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UNIVERSITY OF VIRGINIA
DEPARTMENT OF PHYSICS
ORDNANCE RESEARCH LABORATORY

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DIRECT DETERMINATION OF THE ADHESIVE BOND STRENGTH OF CHROMIUM ELECTRODEPOSITED ON STEEL

by

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I. SCOPE OF PROJECT

The Scope of Work: To determine (a) if a technique of electrodepositing chromium plate on steel can be established for the purpose of controlling the degree of adhesion of the plate and holding the adhesion of each of several degrees within fairly narrow limits, (b) the influence of a wide range of temperature on the yield and fracture strengths of plated steels having various microstructures.

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III. SUMMARY

Small chromium plated rotors have been freely suspended in a magnetic field and accelerated until the centrifugal forces acting on the plate cause it to be torn from the hardened steel base metal. A knowledge of the size of the rotor, the thickness and density of the coating and the speed of the rotor at failure allows one to compute directly the maximum stress imposed on the adhesive bond or other points of failure in the system as the case may be.

A complete description of the apparatus is presented, and a test program involving a study of the effect of various surface treatments, both of cleaning and contaminating types, on the adhesion value is described. It is shown that the adhesion can be controlled by the use of prescribed contaminants. Further tests involving the effect of plate thickness and the length of use of plating bath on adhesion are described.

The problem of the effect of the thickness of plated films on the tensile strength of steels having microstructures representative of the Martensitic, Bainitic, and Pearlitic systems was studied experimentally. Due to the lack of time and shortage of funds the work on the effect of temperature on the tensile properties of various steels was postponed under agreement with the technical supervisors.

IV. INTRODUCTION

For many years Professor J. W. Beams and his colleagues at the University of Virginia have been involved in the application of high rotational speed techniques to the study of various fundamental problems in physics. Several years ago it was demonstrated that a silver film electroplated on a steel rotor could be "thrown off" provided that it was rotated at a speed great enough so that the centrifugal field produced by the silver film acting on the interface between the silver and the steel was sufficient to overcome the adhesion force between the silver and the steel, or, as occurred in some instances, the centrifugal force exceeded the cohesive forces in a layer of silver very close to the interface and the rupture occurred at this point.

At that time extensive work on the study of adhesion was by-passed for the then more exciting problem of the study of hoop stresses in thin silver films, during which measurements adhesion was purposely reduced to a very low value. For silver this was readily accomplished by dipping the steel rotors in human serum albumin before plating.

During the summer of 1954, the Ordnance Research Laboratory of the university of Virginia at the request of the Watertown Arsenal Laboratory of the Army Ordnance Corps initiated a program to explore the possibilities of applying such high rotational speed techniques to the direct measurement of the "adhesion forces" of chromium electrodeposited directly on steel.

Essentially the technique is based upon the following considerations. The test sample is a small solid cylinder of steel varying from 1/8 inch diameter to 3/8 inch diameter depending upon the plate thickness under investigation, and of a length about 2/3 of the diameter. A layer of chromium is electrodeposited on the periphery of the cylinder and the sample is then freely supported in a controlled magnetic field. The sample is then caused to rotate at the extremely high speeds possible in such a friction free bearing. According to calculations based on an elastic theory of matter, such as found in any standard text on the strength of materials, the stress on the chromium-steel interface is given by

$$\sigma = \rho_b \frac{\pi^2 N^2 (b-a)(b+a)^2}{4a}$$

where ρ_b = density of plate

a = diameter of steel

b = diameter of steel plus chromium

N = angular speed of rotor in RPS

When σ exceeds the tensile strength of either the chromium or the steel, or when it exceeds the adhesive force at the interface, a failure will occur. Hence, knowing all quantities on the right side of this equation, the stress relating to the particular failure involved can be directly computed.

The unique features of this method of adhesion testing are that the stresses on the interface are applied uniformly in tension through the plate with the maximum stress (as far as the plate is concerned) occurring at the interface, and that no physical connections need be made to either the base metal or the electrodeposited coating. It is obvious that this method could be equally well applied to an evaporated film, or to other than metallic films, as indeed has been done. The only limitations on the method are that the rotor must contain at least a core of magnetic material, and the size of the sample must be small enough to permit centrifugal fields to be attained which are sufficiently large to overcome the forces to be measured. Needless to say, this is a destructive test and cannot be applied "in the field".

V. OBJECTIVES

The main objectives of the present program are two-fold:

1. To establish the feasibility of utilizing the method in making direct quantitative measurements of adhesive forces for metallic coatings.

2. To undertake a detailed study of the effects of the many variables inherent in the electrodeposition process upon the adhesion of the resulting deposit.

It is felt that the first objective has been successfully demonstrated, and while the second objective is by no means completed, several important effects have been noted and should be of sufficient interest to report at this time.

VI. APPARATUS

The basic electromagnetic support apparatus is shown in Figure 1. The rotor, R, is suspended on the axial magnetic field of the solenoid, S, and is spun in the two phase rotating magnetic field in the two pairs of coils, D. The horizontal position of the steel rotor is maintained by the symmetrically diverging field of the solenoid, while its vertical position is maintained by an automatic regulation of the current through the solenoid, S. The small coil, L_1, is part of the grid circuit of a tuned-plate, tuned grid radio

frequency oscillator which regulates the current through S. This circuit is shown in detail in Figure 2 and briefly its operation is as follows.

Consider a downward displacement of the rotor. This will change the properties of the coil, L_1, in such a way as to reduce the amplitude of the oscillation in the grid circuit of the oscillator and, since the oscillator is partially neutralized, the amplitude of oscillation in the plate circuit will also decrease. As a result the potential across the cathode follower stage will also drop and cause the potential on the control grid of the 6L6 power tube to increase. This causes an increase in the current through the supporting solenoid which increases the lifting force on the rotor and restores it to its original position. Vertical damping is accomplished by mixing the derivative of the error signal with the error signal itself. Returning to Figure 1, a small iron wire, H, mounted in a glass tube, G, filled with a liquid assists in damping any horizontal motion of the rotor. An iron tube, I, is placed inside the solenoid to increase the field per unit of current. The reader is referred to the literature for a more detailed discussion of this circuit.*

The rotor is operated in a vacuum, and the rotor speed is measured by a phototube arrangement as follows. A dark spot is provided on some portion of the rotor and light is focused on the rotor in the region of that spot. The resulting scattered light is in turn focused on an electron multiplier phototube. The phototube produces a signal due to the difference in the scattered light intensity from the dark and bright portions of the rotor. This signal is amplified and fed to one pair of plates of a cathod ray oscillograph. This frequency is then compared with the output of a standard variable frequency oscillator.

The drive oscillator is shown in Figure 3. This is a conventional design employing a Hartly Oscillator driving a pair of 6L6's in AB operation. The circuit operates at a frequency of 125 KC and is capable of a power output of approximately 100 watts.

The apparatus is pictured in Figures 4, 5, and 6. Figure 4 presents a view of the entire assembly. The actual test apparatus is inside the protective wooden barricade at the right of the picture and the operator is viewing the test specimen through a periscopic arrangement. The associated electronic equipment is seen in the racks behind the operator. Vacuum system controls are mounted on the front of the supporting stand for the test apparatus.

Figure 5 is an interior view of the barricade showing the test apparatus completely assembled and ready for operation. The support coil can be seen at the center of the picture. The actual test specimen is not visible, being obscured by the wooden frame which supports the four driving coils. It is located within a glass chamber attached to the vacuum pumps through the tubulation seen at

* see eg. F. T. Holmes, R.S.I., 8, 444, (1937).
Beams, Young & Moore, J. Appl. Phys. 17, 886 (1946).

the left of the picture. The detector coil is mounted on the pedestal, the lower part of which is visible in the picture. The rotor is illuminated through the axis of one of the drive coils by the lamp at the right, and the speed control circuit, the base of which can be seen in the background, receives reflected light through the core of another of the drive coils.

Figure 6 is a close-up view of the test specimen seen with the driving coils and vacuum chamber removed (the rotor is actually cylindrical in shape - the apparent bump being caused by halation of the film due to the large amount of reflection from the chrome plate). The specimen is freely supported and is rotating at a moderate speed. The detector coil is at the bottom of the picture and the plastic case housing the damping core can be seen protruding from the support coil at top.

VII. PREPARATION OF SAMPLES

The samples are prepared in the following manner. A steel rod of the proper diameter for the test under consideration, and usually about 6 inches long, is ground to be of uniform dimensions. A wet centerless grinding process is preferred. All samples are of heat treated 4140 steel stock and are given identical cleaning treatments prior to whatever special preparation is in order. This consists of each sample being washed in carbon tetrachloride, given an alkali cleaning according to ASTM specification B-177-49, and rinsed in running water. The surface of the rod is then prepared according to the procedure under study, after which the rod is immersed in the plating bath on the axis of a cylindrical anode and the metal is deposited under the desired conditions. Upon removal from the bath, the rod is cut into short pieces suitable for rotor size.

In order to test the influence of hoop stress in the chromium on the measured "adhesion", two types of rotors are used in the tests. In the first type, the rotors are used with a solid chrome surface just as they come from the plating bath. In the second type, axial slots are ground in the rotor about the periphery in such a way that the chrome deposit is clearly broken into eight axial bands and there is no hoop stress whatsoever on the chrome. As will be evident in considering the data, on the average there seems to be little or no increase in the adhesion values due to the presence of the solid band of chrome.

VIII. EXPERIMENTAL RESULTS

A. Effect of Length of Use of Bath on Adhesion of Deposit

This effect was discovered quite unexpectedly about midway in the present testing program, and unfortunately was so pronounced that it automatically invalidated several weeks of previously obtained data. According to usual plating custom, the plating bath, which was in almost daily use, was kept freshened by the addition of CrO_3 to maintain a constant chromate ion concentration. However, periods would occur during which the bath was not used for a week or two. After such periods it was customary to prepare a fresh bath before further plating was done. It became apparent that the adhesion values would jump considerably every time a new bath was used. A further reorganization and appraisal of previous data indeed indicated that the adhesion values dropped off steadily with repeated bath usuage even though the chromate ion concentration was maintained constant. In fact, the adhesion values obtained for even the second plating from a given bath was much less than those obtained the first time the bath was used. When a new bath was used the adhesion values would revert to their "normal" levels. This effect is presented in Table 1 and illustrated in Figure 7. In this chart the various groups shown were plated under the following conditions. The particular history of the different test groups are given below. (O's represent rotors with solid chrome bands, X's represent rotors with slotted chrome bands).

Group A - Dip 2 minutes in # 30 motor oil; wipe off excess oil with paper towel; plate 16 hours, 10 minutes at $2A/in^2$ in a fresh plating bath; chrome thickness .006".

Group B - Dip 2 minutes in # 30 motor oil; wipe off excess oil with paper towel. Plate 15 hours, 30 minutes at $2A/in^2$ in a fresh plating bath. Chrome thickness .007".

Group C - Same preparation as Group A and plated in same bath as Group A except at a later date after CrO_3 had been added to maintain bath concentration. Chrome thickness .0065".

Group D - Same motor oil dip as above. Use fresh plating bath. Reverse etch in plating bath for 15 seconds, then plate for 16 hours at $2A/in^2$, chrome thickness .0085".

Group E - Dip 30 seconds in Ortholeum-162, 10 cc/liter, rinse in running water 2 minutes, reverse etch in fresh plating bath 15 seconds; plate 15 hours, 56 minutes at $2A/in^2$. Chrome thickness .009".

Group F - Same treatment as Group E except that etching and

plating was done in a bath which had been used and to which CrO_3 had been added to maintain concentration. Chrome thickness .0105".

Note: The column heights indicate the average adhesion values for a given group.

It is obvious that repeated use of the bath reduced the adhesion values by a factor of 2 or greater and so all data which are presented from here on represent experiments performed on rotors which in every case had been plated in a fresh bath.

B. Effect of Reverse Etch on Adhesion

One of the principal programs undertaken was to study the effect of various controlled pre-bath contaminating procedures on the adhesion of the chrome. It soon became obvious from the test results that no matter what pre-bath contaminant dip was used, a 15 second reverse etch in the bath just prior to plating almost completely eliminated all effects of the contamination procedure and restored the adhesion of the deposit almost to a maximum value. In fact, as will be demonstrated throughout the report, it appears as though of all things tried thus far, a 15 second reverse etch is as equally good as, or better than any other treatment for assuring maximum adhesion.

The past history of the specimen apparently made little difference. No matter what the treatment, the reverse etch seemed equally effective. Thus far, only one length of reverse etch; namely, 15 seconds, has been used but it is obvious that tests should be run with other times.

Results illustrating this effect are presented in Table 2 and illustrated in Figure 8. The fact that reverse etch improves the adhesion of the deposit to the base metal can be further demonstrated by considering the photomicrographs shown in Figure 9 and 10. Figure 9 shows a section of the cylindrical side of a piece of rotor which had been dipped in Ortholeum-162 and then plated with no reverse etch. Upon rotation to failure a chunk of chromium was thrown off of the plate, and the rather prominent line through the center of the figure is the edge of this crater. The grinding marks on the steel can be clearly seen on the left hand side of the picture illustrating that the failure occurred at the interface. Figure 10 shows a similar picture taken with a sample in which a reverse etch was used. In this case no evidence of the steel can be seen and the break clearly occurred in the chrome.

C. Effect of Pre-Bath Cleaning Treatments on Adhesion

At the request of the Watertown Arsenal Laboratories a group of samples especially prepared by Watertown were tested to study the relative merits of electropolishing vs liquid honing as a pre-bath

treatment. The results of these tests are given in Table 3, and shown graphically in Figure 11.

The treatment as described by Watertown for the three groups shown on the figure are as follows:

Group A

1. dipped in 50% HCL for 1 minute.

2. electropolished 2 minutes in 80% H_3PO_4, 20% H_2SO_4.

3. cold water rinse.

4. insert in plating bath and raise plating current slowly to 2A/in

Group B

1. vapor hone with # 100 grit at 90 PSI.

2. dip in 50% HCL solution for 1 minute.

3. cold water rinse.

4. insert in plating bath and raise current slowly to $2A/in^2$.

Group C

1. dip in 50% HCL solution for 1 minute.

2. vapor hone with # 100 grit at 90 PSI

3. cold water rinse.

4. reverse etch at $1A/in^2$ for 20 seconds in bath before plating.

Examination of Group A indicates that electropolishing in combination with the HCL dip is of some value in increasing adhesion. A comparison of Group B and Group C; however, shows that the vapor honing as done in this instance, was quite detrimental to adhesion and definitely contaminated the surface. In Group B the HCL dip apparently removed the effect of the vapor honing, but in Group C in which the honing followed the HCL dip, an added reverse etch was not even sufficient to counteract the honing treatment. This was somewhat surprising in view of the excellent results obtained with reverse etching in this laboratory. Also the plate film was thinner in this case, which as will be shown later usually tends to an increase in adhesion.

D. Control of Adhesion by the Use of a Contaminant

A detailed investigation was made of the possibility of controlling the adhesion of chrome plate by a controlled process of surface contamination prior to immersion in the plating bath. Various types of contaminants were studied with varied and interesting results.

Human serum albumin was the first contaminant tried. This was selected because of previous success with this protein type contaminant reported by Jaquet in the case of copper films on various base metals, and the success of this laboratory in reducing the adhesion of thin silver films deposited on steel. In this latter case it was found that with a sufficiently concentrated solution the adhesion could be reduced to almost zero by a simple immersion of the sample in the albumin solution immediately prior to plating.

A series of experiments were performed in which samples were dipped in various solutions of albumin, plated according to normal procedure, and then tested. The results are not included herein in detail as they are somewhat complicated by the fact that all samples used in the series were not plated in fresh baths. However, the general conclusion drawn from a careful study of the data was that the albumin had little or no effect upon the adhesion value of chromium to steel. This was indeed surprising in view of the excellent results obtained for copper and silver.

Consequently, a series of tests were run using another protein, fibrin peptone, as a contaminant. The results are presented in Table 4. Here it is again apparent that the fibrin peptone contributes no reduction of the adhesion value. In fact, taking Group A, in which the plating was done without the use of any contaminant, as a control, it appears as though the fibrin peptone actually increased the adhesion of the chromium on the steel. This effect was also noted, although not to as large an extent with the human serum albumin.

A few measurements were next made using an organic phosphate, Ortholeum-162 manufactured by the DuPont Company as a contaminant. The results are presented in Table 5 and Figure 12. Several other exploratory runs were made, but the results are not included in this table since the test procedure did not conform rigidly with the general pattern consistent throughout all the results presented herein. However, as evidenced in Figure 12, the contaminant concentration definitely has an effect on the adhesion, and in considering all the tests it was apparent that the adhesion could be controlled to a certain extent. That is to say that the adhesion values obtained subsequent to the use of a particular contaminant concentration could be reasonably well predicted.

SAE # 30 motor oil was also tested for contaminating action. It proved to be an extremely good contaminant as can be seen by considering Groups A, B, and D of Table 1; however, it was impossible to control the concentration of the contaminant and hence, the adhesion.

E. Effect of Plate Thickness on Adhesion

One important problem concerning which it was desired to obtain information by the use of this test procedure was that of the effect of plate thickness on adhesion. However, the attack on the problem was somewhat limited by the fact that if the standard size test sample was used, the adhesion was so great that the rotor would explode before the film was torn off unless the film was at least .008" thick. Also difficulties were encountered in plating films thicker than about .012" and it was not considered desirable to devote too much time at this stage in an attempt to plate thicker films. Hence, the basic apparatus has recently been modified slightly so as to reduce the test piece to 1/8" diameter allowing much thinner films to be studied. Data with such films are just now being obtained and it is impossible to draw any conclusions at this time.

In lieu of a direct attack on the problem an attempt was made to study the effect of film thickness by using standard size test samples upon which the adhesion had been reduced by contamination in Ortholeum-162. The results are presented in Table 5 and illustrated in Figure 13. While no quantitative conclusions can be made due to the complication of the uncertainty regarding the reproducibility of the contamination effect, it appears certain that a general conclusion of increasing adhesive force with decreasing film thickness is valid. This was also substantiated by other scattered results obtained during the performance of other tests in which slight variations in film thickness were present.

F. Comparative Strengths of Steels of Different Microstructures

A series of tests was conducted to determine the effect of various thicknesses of chromium plate on the tensile strength of steels of three representative microstructures; namely, Pearlite, Martensite, and Bainite. The steel samples were provided and ground to size by the Watertown Arsenal Laboratories, but were plated at the Ordnance Research Laboratory. The tests were made by rotating the specimens until the centrifugal force was sufficient to overcome the tensile strength of the material, causing the rotor to disentigrate. Under such conditions the maximum stress on the rotor is at the center and is given by

$$S = \left[\rho_a a^2 (3+\mu) + \frac{\rho_b}{a}(b-a)(b+a) \right]$$

where N = rotor speed in rps

ρ_a : density of rotor

a : diameter of base rotor plus coating

b : diameter of base rotor only

$(b-a)$: thickness of coating

ρ_b = density of coating material

μ : Poisson's Ratio

This equation assumes that there is no plastic flow in the steel until the sample ruptures. This assumption is deemed valid by observations on a test in progress. Normally, the rate of acceleration of the sample is constant over long periods of time, but just before the sample explodes, there is an abrupt change in the rate of angular acceleration even though the driving force is held constant. This change in rate of acceleration is the result of the increased moment of inertia of the sample due to plastic flow, and as indicated is not in evidence until just prior to the instant of failure.

The data from these tests are presented in Table 7 and the maximum stress is plotted as a function of plate thickness in Figure 14. It can be seen generally the strength of the Martensite and Bainite samples is decreased slightly by increasing the thickness of the chrome plate, but for the thickness plates used the values are only about 10% below the measured strength of the bare material without any plate. In the case of Pearlite, however, the samples show a slight increase in strength with increasing thickness of chromium plate up to a plate thickness of about 0.0035 inches At this point it is about 15% higher than the base metal value. For a plate thickness greater than this value, the maximum stress sustained by the rotor decreases.

All test samples were prepared for maximum adhesion of the plate using the following steps in accordance with the results of previous experiments.

1. degrease in CCL_4.

2. alkali clean 2 minutes cathodic, 30 seconds anodic at 0.35 A/in^2.
   Na_2CO_3 45 gm/l -Boiling temperature.
   Na_3PO_4 30 gm/l
   NaOh 15 gm/l

3. rinse 2 minutes in running water.

4. reverse etch 15 seconds in plating bath at $2A/in^2$.

5. plate $2A/in^2$ in fresh plating bath.
   bath composition
   CrO_3 250 gm/l
   H_2SO_4 2.5 gm/l
   Temperature - 55 degress Centigrade.

IX. DISCUSSION OF RESULTS

It is quite apparent that the program reported above serves merely as an introduction to the study of adhesive forces, and hardly begins to utilize possibilities presented by the method. Perhaps the greatest value, at least in a broad sense, obtained from the present program of measurements has been that one is now able to ascertain with some degree of assurance what the experimental variables should be in a more detailed investigative program. General conditions of cause and effect have been established. One now knows that if one performs a plating operation under a given set of conditions, the resulting adhesive force of the plate will be a certain value. It remains to determine exactly why these conditions affect the adhesion in this manner.

It is felt that sufficient evidence has been presented to establish the experimental method as a powerful tool for the direct quantitative measurement of adhesion forces. As indicated earlier in this report this method has several advantages over previously used testing methods. Foremost among these is the matter of the direct application of the forces involved. In all other methods brought to the attention of this laboratory, the exact nature of the applied force has been somewhat in doubt, but it almost certainly contained a finite amount of shear. The magnetic support system also requires no physical contacts to be made to the test piece, which is a distinct advantage and as demonstrated from the results obtained herein presents great versatility.

Perhaps it is in order to make a few comments and observations regarding the nature of the adhesion force involved. Obviously this term has been rather loosely used throughout the report. Actually what the present measurement gives is the strength of the weakest link in the plated system. In most cases this occurs in the chromium very near to the interface. The force involved, then might be termed the "effective adhesion" of the particular deposit involved, and is of course the force of most practical importance to the plater or the user of the chromium plated sample. One might thus consider such a force to be the adhesion force from an engineering and applications point of view as contrasted to the fundamental adhesion between the layers of plate and substrate nearest the interface. It was definitely observed that the break customarily occurred in the chromium. However, the location of the break was almost entirely random. It was impossible to find any

preferred amount of chromium left on the surface as had been the case for example in the recently reported work of Hammond and Williams in England. They found that after a given adhesion test there was always a given thickness of deposit left, and made attempts to correlate this with lattice sharing theories between the plate and the substrate In the present experiments however, the nature of the chromium deposits after "throw-off" varied from smooth layers of extremely small thickness to very non-uniform deposits of rough (by several order of magnitude) texture. One opinion that might be offered at this time is that quite possibly in any method which involves considerable shear, there would be a tendency to leave a deposit of uniform thickness, and it seems quite possible that there was considerable shear involved in the method used by Hammond and Williams. This would also contribute to the fact that the maximum adhesion values obtained by those workers were considerably lower than those obtained by the magnetic support method.

As far as specific conclusions are concerned, these are presented in the previous section. Perhaps the most interesting of these, at least from a fundamental standpoint, are the decrease of adhesion force with repeated platings in the same bath, even though the bath is apparently maintained under constant conditions by the addition of chemicals, and the powerful effect of a brief reverse etch treatment in the bath prior to plating. It would appear as though there is almost nothing one can do to reduce the adhesion of a given deposit provided that a brief reverse etch is used in conjunction with a fresh plating bath. However, it appears as though one should be very careful as far as the freshness of the plating bath is concerned if good adhesion is desired.

Acknowledgement

In addition to the financial support given by the Department of Defense, through the U.S. Army Ordnance Corps, assistance, advice, and criticism of tremendous value has been received from Dr. Peter R. Kosting, Mr. Murray M. Jacobson, and Mr. Charles Levy of the Watertown Arsenal Laboratories. The success of the program at the Ordnance Research Laboratory has been primarily due to the efforts of Mr. William H. Dancy, Jr. who served as Project Supervisor and Mr. William Bland who so diligently operated the testing equipment.

X. CONCLUSIONS

The conclusions which can be drawn from this work are summarized below. It must be emphasized that in most cases these conclusions must be regarded as preliminary since they are based on only a small amount of data. The reader is advised to consult the body of this report for a more detailed discussion of these points.

1. The experimental method has been established as a powerful tool for the quantitative measurement of "effective adhesion forces".

2. The adhesion decreases rapidly with the length of use of a given plating bath even though concentrations are maintained by the addition of chemicals.

3. A 15 second reverse etch in the bath just prior to plating assures almost maximum adhesion no matter what the prior treatment of the substrate.

4. A hydrochloric acid dip is also a very effective pre-bath cleaning treatment and its use almost guarantees maximum adhesion.

5. A vapor honing just prior to plating reduces the adhesion of the plate considerably.

6. Immersion of the test sample in protein-type contaminants just prior to plating produced erratic results which in no way could be interpreted as definitely reducing the adhesion. In fact, in some cases it appeared to increase the adhesion over the normal. Both human serum albumin and fibrin peptone were used.

7. The use of organic phosphates, as typified by DuPont Ortholeum-162, as a contaminant did cause considerable reduction in adhesion, and there was some evidence that the degree of adhesion could be controlled by controlling the concentration of the contaminant dip.

8. Ordinary motor oil, SAE # 30, when used as a contaminant dip had a marked effect on the reduction of adhesion, but was completely uncontrollable.

9. Very preliminary results indicate that adhesive force increases as the thickness of the plate decreases which is generally in agreement with the results of other workers.

10. The thickness of the chromium plate has very little effect on the tensile strength of Martensitic and Bainitic steels when they are used as the base metal. A plate thickness of 0.01 inches only reduces the tensile strength by about 10% or less.

11. The thickness of chromium plate applied to Pearlitic steel as a base metal has a very curious effect on the tensile strength of the base metal. The tensile strength actually increases with increasing thickness of chromium plate up to

a thickness of about 0.0035 inches. At this point the tensile strength is about 15% higher than for the base metal. A subsequent increase in the film thickness causes the tensile strength of the steel to decrease.

XI. BIBLIOGRAPHY

Originally it had been intended to make a critical review of the literature in connection with the activities under this contract, but lack of time and funds rendered this impossible. Consequently, the following bibliography is presented without comment as containing information of definite value to the factors involved in the consideration of the adhesion of electrodeposited chromium.

In order to assist the user the bibliography is divided into the following classifications. A certain amount of overlapping is unavoidable and no attempt has been made to cross reference.

1. General References - Many of these contain extensive bibliographies.

2. Preparation of the Metal for Plating.

3. Plating Solutions and Processes.

4. Chromium Plating.

5. Adhesion Testing Methods.

6. Adhesion Control Methods.

7. Effects of Grain Orientation and Crystal Structure on Adhesion.

8. Effect of Film Thickness on Adhesion.

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Effect of Length of Use of Plating Bath on Adhesion

Sample No.	Type of Band	Rotor Dia. inch	Plate thickness inch	Max. Rotor Speed-rps	Max.Stress psi	Comments (see text for furthe details)
5.3-A	Solid	.1833	.0065	15,800	4,000	Group A
5.3-C	8 Slots	.1833	.0062	12,500	2,400	Motor oil
5.3-D	Solid	.1833	.0064	28,200	12,400	contaminant.
5.4-A	8 Slots	.1833	.0063	22,800	8,000	No reverse etch, fresh
		Average maximum stress -------			6,700	plating batl
4.27-A	Solid	.1862	.0066	19,000	5,900	Group B
4.28-A	8 Slots	.1862	.0073	16,200	4,500	Similar to
4.28-B	8 Slots	.1862	.0071	17,800	5,600	Group A.
4.29-A	Solid	.1862	.0070	19,600	6,300	Fresh
		Average maximum stress -------			5,600	plating batl
4.25-A	8 Slots	.1862	.0089	5,200	600	Group C
4.25-B	8 Slots	.1862	.0086	9,800	2,000	Same treat-
4.25-C	Solid	.1862	.0081	9,600	1,900	ment as
4.25-D	Solid	.1862	.0084	7,200	1,100	Group B.
4.25-E	8 Slots	.1862	.0089	8,200	1,500	No reverse
		Average maximum stress ---------			1,425	etch, plated in same batl as Group B.
5.5-A	8 Slots	.1815	.0092	41,000	38,500	Group D
5.6-A	Solid	.1815	.0884	44,200	40,500	Motor oil
5.9-A	8 Slots	.1815	.0087	42,600	39,100	contaminant.
5.10-A	Solid	.1815	.0086	42,900	39,300	Fresh plat- ing bath. 1!
		Average maximum stress --------			39,300	sec. reverse etch in plai ing bath.
5.11-A	8 Slots	.1797	.0099	40,000	38,800	Group E
5.16-A	Solid	.1797	.0089	42,200	39,100	Ortholeum-
5.17-A	Solid	.1797	.0090	42,000	39,100	162
5.18-A	8 Slots	.1797	.0098	45,600	50,600	contaminant, 15 sec.
		Average maximum stress---------			41,900	reverse etcl plated in fresh bath.
4.13-A	Solid	.1833	.1017	31,300	26,900	Group F
4.14-A	8 Slots	.1833	.0102	34,800	31,500	Same pre-
4.15-A	Solid	.1833	.0105	33,400	29,800	bath treat-
4.18-A	8 Slots	.1833	.0101	26,900	18,600	ment as Grou E - 15 sec.
		Average maximum stress ---------			26,700	reverse etcl plated in same bath a Group E.

TABLE 2

Effect of Reverse Etch on Adhesion

Human Serum Albumin Contaminant

Sample	Type of Band	Rotor Dia. Inch	Plate Thickness-Inch	Max. Rotor Speed-rps	Max. Stress psi	Comments
7.21-A	8 Slots	.1808	.0089	34,600	25,600	Dip 30 sec. in human serum albumin solution 15 gm/liter; rin 2 min in running water; plate in fresh bath @ 2A/
7.22-A	Solid	.1808	.0085	36,500	28,000	
7.22-B	8 Slots	.1808	.0089	36,400	29,100	
7.25-A	Solid	.1808	.0087	37,300	29,700	
			Average		28,100	
7.26-A	Solid	.1792	.0096	40,500	38,900	Same as above bu with 15 sec. reverse etch in bath before plat ing.
7.27-A	Solid	.1792	.0096	41,500	40,800	
7.27-C	8 Slots	.1792	.0097	37,300	33,600	
7.28-A	8 Slots	.1792	.0097	39,500	37,600	
			Average		37,700	
			Ortholeum 162-Contaminant			
5.24-A	Solid	.1877	.0100	11,800	3,600	Dip 30 sec. Ortholeum-162. 10cc/liter, rins in running water 2 min, plate in fresh plating bath.
5.24-B	Solid	.1877	.0078	16,600	5,500	
5.25-A	Solid	.1877	.0093	12,600	3,800	
5.27-A	Solid	.1877	.0093	11,000	2,900	
			Average		3,950	
5.11-A	8 Slots	.1797	.0099	40,000	38,800	Same as above except 15 sec. reverse etch in plating bath bef plating.
5.16-A	Solid	.1797	.0089	42,200	39,100	
5.17-A	Solid	.1797	.0090	42,000	39,100	
5.18-A	8 Slots	.1797	.0098	45,600	50,600	
			Average		41,900	
			Motor Oil Contamiaant			
4.27-A	Solid	.1862	.0066	19,000	5,900	Dip 2 min in # 30 oil, wipe off excess oil with paper towel; pla in fresh plating bath 2A/in^2
4.28-A	8 Slots	.1862	.0073	16,200	4,500	
4.28-B	8 Slots	.1862	.0071	17,800	5,600	
4.29-A	Solid	.1862	.0070	19,600	6,300	
			Average		5,600	
5.5-A	8 Slots	.1815	.0092	41,000	38,500	Same as above except 15 sec. reverse etch in bath before plat ing.
5.6-A	Solid	.1815	.0084	44,200	40,500	
5.9-A	8 Slots	.1815	.0087	42,600	39,100	
5.10-A	Solid	.1815	.0086	42,900	39,300	
			Average		39,300	
			Base Metal - No Contaminant			
9.27-A	8 Slots	.1787	.0123	30,700	29,200	No contaminant used, no reverse etch prior to plating.
9.27-B	8 Slots	.1788	.0115	31,300	28,300	
9.28-A	Solid	.1790	.0110	28,900	23,000	
9.28-B	Solid	.1791	.0109	29,000	22,900	
			Average		25,800	
3.16-A	8 Slots	.1866	.0105	40,400	44,300	No contaminant used. 15 sec. reverse etch in bath prior to plating.
3.17-A	8 Slots	.1866	.0104	42,200	47,800	
10.3-A	Solid	.1866	.00985	41,100	43,300	
10.3-B	Solid	.1866	.0098	40,500	41,200	
			Average		44,025	

TABLE 3

Effect of Special Pre-Bath Cleaning Treatment on Adhesion

Sample	Type of Band	Rotor Dia. Inch	Plate Thickness-Inch	Max. Rotor Speed-rps	Max. Stress psi	Comments (see text for further details)
8.2-A	8 Slots	.1822	.0092	34,300	27,000	Group A
8.8-A	Solid	.1822	.0071	47,000	38,000*	HCl dip followed
8.10-C	8 Slots	.1822	.0076	48,500	44,200	by electropolish
8.11-A	Solid	.1822	.0070	47,800	38,700*	
			Average maximum stress		37,000	
7.29-A	Solid	.1820	.0081	44,800	40,200	Group B
8.3-B	8 Slots	.1820	.0088	43,500	41,200	Vapor hone
8.9-A	8 Slots	.1820	.0102	36,600	34,500	followed by
8.10-B	Solid	.1820	.0080	44,300	38,800	HCl dip.
			Average maximum stress		38,700	
8.9-B	Solid	.1820	.0047	31,200	10,700	Group C
8.9-C	8 Slots	.1820	.0050	35,800	15,300	HCl dip prior
8.9-D	Solid	.1820	.0045	37,000	14,600	to vapor hone
8.12-A	8 Slots	.1820	.0055	37,500	18,500	which was follow ed by 20 sec.
			Average maximum stress		14,775	reverse etch in plating bath.

* Rotor exploded - stress given is that at time of explosion. Adhesion force certainly exceeded this value.

TABLE 4

Effect of Surface Contaminant Concentration on Adhesion

(Fibrin Peptone)

Sample	Type of Band	Rotor Dia. Inch	Plate Thickness Inch	Max. Rotor Speed-rps	Max. Stress psi	Comments
9.27-A	Slotted	.1787	.0123	30,700	29,200	Group A
9.27-B	Slotted	.1788	.0115	31,300	28,300	rinse 2 min. no
9.28-A	Solid	.1790	.0110	28,900	23,000	special treatment.
9.28-B	Solid	.1791	.0109	29,000	22,900	Plate 16 hrs at $2A/in^2$
			Average maximum stress		25,800	
						Group B
9.20-A	Slotted	.1837	.0106	34,200	31,800	Dip 30 sec. in Fibrin
9.21-A	Solid	.1837	.0098	38,500	36,500	Peptone solution
9.22-A	Slotted	.1837	.0098	36,000	32,300	1 gm/liter; rinse 2
9.23-B	Solid	.1837	.0095	39,900	38,300	min. in running water,
						plate 16 hrs at $2A/in^2$
			Average maximum stress		34,750	
						Group C
9.26-A	Slotted	.1842	.0135	30,900	33,900	Dip 30 sec. in Fibrin
9.29-B	Slotted	.1842	.0146	30,500	34,500	Peptone solution 2
10.6-A	Solid	.1842	.0129	33,000	36,900	gm/liter; rinse 2 min
10.6-B	Solid	.1842	.0133	32,000	35,600	in running water,
						plate 16 hrs at $2A/in^2$
			Average maximum stress		35,250	
						Group D
10.5-A	Slotted	.1828	.0104	35,800	33,700	Dip 30 sec. in Fibrin
10.5-B	Solid	.1827	.0099	31,500	24,900	Peptone solution, 4
10.7-A	Solid	.1827	.0100	31,300	24,700	gm/liter; rinse 2 min.
10.7-B	Slotted	.1827	.0109	35,300	34,700	in running water, plate
						16 hrs. at $2A/in^2$
			Average maximum stress		29,500	
						Group E
10.10-A	Slotted	.1815	.0111	32,500	29,900	Dip 30 sec. in Fibrin
10.10-B	Slotted	.1816	.0109	35,600	35,000	Peptone solution 8
10.11-A	Solid	.1817	.0102	37,100	35,300	gm/liter; rinse in
10.11-B	Solid	.1818	.0099	36,800	33,800	running water 2 min,
						plate 16 hrs at $2A/in^2$
			Average maximum stress		33,500	
						Group F
10.12-A	Solid	.1839	.0095	42,700	43,700	Dip 30 sec. in Fibrin
10.13-A	Solid	.1839	.0098	41,500	42,700	Peptone solution, 16
10.14-A	Slotted	.1839	.0105	38,800	40,300	gm/liter, rinse 2 min.
10.14-B	Slotted	.1839	.0110	36,800	38,200	in running water, plate
						16 hrs. at $2A/in^2$.
			Average maximum stress		41,200	

TABLE 5

Effect of Surface Contaminant Concentration on Adhesion

(Ortholeum-162)

Sample	Type of Band	Rotor Dia. Inch	Plate Thickness-Inch	Max. Rotor Speed-rps	Max. Stress psi	Comments
						Group A
6.6-A	Solid	.1790	.0097	18,700	7,500	Dip 30 sec. in
6.7-A	Solid	.1790	.0101	30,000	22,600	Ortholeum-162
6.8-B	Solid	.1790	.0104	27,200	19,100	solution 3 cc/
						liter, rinse in
						running water 2
		Average maximum stress			16,400	min. plate 16 hr.
						in fresh plating
						bath.
						Group B
5.24-A	Solid	.1877	.0100	11,800	3,600	Dip 30 sec. in
5.24-B	Solid	.1877	.0078	16,600	5,500	Ortholeum-162
5.25-A	Solid	.1877	.0093	12,600	3,800	10 cc/liter, rinse
5.27-A	Solid	.1877	.0093	11,000	2,900	in running water 2
						min. plate 17 hr.
		Average maximum stress			3,950	in fresh plating
						bath.

TABLE 6

Effect of Plate Thickness on Adhesion Using Ortholeum Contaminant

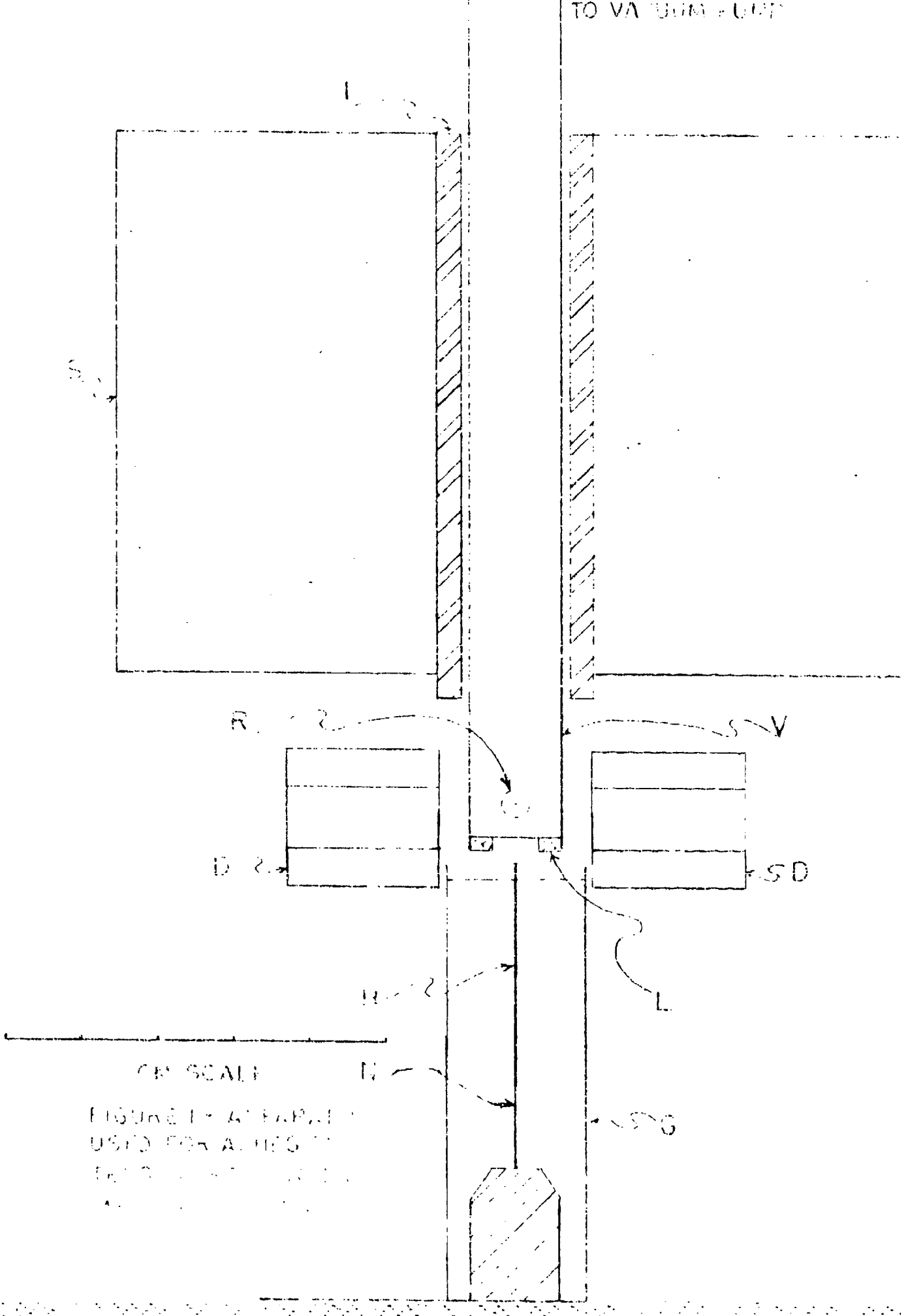
Sample	Type of Band	Rotor Dia. Inch	Plate Thickness-Inch	Max. Speed rps	Max. Stress psi	Comments
						Group A
5.24-A	Solid	.1877	.0100	11,800	3,600	Dip 30 sec. in
5.24-B	Solid	.1877	.0078	16,600	5,500	Ortholeum-162
5.25-A	Solid	.1877	.0093	12,600	3,800	10 cc/liter, rinse
5.27-A	Solid	.1877	.0093	11,000	2,900	in running water, 2 min., plate 17 hrs. in fresh bath.
						Group B
5.31-A	Solid	.1820	.0038	29,500	7,900	Same as Group A
5.31-B	8 Slots	.1820	.0038	39,800	14,100	except plated 6
6.1-A	8 Slots	.1820	.0036	21,500	3,900	hrs. 15 min. in
6.1-B	Solid	.1820	.0030	25,100	4,400	fresh bath.

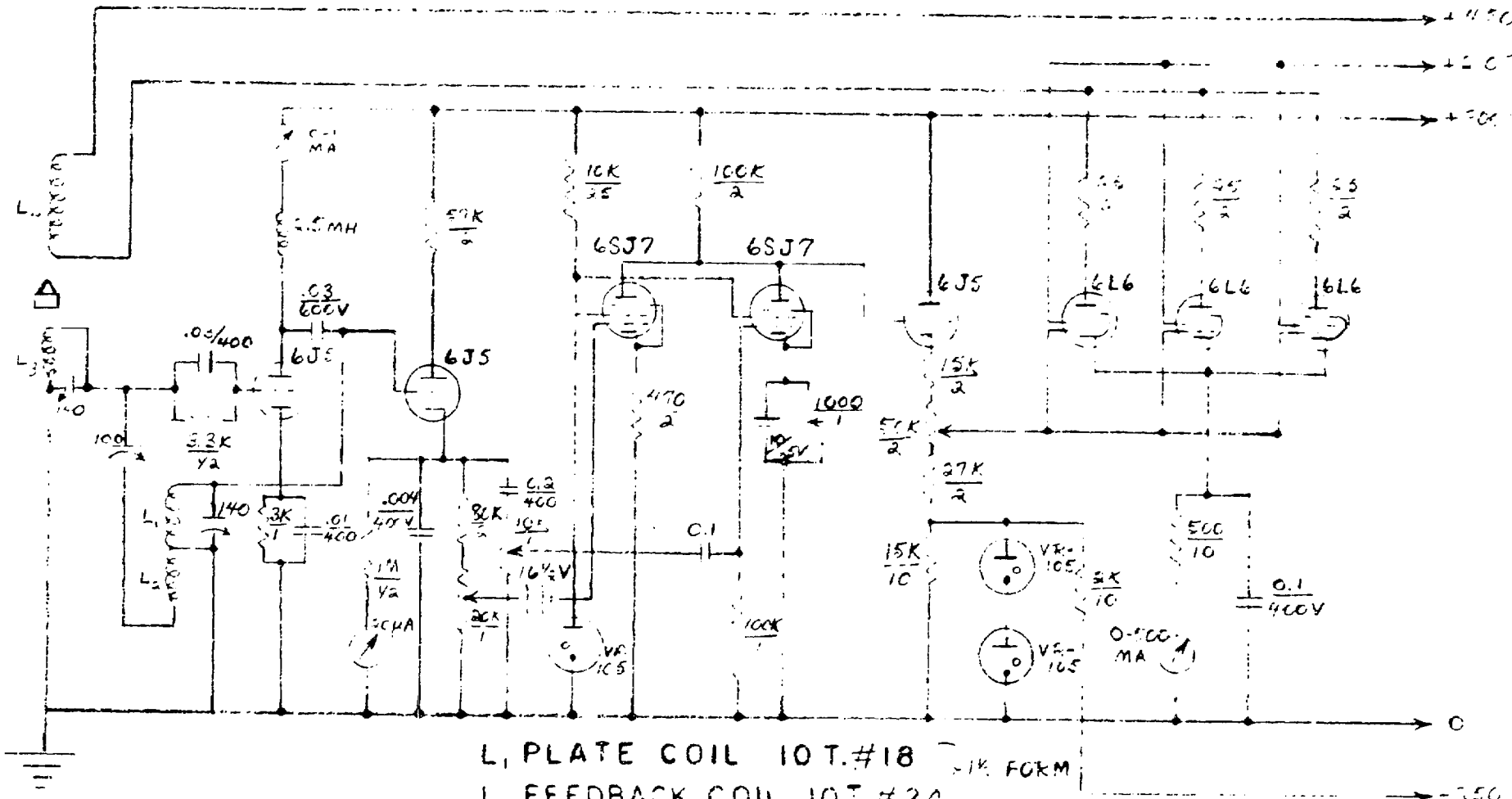
TABLE 7

Comparative Strengths of Steels of Different Microstructures

(All Samples had solid bands of chromium)

Sample	Rotor Dia. Inch	Plate Thickness-Inch	Max. Speed rps	Max. Stress on axis of rotor-psi	Comments
6.29-A	.1879	0	32,200	109,000	Pearlite Microstructure
7.8-B	.1879	0	32,000	107,700	
7.14-A	.1879	.0017	32,700	114,600	
7.15-A	.1879	.0017	33,400	119,200	
8.24-A	.1877	.0035	33,700	124,000	
8.24-C	.1877	.0035	33,700	124,200	
8.26-A	.1877	.0054	33,000	121,600	
8.29-A	.1877	.0060	32,600	115,900	
9.8 - A	.1877	.0118	30,600	112,800	
9.13- B	.1877	.0099	30,900	112,500	
10.19-A	.1878	.0130	29,000	103,000	
10.20-A	.1878	.0125	29,800	108,000	
7.7-A	.1881	0	44,100	204,700	Martensite Mircrostructure
7.12-A	.1881	0	43,900	202,000	
7.14-B	.1881	.0017	43,200	200,600	
7.19-B	.1881	.0017	43,500	202,400	
8.18-A	.1879	.0039	43,000	203,000	
8.19-A	.1879	.0037	42,500	198,200	
8.31-A	.1879	.0067	41,300	193,800	
9.1-A	.1879	.0068	40,800	189,600	
9.8-B	.1879	.0108	38,000	172,400	
9.13-A	.1879	.0100	40,300	192,100	
6.28-B	.1884	0	50,300	268,000	Bainite Microstructure
7.11-A	.1884	0	51,200	276,600	
7.18-A	.1884	.0017	49,800	266,300	
7.19-A	.1884	.0018	49,700	266,600	
8.24-B	.1882	.0045	49,400	271,000	
8.23-A	.1882	.0049	48,000	257,300	
8.30-B	.1882	.0061	47,000	250,000	
8.30-A	.1882	.0061	46,900	251,200	


TO VA
R
V
D
SD
L
G



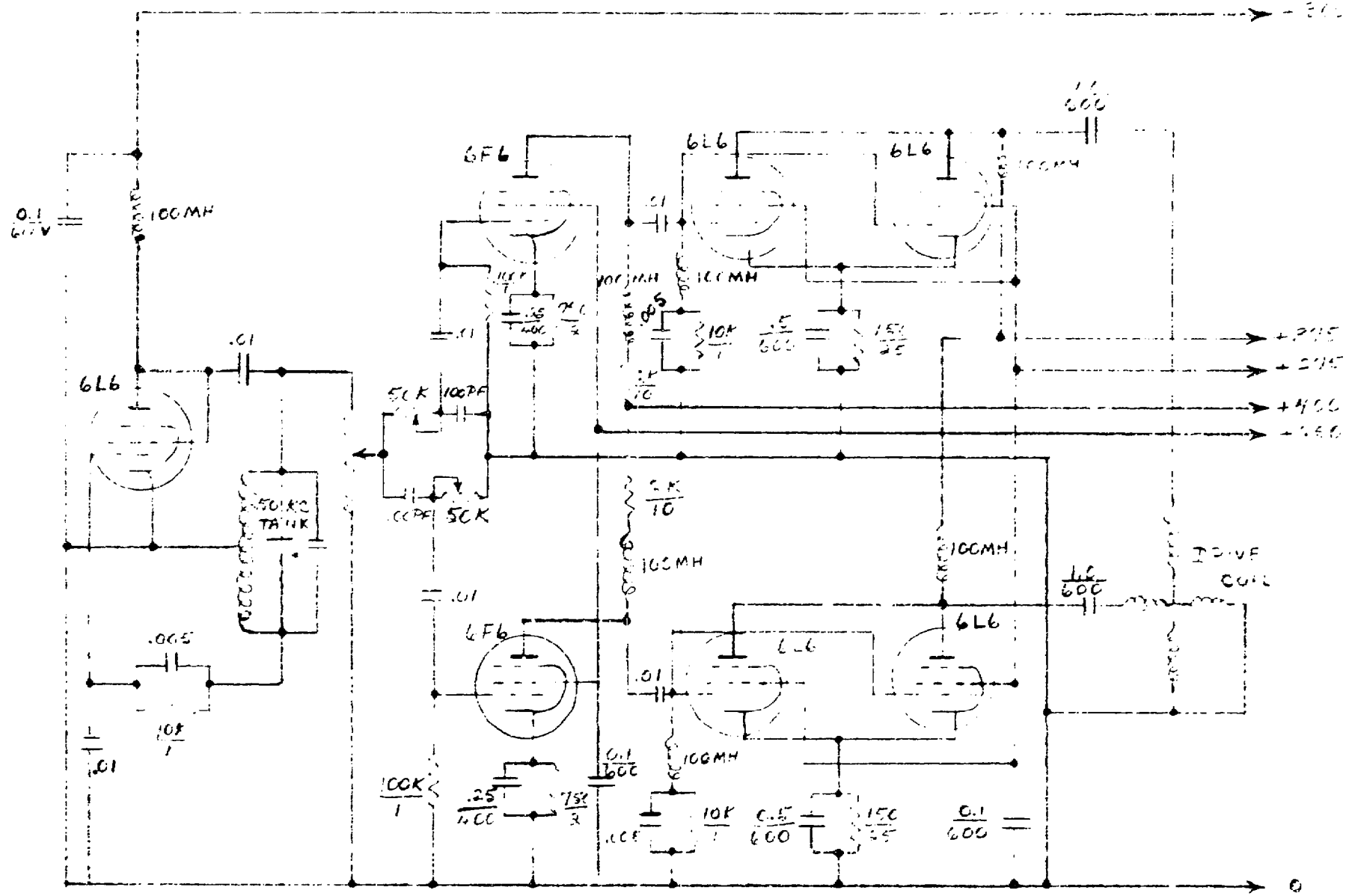
L_1 PLATE COIL 10 T. #18 - 1½" FORM

L_2 FEEDBACK COIL 10 T. #24

L_3 Q-COIL 10 T. #30 - 1½" FORM

L_4 SUPPORT COIL 25,000 T. #30

SUPPORT CIRCUIT



DRIVE CIRCUIT

FIG.3

FIGURE 4 - THE COMPLETE MAGNETIC SUSPENSION EQUIPMENT

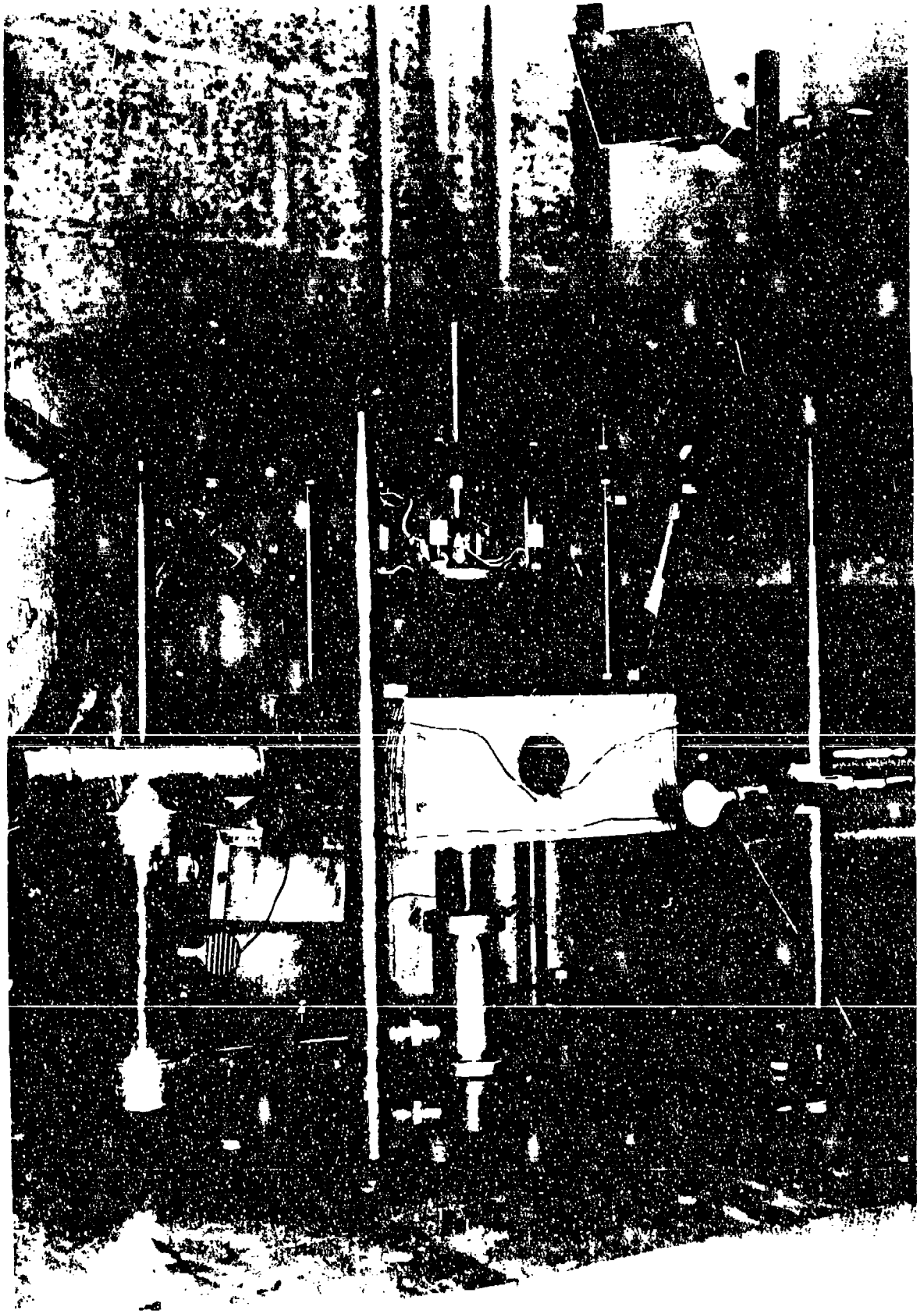


FIGURE 5 - THE MAGNETIC SUPPORT APPARATUS SHOWING SUPPORTING COIL, DRIVING COILS AND VACUUM SYSTEM

FIGURE 6 - A MAGNETICALLY SUPPORTED TEST SPECIMEN

ADHESION

AS A FUNCTION OF

LENGTH OF BATH USE

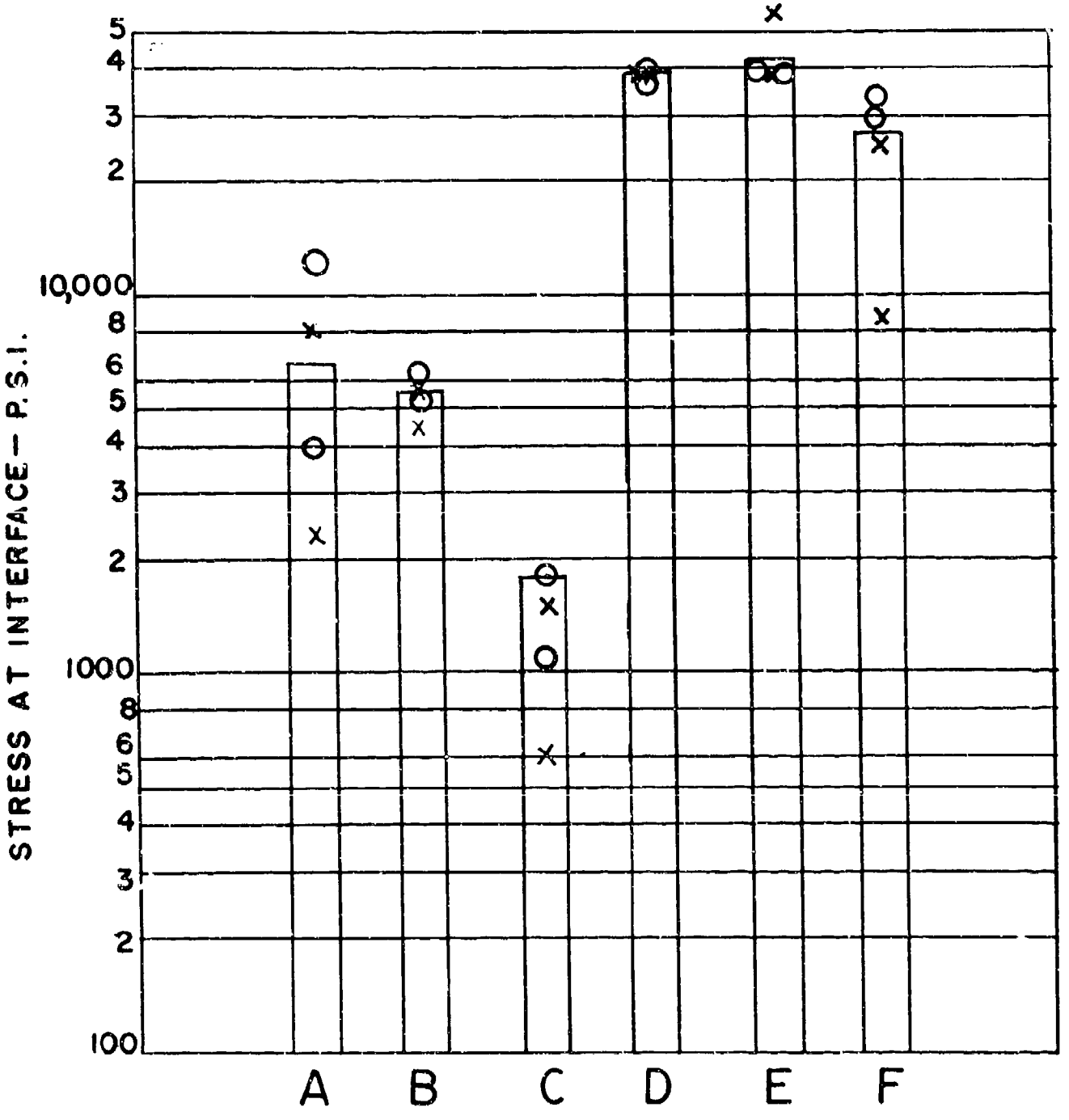


FIGURE 7

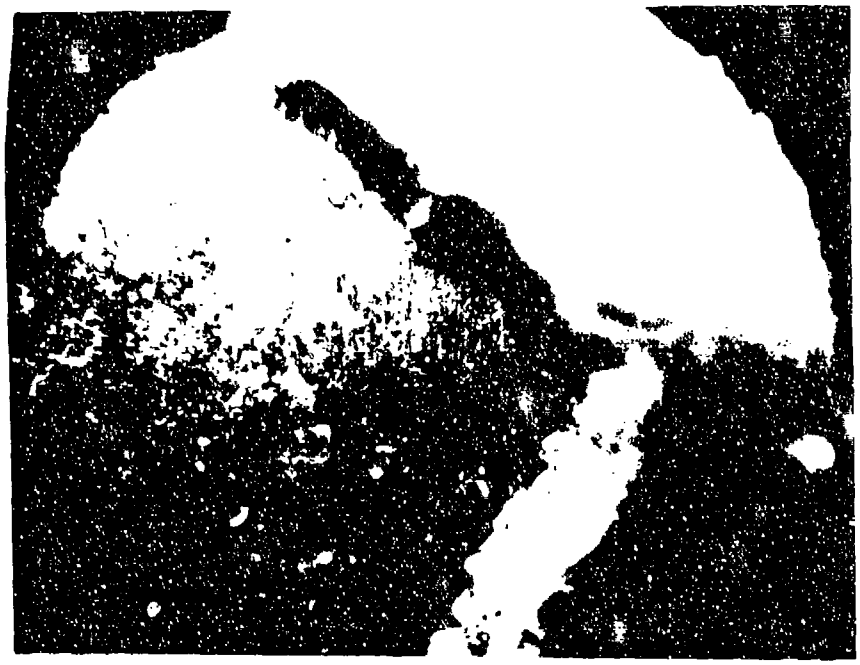


FIGURE [illegible] - CHROMIUM PLATE PULLED FROM A SPECIMEN CONTAMINATED WITH ORTHOLEUM-162 AND PLATED WITHOUT ADDITIONAL TREATMENT X45

FIGURE [illegible] - CHROMIUM PLATE PULLED FROM A SPECIMEN CONTAMINATED WITH ORTHOLEUM-162 AND PLATED AFTER A 15 SECOND REVERSE ETCH IN THE PLATING BATH X45

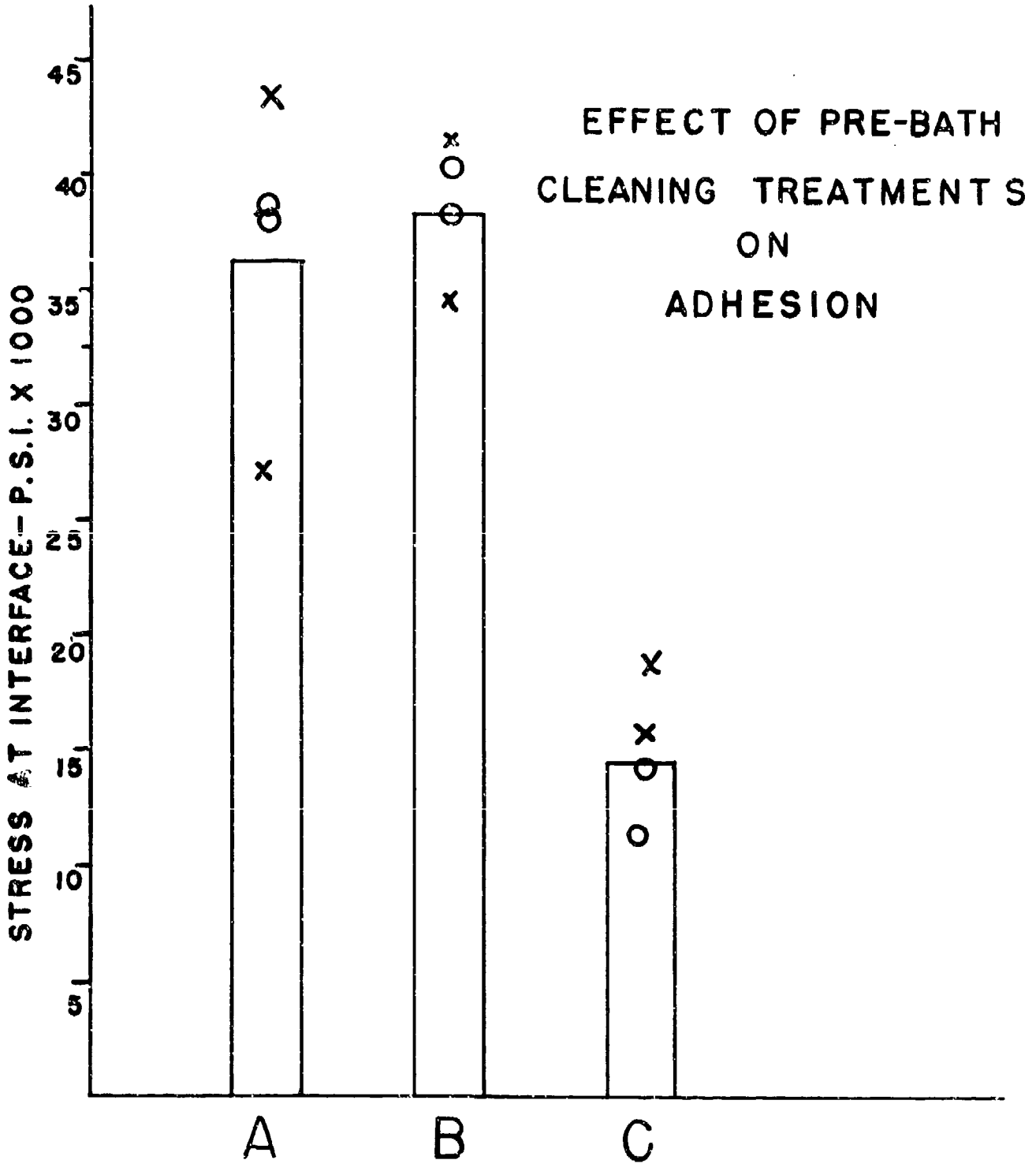

EFFECT OF PRE-BATH
CLEANING TREATMENTS
ON
ADHESION
STRESS AT INTERFACE – P.S.I. X 1000
45
40
35
30
25
20
15
10
5
A
B
C

FIGURE 11

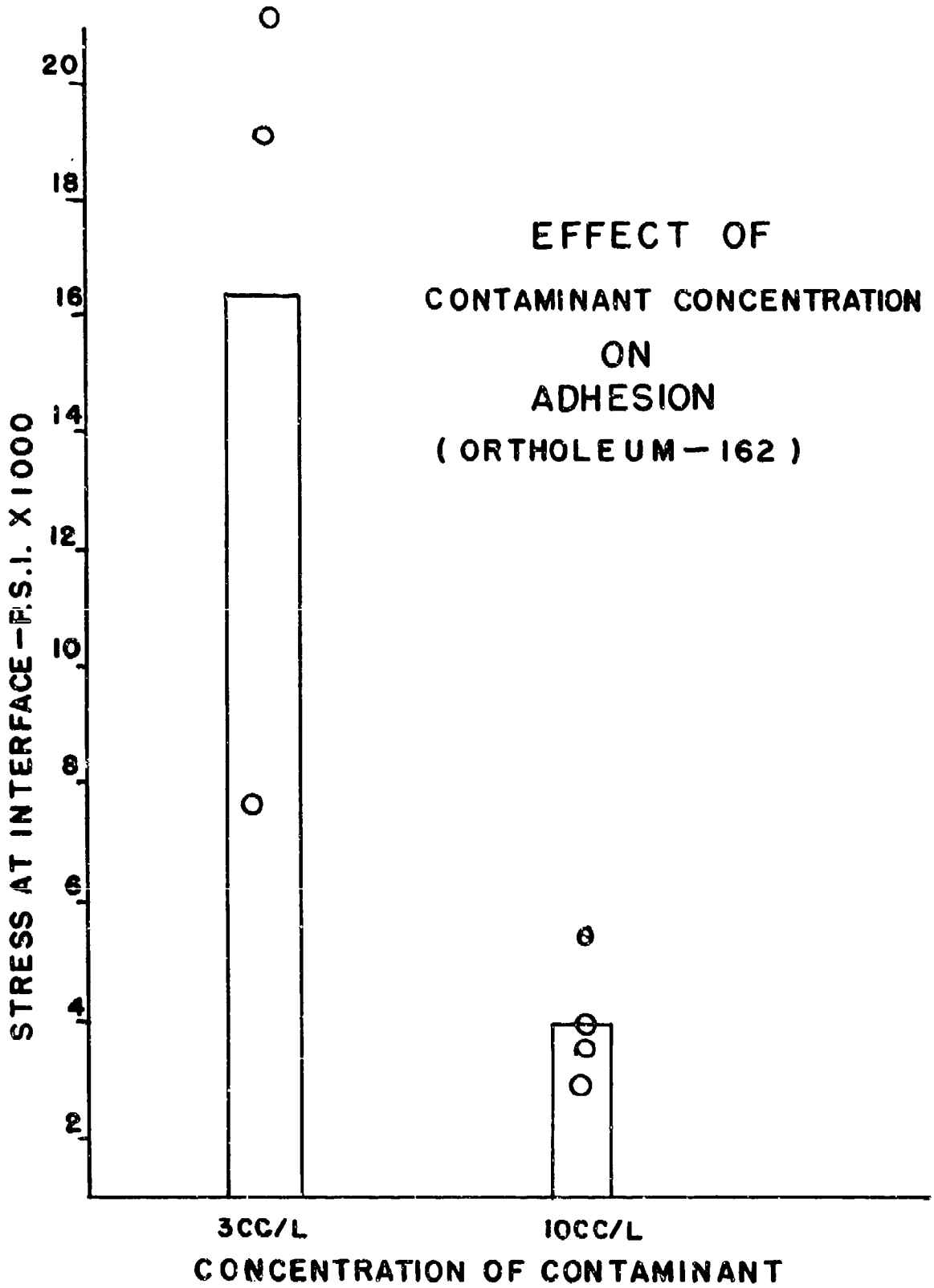

EFFECT OF
CONTAMINANT CONCENTRATION
ON
ADHESION
(ORTHOLEUM – 162)
STRESS AT INTERFACE – P.S.I. X 1000
20
18
16
14
12
10
8
6
4
2
3CC/L
10CC/L
CONCENTRATION OF CONTAMINANT

FIGURE 12

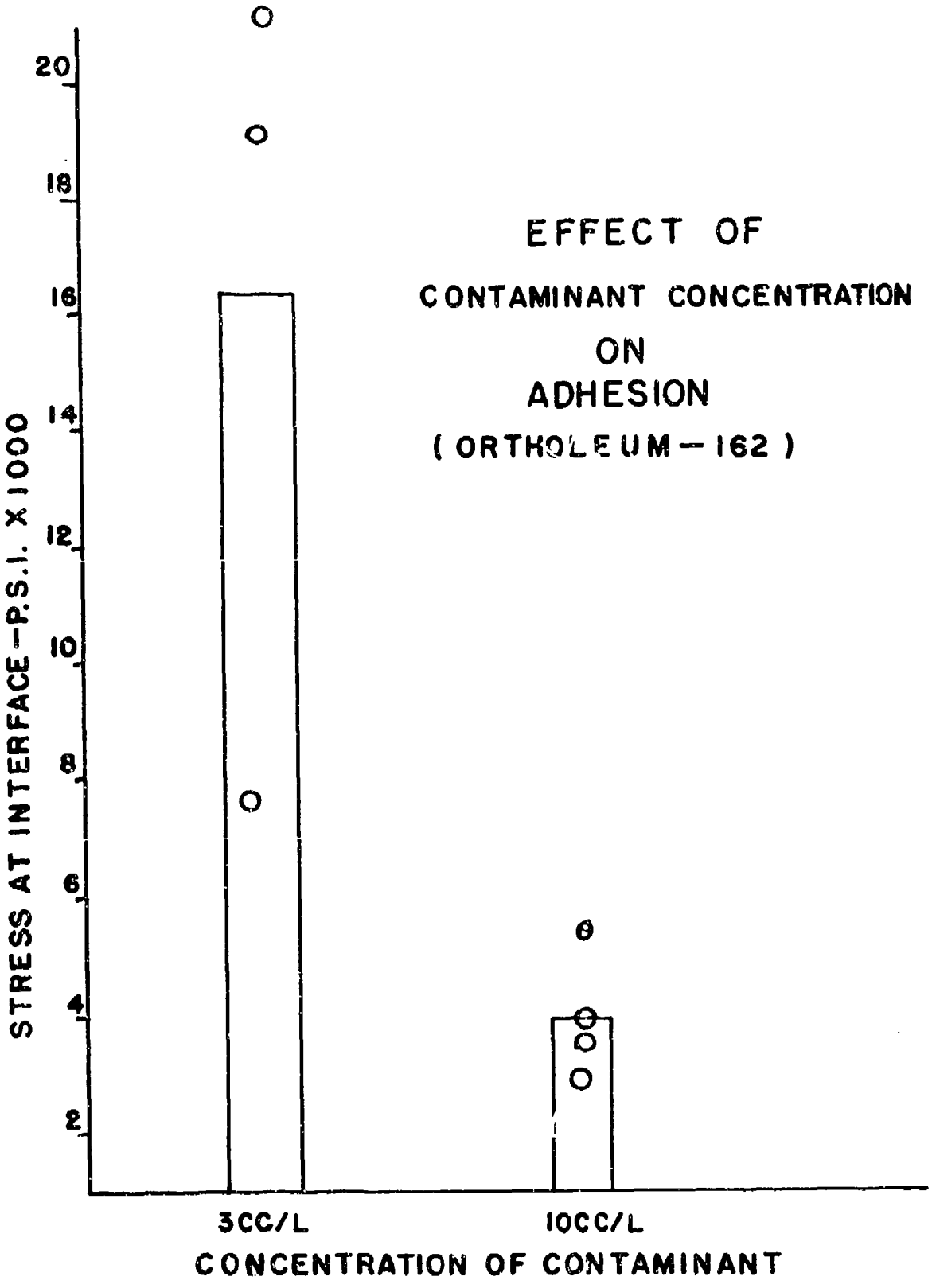

EFFECT OF
CONTAMINANT CONCENTRATION
ON
ADHESION
(ORTHOLEUM – 162)
STRESS AT INTERFACE – P.S.I. X 1000
20
18
16
14
12
10
8
6
4
2
3CC/L
10CC/L
CONCENTRATION OF CONTAMINANT

FIGURE 12

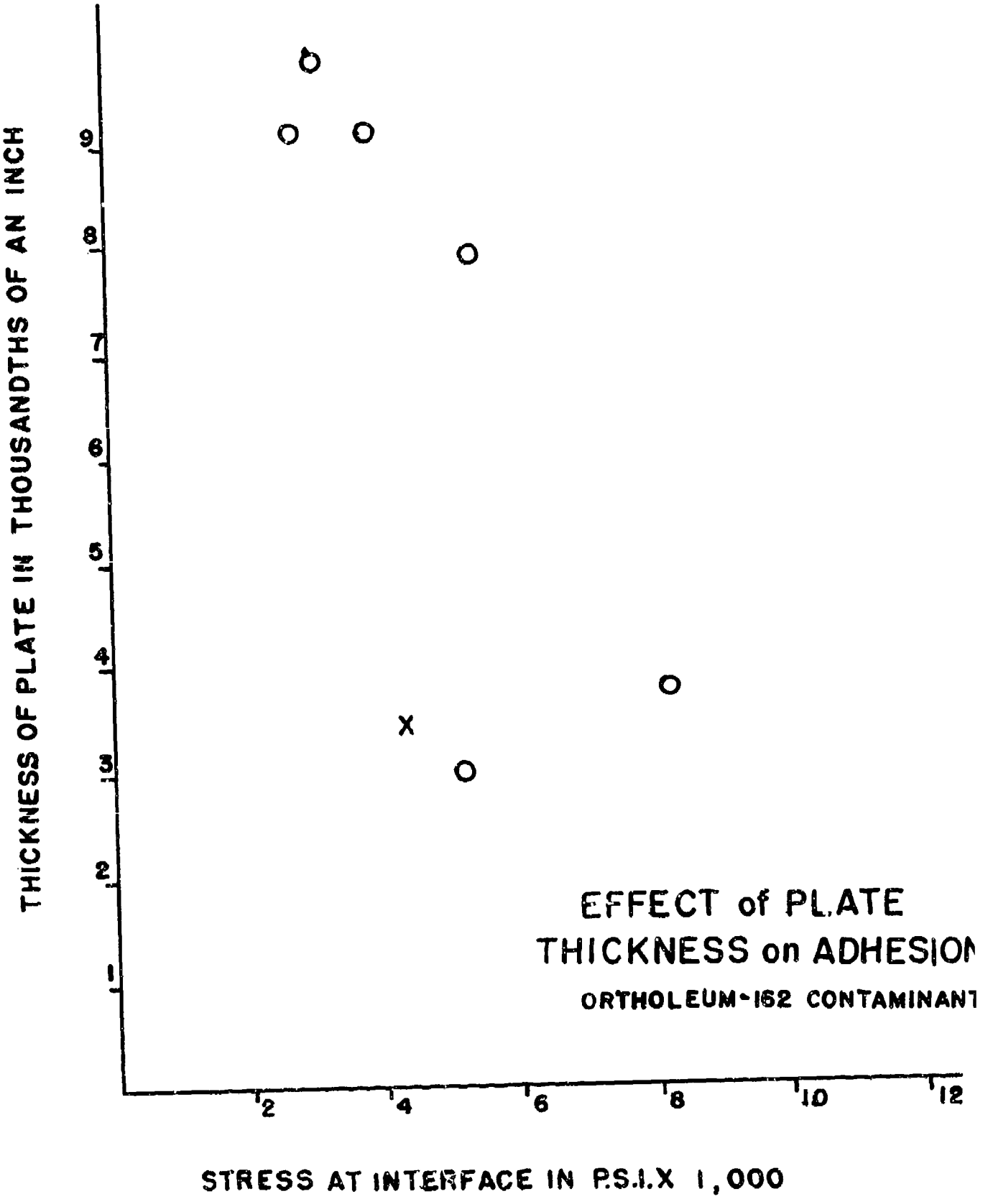

THICKNESS OF PLATE IN THOUSANDTHS OF AN INCH
1
2
3
4
5
6
7
8
9
2
4
6
8
10
12
STRESS AT INTERFACE IN P.S.I.X 1,000
EFFECT of PLATE
THICKNESS on ADHESION
ORTHOLEUM-162 CONTAMINANT

FIGURE 13

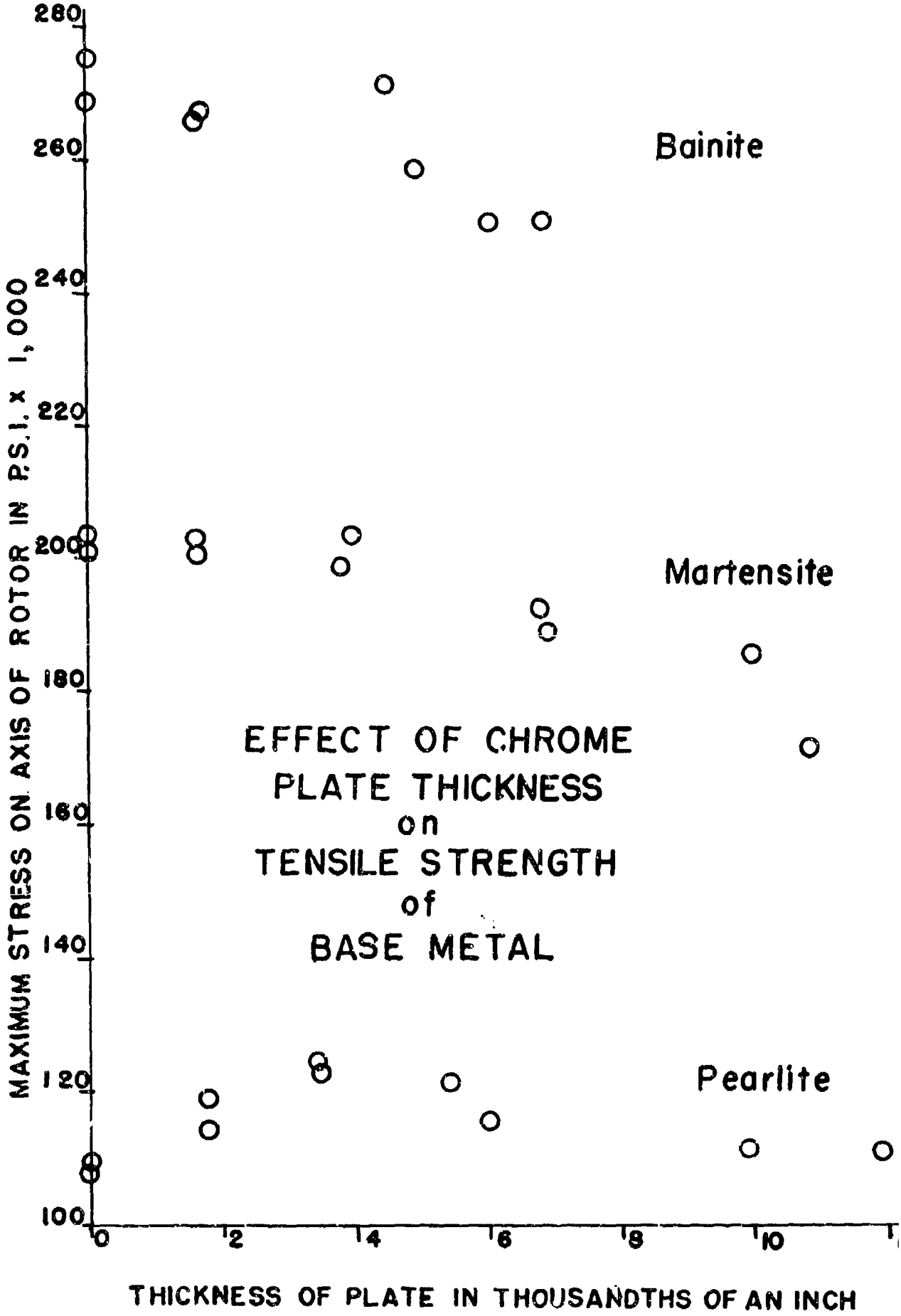

280
260
240
220
200
180
160
140
120
100
MAXIMUM STRESS ON AXIS OF ROTOR IN P.S.I. x 1,000
Bainite
Martensite
Pearlite
EFFECT OF CHROME
PLATE THICKNESS
on
TENSILE STRENGTH
of
BASE METAL
0
2
4
6
8
10
THICKNESS OF PLATE IN THOUSANDTHS OF AN INCH

FIGURE 14

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