
22	1004	1003	1002	1002	1003	1002	1002	1002	1002
32	1010	1009	1008	1008	1007	1008	1007	1007	1007
52	1028	1027	1027	1027	1027	1027	1027	1027	1027
72	1063	1063	1063	1062	1063	1063	1062	1063	1063
82	1017	1015	1015	1015	1015	1015	1015	1014	1015
92	947	871	868	943	962	972	878	879	898
102	846	1006	982	1020	796	1004	780	972	996
13	821	821	839	863	896	894	899	903	907
23	763	763	764	774	778	779	779	779	780
33	1001	1001	1002	1001	1002	1000	1000	1000	1000
43	1002	1001	1002	1001	1002	1000	1001	1001	1001
63	975	974	974	975	975	974	973	974	973
73	1003	1002	1002	1003	1002	1001	1001	1002	1001
83	797	770	793	768	776	759	760	752	760
103	804	799	886	766	799	788	775	803	818
14	885	879	893	893	891	891	888	892	888
24	722	722	721	721	721	721	721	722	721
34	1006	1006	1005	1005	1005	1005	1005	1005	1005
44	984	985	984	984	984	983	983	984	983
74	989	990	990	989	989	989	989	989	989
84	991	991	991	990	990	990	991	991	991
94	831	895	880	878	812	930	863	929	870
104	803	1006	887	1006	931	1010	908	1008	845

TABLE 5 (cont.)

RATIOS OF SPECIMEN TO CONTROL RESISTANCE (R/R_c) ($\times 10^{-3}$)

			Elapsed Annealing Time (Hours)						
(a)	(b)	(c)	4	8	12	16	24	32	40
15	1045	895	1028	1034	1043	1045	1046	1046	1047
25	999	703	711	713	713	714	715	714	715
35	1008	1009	1013	1014	1013	1014	1016	1016	1017
55	996	994	996	997	996	997	998	998	998
75	997	997	997	997	997	997	998	998	999
85	1062	1044	868	847	1062	1062	1063	1062	878
95	997	996	768	776	760	796	758	780	752
105	1035	1036	857	845	769	960	804	774	863
	(e)								
16	925	642	788	799	811	819	832	844	848
26	916	663	728	750	767	774	776	780	781
36	925	925	924	924	924	923	923	923	922
46	948	944	946	948	947	948	948	948	948
66	1004	1003	1003	1004	1003	1003	1003	1003	1002
76	1008	966	885	985	986	985	969	985	985
86	999	998	927	754	911	948	924	803	909
96	985	984	808	777	803	813	809	848	948
	(d)								
17	935	674	691	695	704	711	713	715	716
27	953	746	844	884	883	844	860	887	890
37	949	949	949	949	949	949	949	950	949
47	962		961	962	961	961	960	960	960
67	952	951	952	952	952	952	953	953	953
77	1009	1009	1012	1012	1013	1012	1014	1014	1015
87	952	953	721	730	735	739	814	788	750
97	1074	1076	817	797	786	845	915	902	866
	(b)								
18	995	873	930	937	940	941	942	942	940
38	1009	1008	1008	1010	1008	1008	1008	1009	1008
48	1009	1009	1007	1009	1008	1007	1007	1007	1007
68	1068	847	837	1033	1060	857	862	850	1061
78	1055	878	1065	1066	1065	1065	1065	1065	1065
88	1036	1037	784	749	775	763	778	792	792
98	1044	1045	881	788	839	886	986	854	8[illegible]7

TABLE 5 (cont.)

RATIOS OF SPECIMEN TO CONTROL RESISTANCE (R/R_c) $(x\ 10^{-3})$

	Elapsed Annealing Time (Hours)								
(a)	48	64	80	96	112	128	144	184	224
15	1047	1046	1046	1046	1046	1046	1046	1046	1046
25	715	715	715	714	715	715	715	716	716
35	1016	1017	1022	1015	1015	1014	1014	1015	1015
55	997	998	998	997	997	997	997	996	997
75	997	997	997	997	997	997	997	998	998
85	1061	1061	1062	1062	1060	1062	1061	1061	1062
95	754	774	804	802	820	803	785	797	818
105	794	768	904	797	784	786	807	775	786
16	850	851	852	842	855	856	855	857	857
26	781	782	782	781	782	782	783	783	782
36	921	922	921	921	921	921	921	921	921
46	947	948	948	948	948	948	948	948	948
66	1002	1002	1001	1001	1001	1002	1002	1001	1001
76	985	985	985	985	985	985	983	983	983
86	871	838	966	974	998	941	898	873	805
96	944	854	830	847	876	840	897	798	865
17	717	716	716	734	735	735	736	736	736
27	893	895	899	899	890	888	900	903	896
37	948	948	948	949	948	948	949	949	948
47	960	959	958	959	960	959	960	959	959
67	953	953	952	953	954	953	954	954	953
77	1016	1016	1015	1016	1018	1018	1018	1019	1019
87	875	770	741	737	746	754	744	727	751
97	980	860	838	984	866	927	874	973	1007
18	Broken	Lead							
38	1007	1007	1007	1007	1007	1008	1007	1008	1008
48	1006	1006	1006	1006	1007	1007	1006	1006	1006
68	1060	1060	1061	1061	1061	1062	1061	1061	1061
78	1064	1065	1066	1067	1066	1067	1066	1064	1064
88	822	784	819	767	800	867	782	771	804
98	849	979	996	944	1023	915	929	858	941

TABLE 5 (cont.)

RATIOS OF SPECIMEN TO CONTROL RESISTANCE (R/R_C) ($\times 10^{-3}$)

			Elapsed Annealing Time (Hours)						
(a)	(e)	(c)	4	8	12	16	24	32	40
19	1021	768	823	842	845	854	859	863	866
39	1020	743	791	799	801	806	808	820	827
49	1026	1025	1030	1032	1032	1033	1033	1034	1035
59	995	991	997	997	997	997	997	997	998
79	994	975	993	995	993	994	994	994	995
89	1008	1005	1009	1009	1009	1009	1009	1009	1010
99	1031	1031	935	807	926	970	940	938	940
109	1058	1059	979	825	819	1060	1058	933	1059

(a)	48	64	80	96	112	128	144	184	224
19	872	874	874	877	877	879	878	879	879
39	837	837	838	841	839	840	839	840	841
49	1034	1034	1034	1035	1035	1035	1034	1035	1035
59	998	999	998	999	999	999	999	999	999
79	994	994	995	995	995	995	994	995	994
89	1010	1010	1010	1010	1010	1010	1010	1010	1010
99	929	978	852	938	910	930	904	884	993
109	1060	1060	995	1061	886	1028	903	898	896

HEADINGS:
(a) Specimen Number
(b) As Deposited Aluminum Film
(c) After Deposit of Alloying Element
(d) Aluminum Film with 10 Hr. Anneal
(e) Aluminum Film with 35 Hr. Anneal.

TABLE 6

AVERAGE R/R_c Values (x 10^{-3})

	Pre-Anneal or Terminal Polarity		0	4	8	12	16	24	32	40	48	64	80	96	112	128	144	184	224
Zn	As Deposited	(1)	774	813	818	822	823	824	826	827	837	836	841	849	859	859	860	861	862
	10 Hour	(2)	772	830	844	860	848	853	860	862	863	863	866	871	869	869	872	872	872
	(Corrected)		774	832	846	862	850	855	862	864	865	865	868	873	871	871	874	874	874
	35 Hour	(3)	721	795	810	818	823	828	838	839	845	845	848	847	849	850	849	850	849
	(Corrected)		774	848	863	871	876	881	891	892	898	898	901	900	902	903	902	903	902
	Positive	(4)	728	759	769	774	779	781	786	788	791								
	(Corrected)		765	796	806	811	816	818	823	825	828								
	Negative	(5)	765	836	849	864	860	864	868	870	879								
Sn	As Deposited	(6)	1027	812	782	787	835	821	799	801	803	812	867	807	834	820	806	793	821
	(Corrected)		1012	797	767	772	820	806	784	786	788	797	852	792	819	805	791	778	806
	10 Hour	(7)	1038	838	855	845	865	938	930	884	935	877	901	932	886	924	883	883	914
	(Corrected)		1012	821	829	819	839	912	904	827	908	851	875	906	860	898	857	857	888
	35 Hour	(8)	1012	874	834	870	931	920	871	937	906	939	902	951	902	947	896	898	879
	Positive	(9)	1040	857	835	838	894	915	885	921	895	916	918	936	888	918	879	885	887
	Negative	(10)	1008	824	817	833	859	870	848	840	869	836	862	857	860	876	845	830	856
	(Corrected)		1040	856	849	865	891	902	880	872	901	868	894	889	892	908	877	862	888
Cd	As Deposited	(11)	1002	1005	1006	1005	1005	1005	1005	1005	1005	1005	1006	1005	1005	1004	1004	1004	1005
	10 Hour		994	994	994	993	993	994	994	993	993	993	992	993	993	992	992	992	992
	35 Hour		979	981	982	982	982	982	983	983	982	983	982	982	982	982	982	982	982
In	As Deposited	(11)	956	957	986	1026	993	994	992	997	1027	1027	1027	1028	1027	1027	1027	1027	1027
	10 Hour		1011	1013	1012	1013	1013	1013	1014	1008	1014	1014	1014	1014	1015	1015	1015	1015	1015
	35 Hour		972	978	996	995	995	996	995	995	995	995	995	995	995	995	995	995	995

SPECIMENS FOR WHICH AVERAGES WERE MADE: (1) 13, 23, 15, 25; (2) 11, 21, 12, 17, 27; (3) 14, 16, 26, 19, 39; (4) 21, 23, 25, 26, 39; (5) 11, 13, 15, 16, 19; (6) all; (7) all; (8) all; (9) 96, 97, 98, 101, 102, 103, 104, 105, 109; (10) 86, 87, 88, 91, 92, 93, 94, 95, 99; (11) all,

BIBLIOGRAPHY

1. A.S.M. Handbook, Properties and Selection, Vol. 1, p. 1169, American Society of Metals, New York, 1961.

2. Rhoderick, E. H., "Electrical Properties of Thin Films," Brit. J. Appl. Phys., Vol. 15 (1964), p. 613.

3. Levinstein, H., "The Growth and Structure of Thin Metallic Films," J. Appl. Phys., Vol. 20 (1949), p. 306.

4. Basset, G. A. and Pashley, D. W., "The Growth, Structure and Mechanical Properties of Evaporated Metal Films," J. Inst. Metals, Vol. 87 (1959), p. 449.

5. Shewmon, P. G., "High Diffusivity Paths," Diffusion in Solids, p. 164, McGraw-Hill Book Co., New York, 1963.

6. Kubaschewski, O. and Hopkins, B. E., "Basic Information," Oxidation of Metals and Alloys, p. 1, Butterworth and Co., Ltd., London, 1962.

7. Hauffe, Karl, "Defect Phenomena in Nonstoichiometric Ionic Crystals," Oxidation of Metals, based on the German edition of Oxidation von Metallen and Metallegierungen, p. 14, Plenum Press, New York, 1965.

8. Smallman, R. E., "Creep," Modern Physical Metallurgy, p. 343, Butterworth and Co., Ltd., London, 1963.

9. Azaroff, L. V., "Atomic Radii," Introduction to Solids, p. 437, McGraw-Hill Book Co., New York, 1960.

10. Holland, L., Vacuum Deposition of Thin Films, John Wiley, New York, 1961.

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