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ADHESION BETWEEN ATOMICALLY PURE METALLIC SURFACES

PART IV

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REVIEW OF METALLIC ADHESION RESEARCH AT SYRACUSE UNIVERSITY

The Phenomena of Adhesion between metallic systems has been of considerable interest in our laboratory over the past few years due principally to the fact that through the interpretation of the adhesion process one is led to a unique understanding of such diverse fields as friction, wear, soldering, brazing, powder compacting, cold welding, machining, diffusion bonding, etc. Although apparently quite diverse in nature the common denominator of each of these fields is that two free surfaces must come into intimate contact; during each dynamic process and depending on the case, form a structurally significant or a structurally insignificant interface without the whole system ever achieving complete equilibrium. Hopefully, if the micromechanisms occurring at the interface, when two metal surfaces are brought into contact, are thoroughly and accurately defined with regards to the variables of temperature, loading pressure and contamination states for various materials and surface conditions the business of others attempting to define the mechanisms of the dynamic systems e.g., friction, soldering, etc. will be made considerably more direct. ^pTo these ends our investigations have indicated that contact resistance measurements between crossed wire samples will permit a rather fine degree of characterization of the contacting surfaces with regards to contaminants, surface roughness and mechanical properties during loading. The results have also allowed a rather complete development of the understanding of low temperature metallic adhesion. Before examining these results, however, a few definitions ought to be considered.

Adhesion may be defined (1) as the establishment of quasi-equilibrium attractive forces between two bodies in, or near, physical contact. Although

the technology of adhesion also accepts the existence of mechanical and hydrostatic effects between the two bodies in contact as adhesion forces, the phenomenological description of adhesion, as a science, should necessarily distinguish between the two. In considering only the first case, the phenomena of adhesion is common to all materials, or combinations of materials, irrespective of the nature of the cohesive forces within the systems in contact, i.e. ionic, metallic covalent or molecular. Since the adhesion process requires the establishment of a common interface between two free surfaces and the free surface properties of a material are directly related to the bulk cohesive forces within that system, the investigation of the interface resulting from an adhesion process depends on an intimate knowledge of the material which constitutes the free surfaces prior to contact. For example, the cohesive forces established across the interface between the atoms or molecules, from each free surface will establish the strength of the interface system. Or, if the free surfaces on a bulk silicate are made up of adsorbed water molecules and the contact load is insufficient to grossly disturb these adsorbed layers, then the adhesion interface is between water molecules and not silicate ions. A recent investigation by Ryan (2) conveniently emphasizes this point by demonstrating that extremely large precontact attractive forces exist between atomically clean silicate surfaces over surprisingly large separation distances; and further, these forces are immediately eliminated with foreign gas adsorption.

In order to place metallic systems into the framework of adhesion phenomena just outlined, one must recognize the monatomic nature of metallic systems and the extreme reactivity of an individual metal atom with itself or any other atomic, or molecular species. The fact that metallic crystals are formed

through cohesive forces in excess of 50 Kcal/mole between atoms suggests that interfacial linkages will be of high energy (approaching the cohesive force) and due to the atomic nature will be strongly influenced by interdiffusion across the interface at all temperatures and particularly those in excess of $0.2 T_{mp}$ (20% of absolute melting point temperature). In the very crudest of approximation for a pure system of adhesion between two surfaces of the same metal, where we might assume that the cleavage energy is equivalent only to atomic bond fracture or reverse process adhesion, "the creation of atomic bonds", the surface energy as given by Adamson (3) is $E_s = E_c/2$. Since two surfaces are lost during adhesion and all atomic bonds are assumed to be reconstructed (ideally), then the system returns to its natural state after the process: the interface loses its significance and an energy equivalent to the cohesive energy has been consumed. In dissimilar metal adhesion systems the energy consumption ought to range somewhere in the range of the sum of one half of the cohesive energy of each body making up the interface plus an additional interaction energy; i.e. heat of solution of one metal into the other. Since heats of solution between two metal systems are rarely positive and never could be expected to be more positive than the sum of the two halves cohesive energies, adhesion is anticipated between all combinations of metal pairs.

The violence of the reaction between sodium or lithium with air or water is accepted readily by any novice in chemistry; however, there seems to be a resistance to the acceptance of the statement that magnesium, iron, aluminum and most of the transition metals also react with air or water with at least

as much violence as that of sodium. The heats of oxide formation are recorded (4) which attest to this fact; however, because reactions in these systems are interrupted at one, two or several atomic layers of reaction product due to the imperviousness of this layer, we naturally and simply assume that no reaction has taken place, e. g. the mass of metal remains bright and was not massively consumed. Since adhesion is dependent on the interaction of the atomic or molecular species at the interface, a metal with a chemisorbed species at the free surface, such as water, or oxide, the contacting surfaces no longer present metallic bonds to the constructed interface and a plane of weakness is developed; that is, the adhesional forces are developed between molecular or ionic bonds in the interface rather than those of the metallic bonds which possess a relatively high shear strength. One might interject that this is the most desirable property of a good lubricant.

One of the well established rules in surface chemistry indicates that the direction of chemical reactions in the surficial layer will proceed such as to reduce the surface energy of the system. Since free metal surface energies are amongst the highest known and the heats of reaction of most metals with non-metallic species are generally large, we would expect that most free metal surfaces will accumulate almost any other non-metallic atom by any mechanism available. This also has been adequately substantiated in the literature (5). Three sources or mechanisms of impurity accumulation on an ideally clean metal surface have been generally discussed; first, from the ambient gas phase which can be eliminated by an investigation in ultra high vacuum below 10^{-9} Torr; from the solid phase by diffusion to the surface which can only be eliminated by

reduction of the contaminant species in the bulk prior to experiment and from surface creep or diffusion, processes along supporting components in physical contact with the sample under investigation. A metal surface exposed to ultra high vacuum without surface contaminants constitutes what is generally accepted as an ideal metal-vacuum interface.

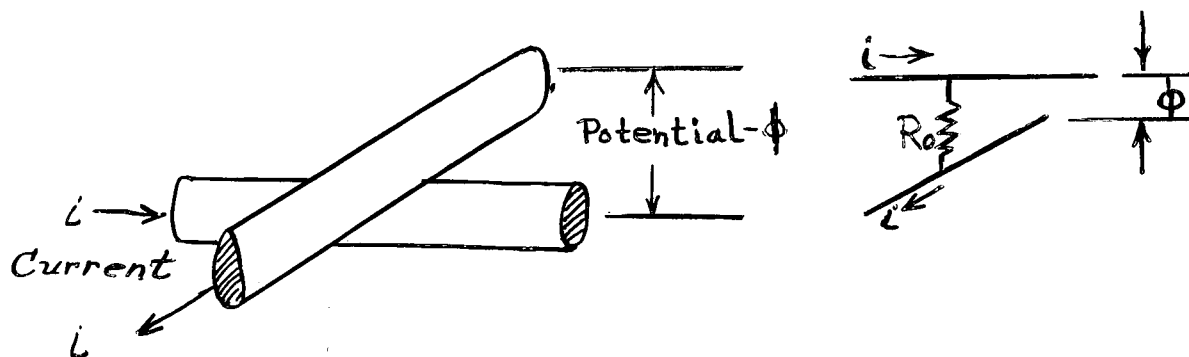
From an experimental standpoint the investigation of the adhesion forces between two metallic bodies, having ideal metal-vacuum interfaces prior to contact, reduces to an investigation of the deformation of surface asperities in order to accept the applied load impressed between the two bodies. i.e. the nature of the real contact area under an applied load. As developed by Bowden (6), Tabor (6), Archard (7), Williamson (8), and others (9) the morphology of a metallic surface under average laboratory conditions is never an atomically flat plane but usually consists of a multitude of asperities the shape of which is dependent on the prior history of the sample. Under the most ideal conditions these asperities establish a rounded hill and valley contour in the size range of one micron and presumably deform during contact according to laws developed through bulk deformation analysis. Surface contaminant effects on this deformation process have been proposed but as yet have not been investigated, even though they are intimately involved in a number of the dynamic processes and in particular the friction process. For the time being, therefore, it must be assumed that both clean and surficially contaminated metal systems in contact expand the real contact area during normal loading by a relationship to the applied load in a similar manner. The asperity deformation process during metal-metal contact produces a secondary effect on contaminated surfaces; that is the surficial

contaminant phase is mechanically dispersed into the substrate as is so prevalent in roll bonding studies (10) where the contact area is grossly expanded under conditions of restricted availability of contaminant. Although one would suspect that the rate of contaminant phase dispersal would be dependent on the amount, availability and nature of the contaminant as well as the mechanism of asperity deformation, no quantitative data are yet available and none should be expected in the immediate future due to the complexity of the system and the limited amount of study that has directly attended this problem. There is little question that in the process of friction, wear probably enters in at this point, that is the interaction of two solid surfaces to disperse brittle or weakly bound phases to expose pure substrate material along the mutual interface which results in metal transfer.

Since the purpose of these investigations was to clearly characterize the process of metallic adhesion, a technique had to be established which would permit two surfaces of known degree of contamination to be brought into contact at a known normal load; and also be capable of the detection of the fracture process while the system is unloaded. Interfacial or junction fracture strength is the only technique presently available to characterize the stability of an adhesion interface. Since, it has long been known that contaminants reduce metallic adhesion junction strength and that the contaminant can be dispersed by putting energy into the interface region, e. g. heat, plastic flow, vibration, etc. The investigation of adhesion immediately reduces to the determination of the behavior of the uncontaminated pure state, e.g. ideal interface between two metals or a standard state. Thereafter, the effect of specific amounts of

particular contaminants on this pure system must be ascertained.

An added advantage to such an investigation would be effected, if a secondary measurement could be added to the system which has a direct relationship to the character of the interface of the contacting system. Although numerous possibilities for such a measurement exist, e.g. contact potential, low energy electron diffraction, capacitance, thermal conductivity, acoustical conductivity etc., only contact resistance has been characterized (11) sufficiently to provide a simple direct method. Clearly, contact resistance has numerous disadvantages; however, it is felt that as experience in this technique is developed it will provide far fewer disadvantages than the other methods since each of the techniques is structure sensitive and subject to the multi-point contact problem. The cross wire contact resistance method provides only the interface resistance (R_0) of the system as shown



Since only the material in the interface plane is involved in the measurement, the resistance values are extremely sensitive to the presence of contaminant layers. This fact is clearly illustrated in Figure 1 in which the contact resistance between silver-silver and silver-tungsten couples is examined under various conditions of surface cleanliness. In effect nearly three orders of magnitude change are experienced in contact resistance values between contaminated surfaces and clean surfaces. Since contact resistance data can be measured readily to the third significant figure in the milliohm range; and if desirable, in a continuous fashion by recording techniques during loading processes, the resistance measurement appears to be the most suitable for characterization of contacting surfaces as well as the adhesion process itself. Earlier investigations (12) have characterized the nature of electrical and thermal currents through extremely small contact regions and have established suitable operating limits such that current flow during the contact resistance measurement does not disturb the system under investigation. Qualitative characterization of the nature of the conductivity of the contaminant in the interface layer may also be made by low temperature and/or alternating current resistance measurements as has been shown in previous investigations (11).

Since contact resistance is so appealing for the characterization of the surfaces during metal contact phenomena let us briefly examine what is known about contact resistance and what further information may be extracted from such measurements. Kisluik (13) showed that the observed contact resistance (R_o) is related to two factors:

$$R_o = R_c + R_t$$

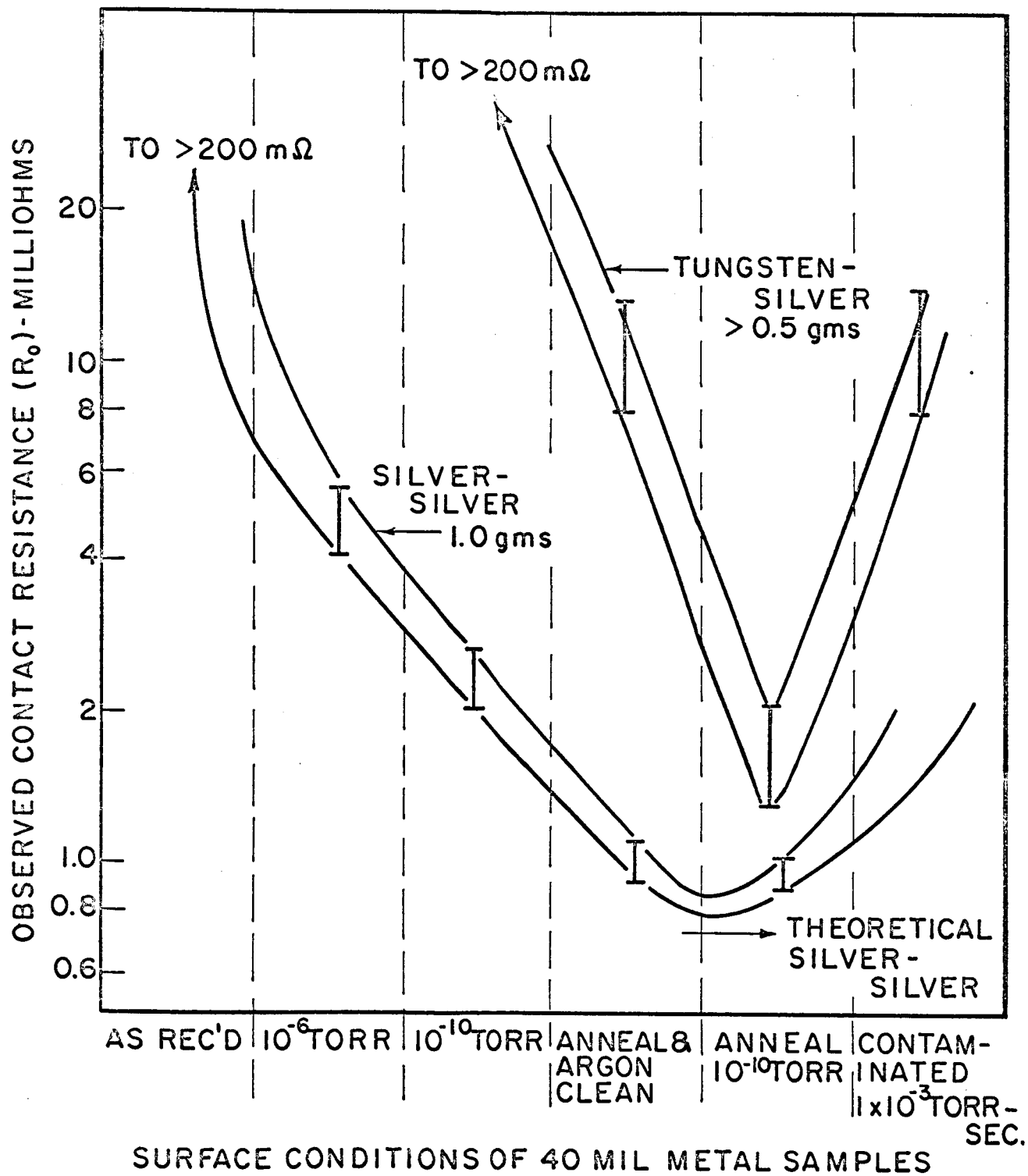


Figure 1 Contact Resistance as a function of surface cleanliness for silver-silver and silver-tungsten couples under a one gram load.

where R_c is called a constriction resistance or that due to the narrowing of the lines of force through a metallic neck region and R_f is the resistance due to the resistance of a contaminant film or the increase in conductive path area due to tunneling effects. If contact resistance of an ideally clean surface system is involved, R_f may be assumed quite small (11) and neglected without serious error.

For a single contact point the constriction resistance (R_c) was shown by Holm (11) to be:

$$R_c = \frac{\rho}{2a} \quad 2$$

where ρ is the bulk resistivity of the metal (ohm-cm) and a is the radius of a single contact in centimeters. Recently Greenwood (14) explored the effect of multicontact points, e.g. asperity effects, in the contact zone on the constriction resistance. The relationship was reported as a ratio between the area of contact as given by Holm's equation (A_I) to that of the real area of contact (A_n) and may be expressed as

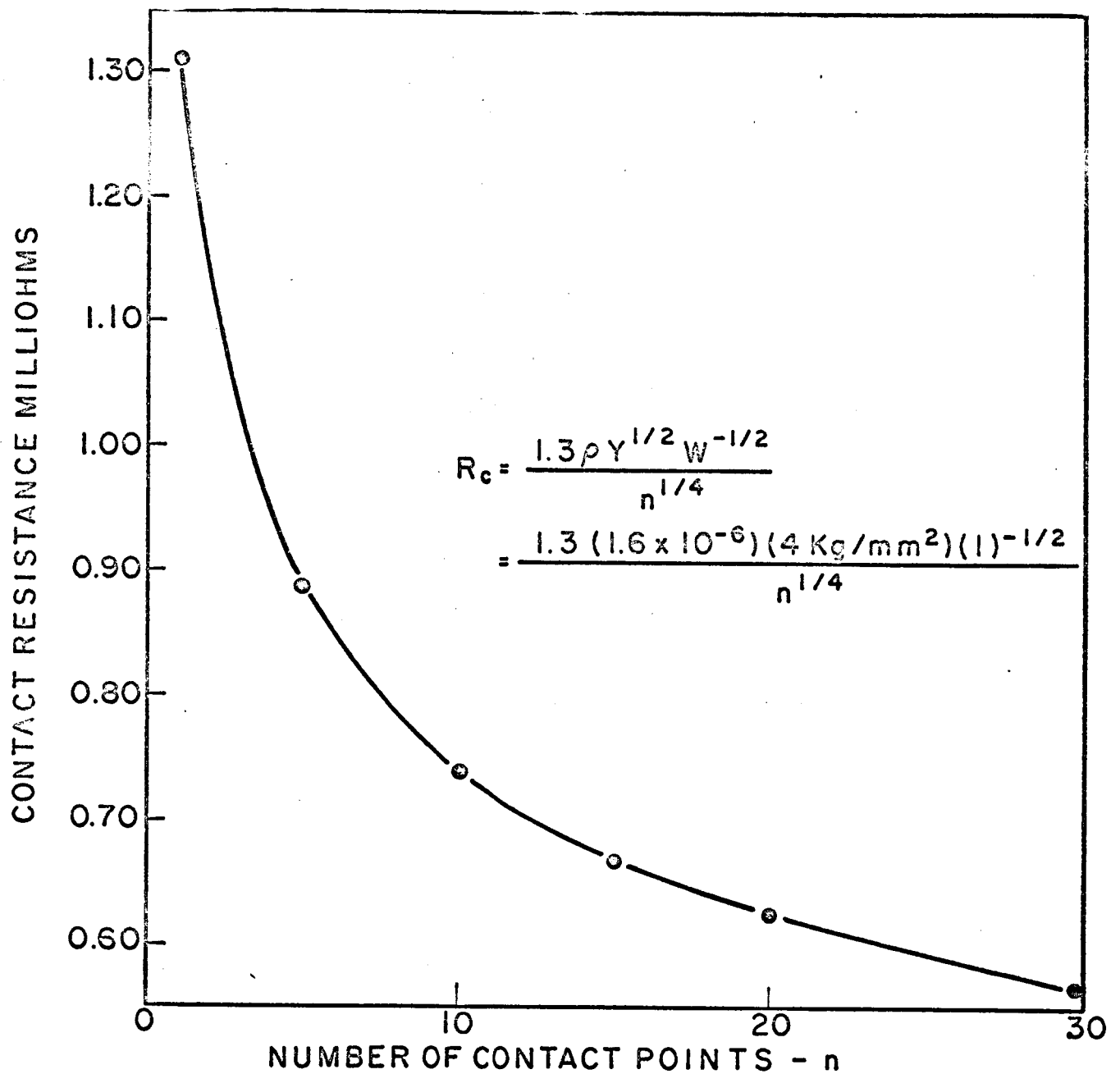
$$\frac{A_I}{A_n} = 1.4n^{1/2} \quad 3$$

where n is the number of contact points involved in the real contact area. By substituting into equation 3 both Holm's relationship ($A_I = \pi a^2 = \pi \left(\frac{\rho}{2R_c} \right)^2$) and a very elementary expression for the increase in the real area as the load in the system is increased presuming only plastic deformation or ($A_n = \frac{W}{3Y}$), where W is the impressed load and Y the yield point of the metal involved in the contact a relationship between contact resistance and load is achieved

$$R_c = 1.3N^{-1/4} Y^{1/2}_p W^{-1/2}. \quad 4$$

Equation 4 indicates that the observed contact resistance (R_o , equation 1) between two clean metal surfaces is related to the reciprocal of the square root of the load through three variables; 1) number of contact points (n) 2) yield point (y) of the material in the surficial region 3) conductivity ($1/\rho$) of the material in the surficial region. Let us consider each variable and its particular relationship to the loading and unloading of two ideally clean crossed rods of about 60 mills diameter in a normal clean adhesion experiment. In the case of silver couples and assuming all other variables constant, the effect of the number of contact points under a one gram load is illustrated in Figure 2. The variation in contact points changes the contact resistance from 1 to 30 by about a factor of two which is quite negligible when compared to the effects of gross contamination. This effect appears even less consequential when one considers that at a one gram load the contact radius is only about two microns. Since the number of asperities for a metallurgically polished and etched surface lies somewhere in the region of 20-30 for this area, one would suspect that the number of contact points would be about this value.

Metal wire samples examined by metallographic techniques after exposure to several argon ion bombardment cleaning and ultra high vacuum annealing cycles usually produced an average of 20-30 asperity contacts per unit area (2μ radius). Williamson (15) in describing the real contact area of bead blasted aluminum surfaces illustrated that, during loading the number of contact points with load varied rapidly to some point at which the number of contact points remained remarkably constant as the load increased. This was rationalized as the major asperities expanded, satellite asperities in the adjacent areas were captured



VARIATION IN THE CONTACT RESISTANCE (R_c) OF SILVER WITH A VARIATION IN NUMBER OF CONTACT POINTS (n) AT A CONSTANT YIELD POINT AND ONE GRAM LOAD

Figure 2

(loss of one point) at a rate very close to that at which new regions were brought into contact (gain one point). The value $\underline{n}$, therefore, could be expected to increase rapidly during the very light loading stages of contact and become relatively constant until gross plastic deformation consumes the entire contact region which takes place at some point near 10% deformation.

Figure 3 examines the effect of the assumed number of contacts (n) on a theoretical curve of contact resistance versus load for high purity iron. Curve A-B assumes only one point contact. Curve C-D assumes continually increasing the number of contact points as the load is increased. The slope of A-B is $-1/2$ as indicated by the equation; and that of C-D is larger than $-1/2$. If we assume that the number of contacts is one to 0.5 gm and 30 at 1.0 gm; and thereafter, remains constant, curve AFD is produced. Initially a very steep slope; and thereafter, a $-1/2$ slope. Such will be illustrated in detail later.

The effect of changing the value of the yield point value for silver under a one gram load with all other variables constant is shown in Figure 4. The yield point may be increased by work hardening effects during compression and/or the absolute value for the metal under investigation may not be precisely known for the condition of the sample under test. Under either condition the effect is not grossly significant to the overall interpretation of one contact resistance versus loading curve during one cycle, i.e. loading and unloading, since each point is related to the prior point plus some small increment ΔY due to work hardening effects. In the overall problem of placing resistivity data

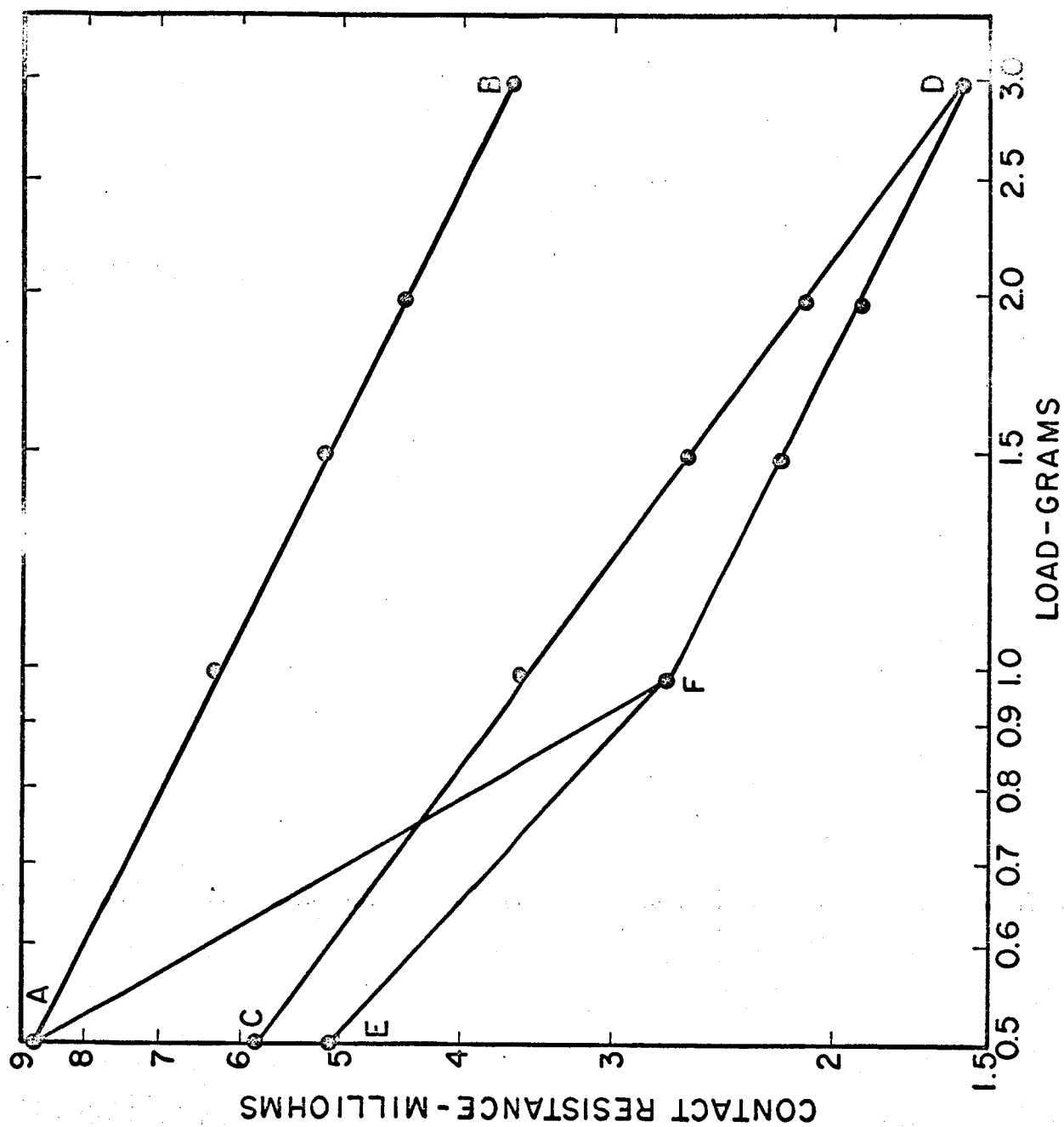
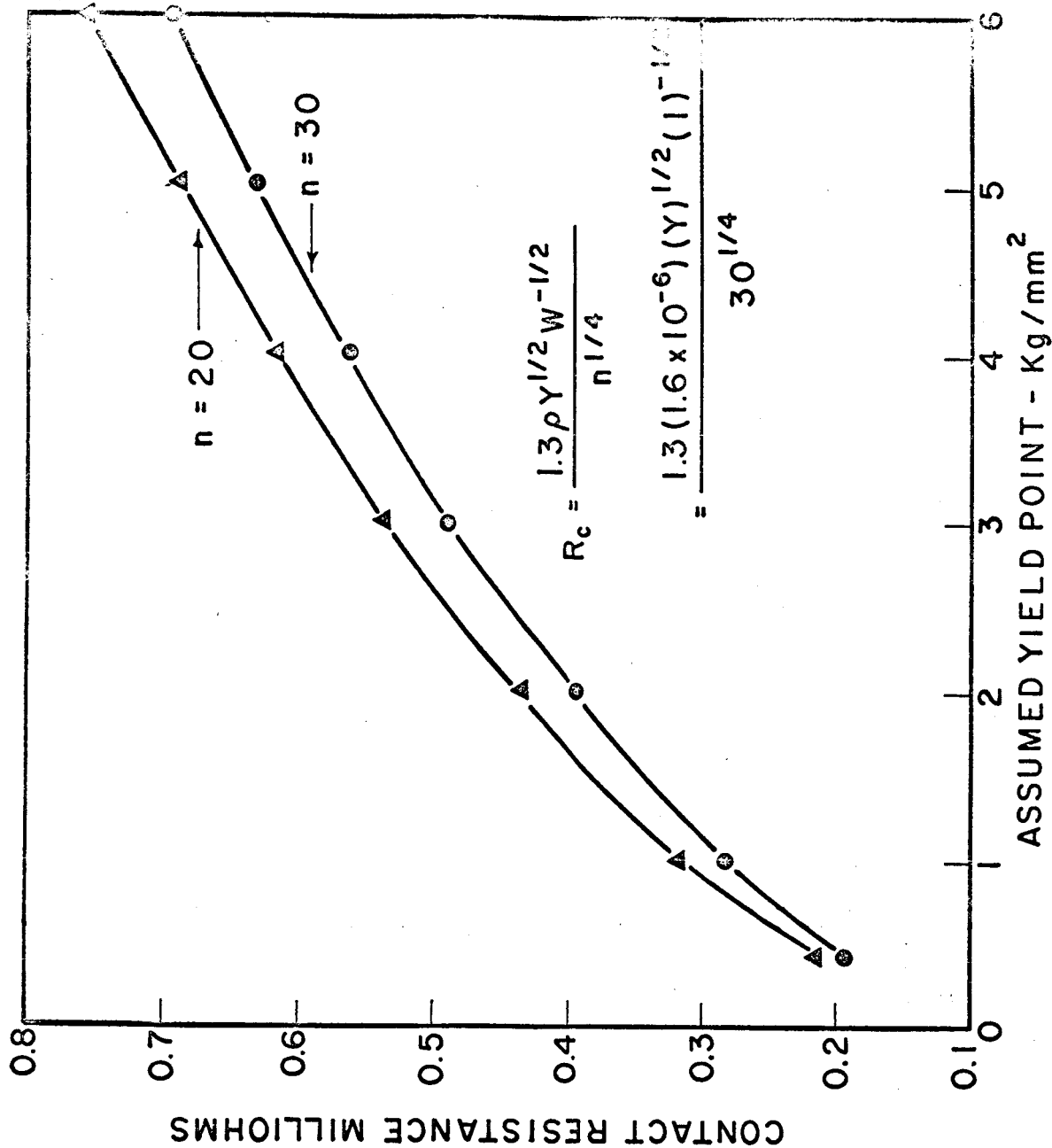


FIG. 3 THEORETICAL CURVES UNDER VARIOUS CONDITIONS OF MULTIPOINT CONTACT. CF. TEXT FOR EXPLANATION



VARIATION IN CONTACT RESISTANCE (R_c) OF SILVER WITH DIFFERENT ASSUMED YIELD POINTS (Y) AND DIFFERENT NUMBER OF CONTACT POINTS (n) AT ONE GRAM LOAD.

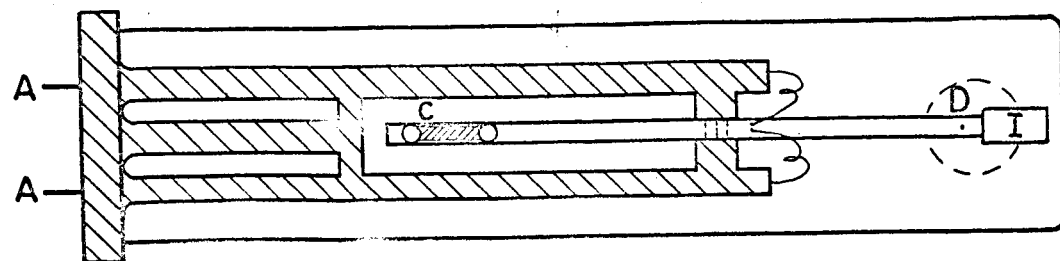
on an absolute scale, however, this may well cause a number of problems in interpretation.

The resistivity of the metal increases with the degree of work hardening, however, due to the low value of the resistivity per centimeter of dislocation line (2×10^{-19} ohm-cm) and the exceedingly small contact area the change would not be expected to be significant.

This point is emphasized by the examination of the contact resistance versus unloading behavior since during this phase the forces at the interface are changed, causing dislocation line movements; however, excessive contact resistivity changes are not evident as will be shown later.

In conclusion, therefore, during one particular experiment as the load between two contacting metal surfaces is increased in a continuous fashion, the relative contact resistance value would detect interfacial changes in contact radius equivalent to less than 0.05μ provided that we make the reasonable assumption that between a load of 1.01 grams and 1.02 grams the values of resistivity, yield point and the number of contact points do not change. Sufficient data has not, as yet, been accumulated to relate the absolute R_c to the absolute contact area. Precipitous changes in any of the variables would be readily detected as major discontinuations in observed curves. Such only occur at extremely light loads or under particular contaminated conditions.

Before considering the data obtained from the study of various metal systems and specific contaminants let us briefly examine the apparatus developed in this laboratory for experiments exploring these relationships. Figure 5



TOP VIEW - TORSION BEAM AND SUPPORT

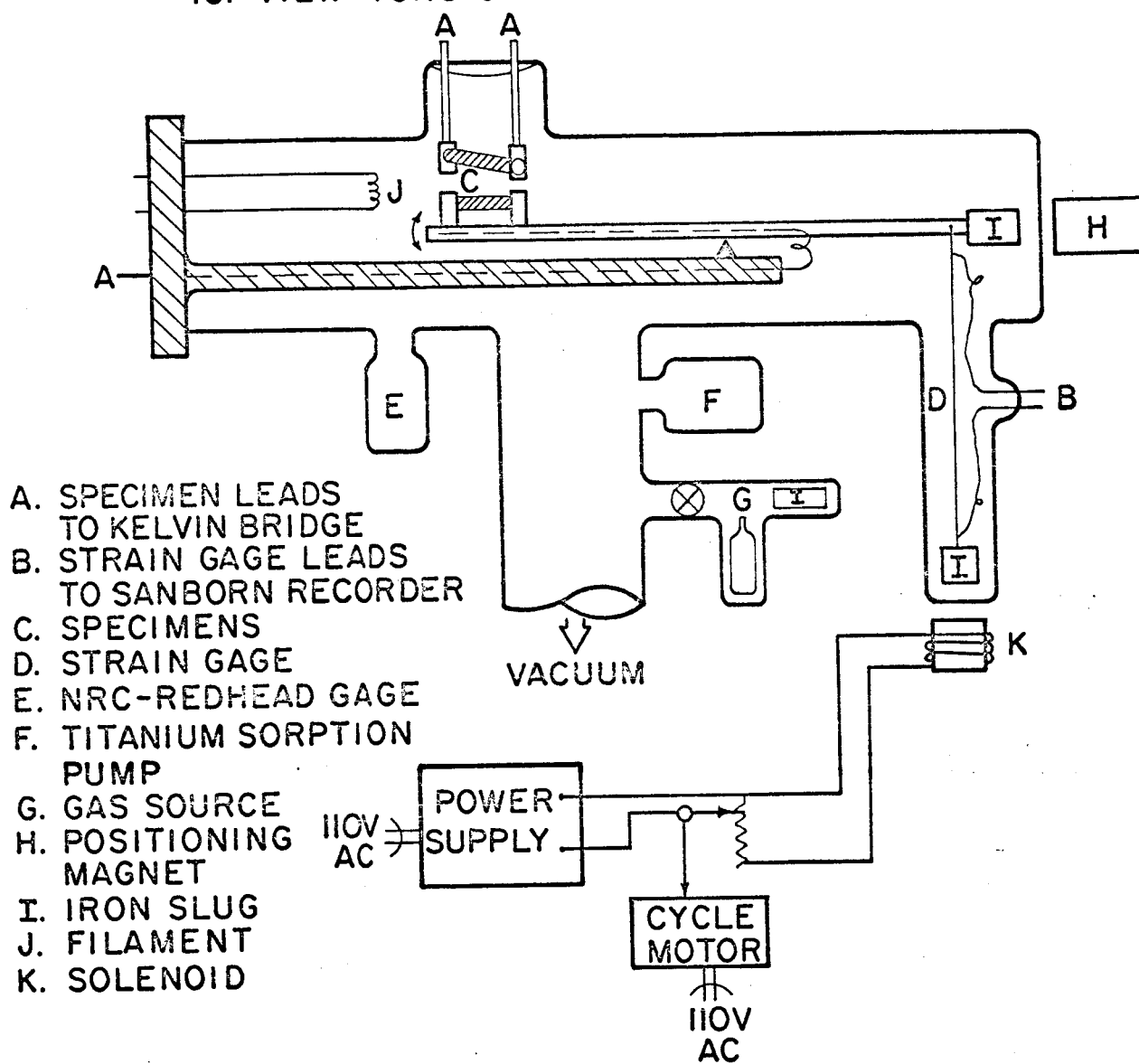


FIG.5 ADHESION CELL

shows the typical adhesion cell used for ultra high vacuum adhesion studies by automatically loading crossed wire samples and continually plotting on an X-Y chart the variation in contact resistance with the variation load.

The 50 mm O.D. x 300 mm pyrex adhesion cell shown in Figure 5 was affixed to the vacuum system isolation valve by means of a 40 mm pyrex-metal conflat flange, which also supported side-arms for the titanium sorption pump (F) and the argon gas supply (G). Upon thorough degassing and partial flashing of the wire immediately after bakeout, the small titanium pump could maintain the adhesion cell pressure below 10^{-9} Torr when the 1 inch isolation valve was closed to prevent oil contamination. Several 20 cc pyrex storage cells were filled with spectrographically pure argon or contaminant gases and isolated from the UHV system by glass break-off seals that could be broken with an iron slug when argon pressure was desired.

The pressure in the adhesion cell was measured by a Redhead Gage (NRC Type 752) (E) mounted on a 25 mm pyrex tube of low conductance in line of sight with the samples.

An alumina tube acted as a torsion balance beam and was supported by an aluminum bracket on a tungsten wire at the balance point. Two 5.0 mm stainless steel support rods were welded to the conflat flange to support the tungsten wire between them in a horizontal position. The conflat flange also supported two 12.5 mm. pyrex-kovar through-seals for filament leads and power leads to the iron sample (C) mounted on the beam. A magnetic rod was fixed to the torsion beam at the end opposite from the sample. This magnetic rod allowed the torsion beam to be moved and thereby position the sample on the beam relative to the fixed

sample. A maximum separation of about 30 mm could be achieved during argon ion bombardment. Also fixed at the end of the beam opposite the sample was an isolated support wire for the 150 mm nude 0.023 mm constantan strain gage wire (D). The lower end of the strain gage wire held a second magnetic rod (I), through which a load was applied to the samples by a solenoid (K) outside the system. The external leads to the strain gage entered through a side arm mounted glass-metal seal.

During a normal adhesion cycle the samples were brought into proximity by adjusting the external permanent magnet (H) relative to the magnetic rod on the end of the torsion beam such that about one mm separation remained between the two samples. In this configuration a load was applied to bring the samples into contact; at the end of the loading cycle this residual separation force was available to cause fracture if adhesion occurred. The load on the contacted samples was applied by varying the line input to an Electro DC power supply between zero and 110 volts, while the power supply output was set at a predetermined voltage corresponding to a solenoid field necessary to establish a desired peak load. This solenoid input power was varied by driving a variac with a synchronous motor which reversed at the 110 volt point and reduced the voltage linearly to zero. The load was applied and removed at a rate of 1.62 gms/min during a normal adhesion cycle.

The strain gage detector consisted of a Sanborn-Model 312 transducer amplifier indicator with the smallest division in this system corresponding to 0.02 gms. readable to within $\pm$ 0.010 gms. After each series of adhesion runs

the strain gage - mass relationship was calibrated through the 0-5 gm range of operation by replacing the fixed upper sample with a calibrated force transducer. The readings of the strain gage amplifier were then compared directly to a known load. This eliminated any question of variables, such as beam flexing or friction, which could arise if the gage were calibrated after removal from the system or by other indirect techniques. The range of sensitivity of the mass measurement was found to be ± 0.010 gm.

Numerous studies were made of the automatic loading profile during the standardization procedure, i. e. when the standardized force transducer replaced the fixed sample. This was accomplished by placing the output from the Sanborn 312 transducer amplifier as the input to the "y" function of an "x - y" recorder, and following the cyclic variation with time. The load was applied in a near perfect sawtooth curve with a slope of 1.42 gms/min. No significant variations from this shape were encountered.

The torsion beam arrangement was designed for pure normal loading. The object being to reduce shear deformation in the interface of the adhesion couple to a minimum during loading, since small tangential movement can rupture the contaminant films. The only tangential motion arose from very small, but unavoidable vibration. Under very light loading (< 30 mg) and non-adhesive conditions, these vibrations could be observed as an instability in the contact resistance reading.

Numerous adhesion cycles from zero load to peak load were made at peak loads between 0.03 gm. to 6.0 gms. in steps of about 0.03 gm for each change of surface state experienced by a sample. That is how the data for Figure 1 was

obtained as well as that for the other systems investigated to date, e.g. silver-silver, silver-tungsten, silver-nickel, copper-nickel, titanium-titanium, molybdenum-molybdenum, iron-iron (65 ppm carbon), and iron-iron (ultra pure).

Figure 6 illustrates two distinct types of curves which have been observed in all of these investigations and which act as one method of characterization of metallic adhesion and the effects of contaminant layers. Upon unloading the samples, if the contact resistance (e.g. related to the contact area) remains constant a significant junction strength has been established along the interface; that is, elastic compressive stresses accumulated in the contact zone can not return the interface to its original cylindrical shape. When contaminants are present, however, or a plane of weakness exists along the interface, the contact area per unit load is completely reversible such as shown in the upper curve demonstrating the effect of released elastic stresses. Actual data distinctively illustrate these facts and the possibility of some intermediate cases as shown in Figures 7 and 8. Figure 9 illustrates a direct reproduction of an X-Y plot of an adhesion cycle. Figures 10 and 11 are taken from the studies on Fe-Fe contaminated with 65 ppm carbon which illustrate the reproducibility of the experimental data. Each curve was transferred from a continuous X-Y recording of contact resistance versus load (as shown in Figure 9) by means of the data points shown. This is the only significance of these points. The observed contact resistance in Figures 10 and 11 is significant since if we apply the ultra pure iron data to our theoretical equation 4 we obtain a curve which is about two orders of magnitude lower in R_c than is shown. The surface cleaning treatment

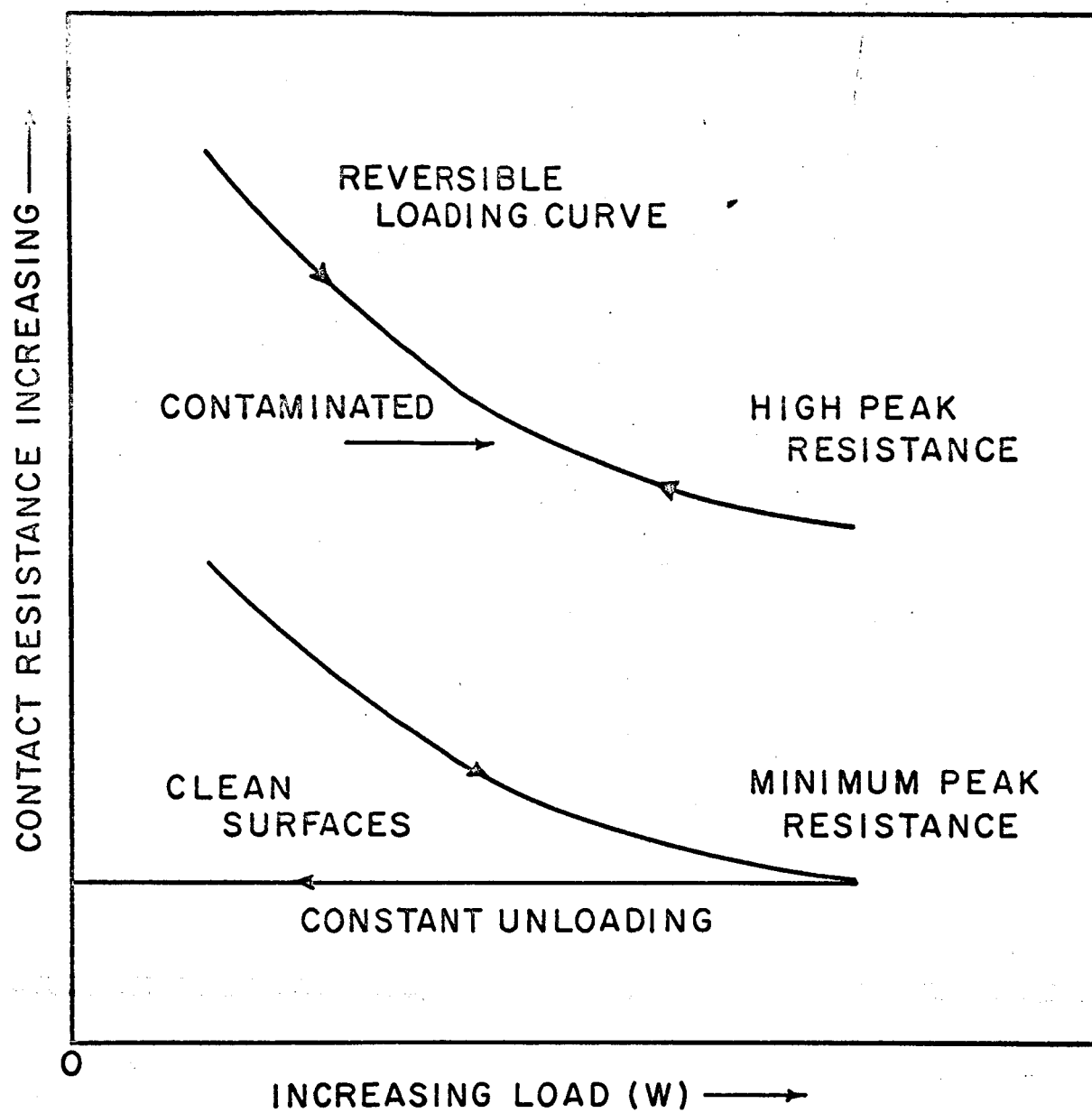


Figure 6 Characterization curves for two adhesion cycles, one contaminated and one clean

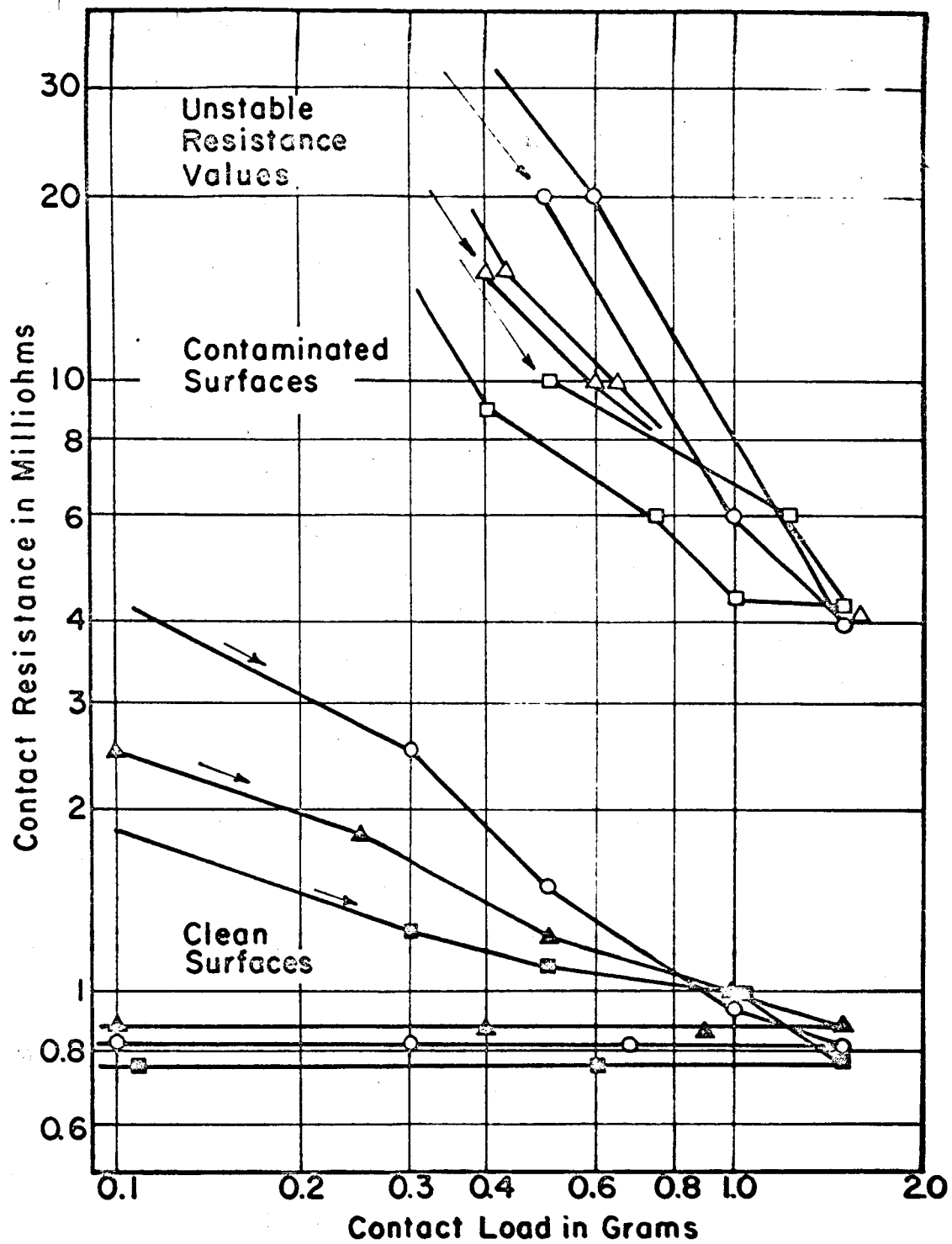


Figure 7. Contact Resistance versus load for clean and dirty silver-silver contacts.

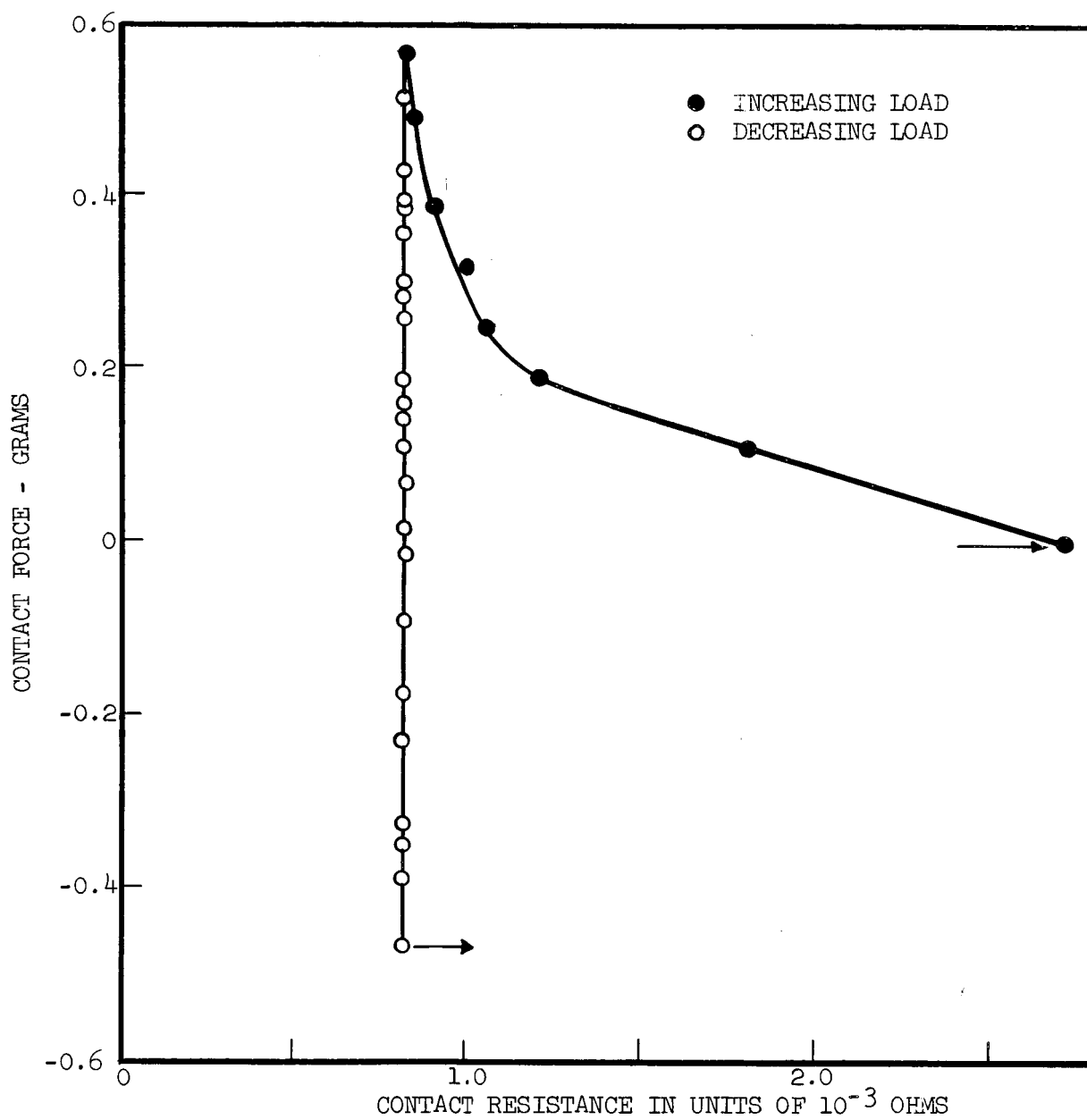


FIGURE 8 CONTACT RESISTANCE VERSUS LOAD FOR SILVER-SILVER COUPLE
COUPLE CLEAN CONDITION (LINEAR PLOT).

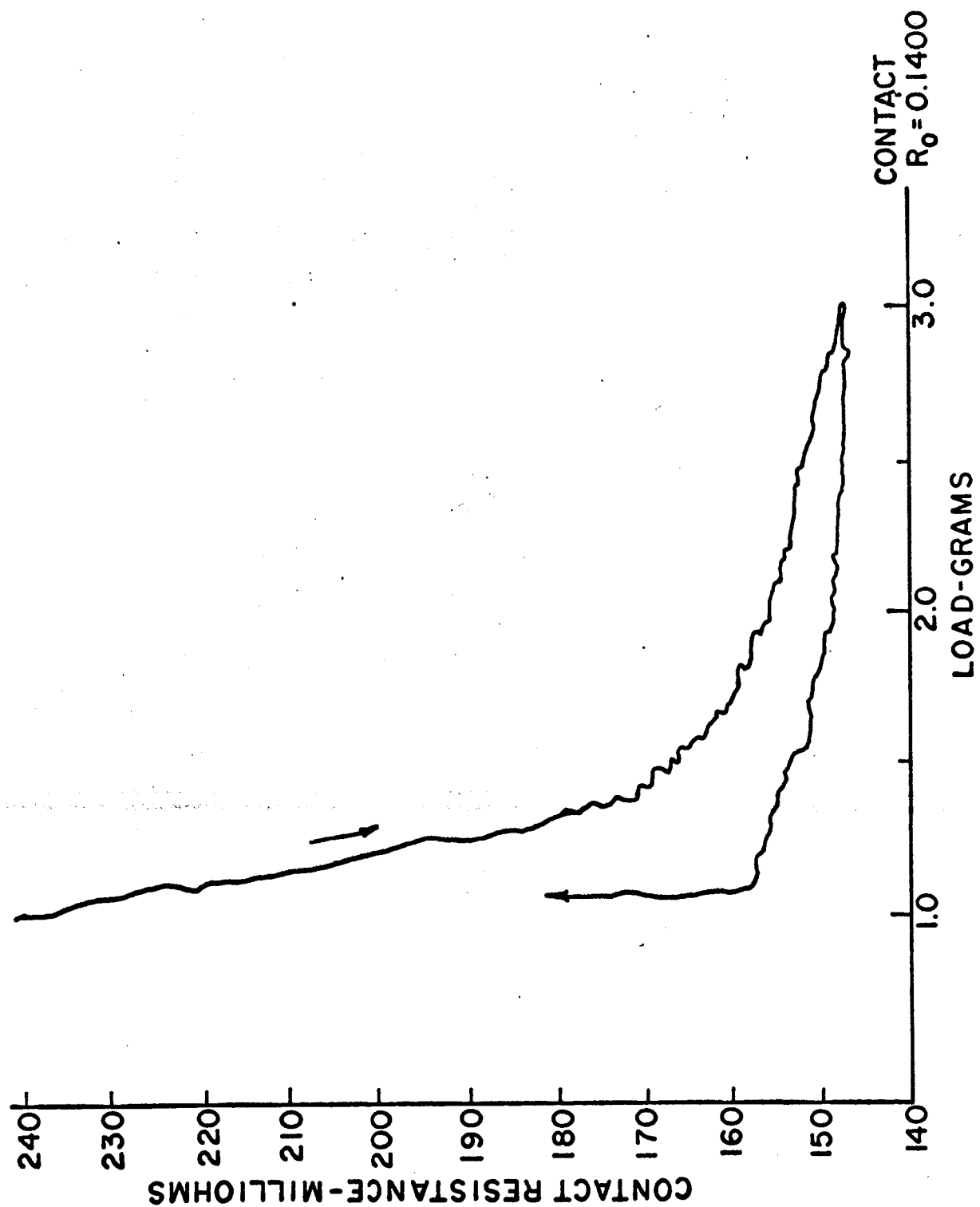


FIG. 9 TYPICAL "X-Y" PLOT FOR IRON - 655PPM CARBON ADHESION CYCLE

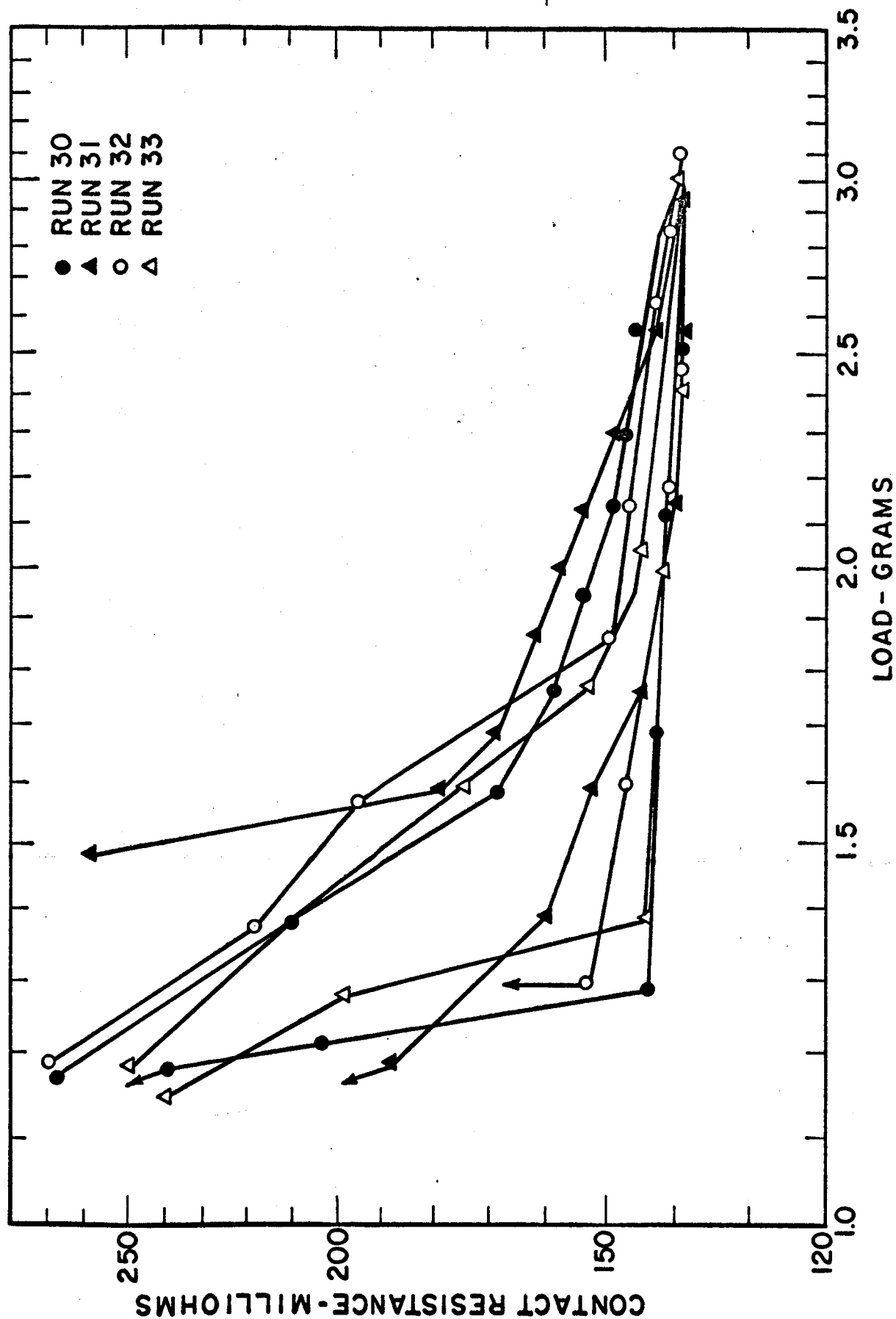


FIG. 10 TYPICAL ADHESION CYCLES BETWEEN IRON - 65 PPM CARBON COUPLES ON THE SAME CONTACT POINT

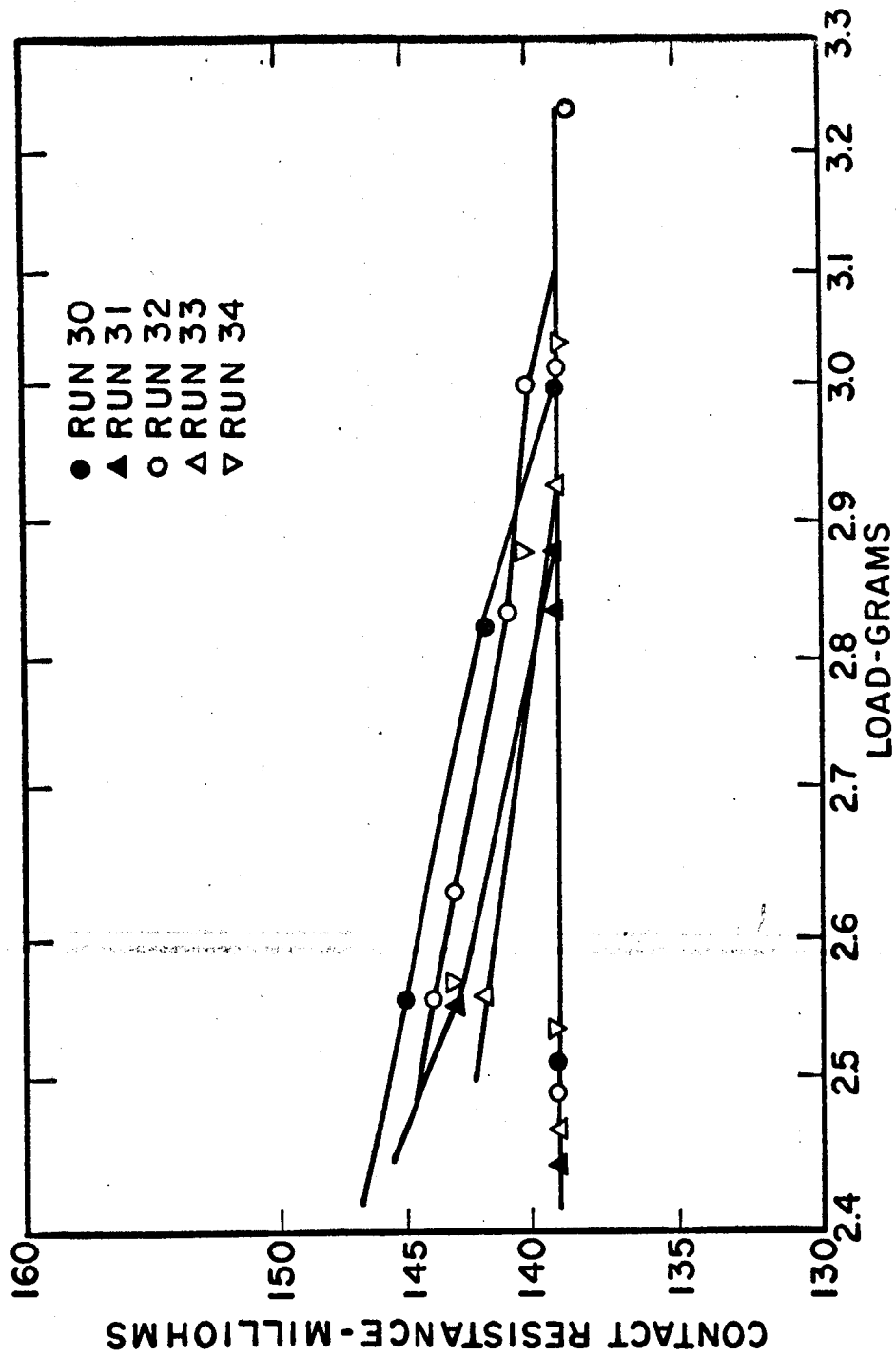


FIG. II EXPANDED SCALE OF FIG. 10

of the impure iron metal samples should, according to all previous work on metal samples of Ti-Ti, Mo-Mo, etc., produce clean surfaces providing rather stable adhesion junctions. Furthermore, numerous repeated cleaning cycles did not produce a lower contact resistance as was observed in the cases of Ti and Mo. Applying the known values for the resistivity ($9.7 \mu \Omega \text{ cm}$) and yield point (2.5 Kg/mm^2) of pure iron to the theoretical equation and assuming a probable number of contact change (cf. Figure 3), the lower half of Figure 12 was developed. Curve 30 from Figure 10 is shown relative to a second theoretical curve (A'F'D') displaced two orders of magnitude higher. It was assumed at this time that surface contamination by bulk diffusion to the surface layers caused this discrepancy. For example, if carbon accumulated in the surficial layers to a concentration near that of normal cast iron the resistivity would be about an order of magnitude higher as would that of the yield point of the surficial system. Dissolved oxygen or nitrogen might also cause the same phenomena. In any event, the data from run 30 etc., indicated that a surface contaminant was present and pure iron was not involved. The interface did not indicate bulk strength adhesion, a further case for contamination. Recently during the investigation of ultra pure iron these observations were confirmed as were the general predictions of contamination and contact resistance. Several data points from ultra pure iron are compared to the theoretical curve of pure iron in Figure 13. The pure iron system with clean surfaces was contaminated with oxygen at 1×10^{-5} Torr and at room temperature for less than ten minutes and the contact resistance rose from 6 milliohms to greater than an ohm under a one gram load.

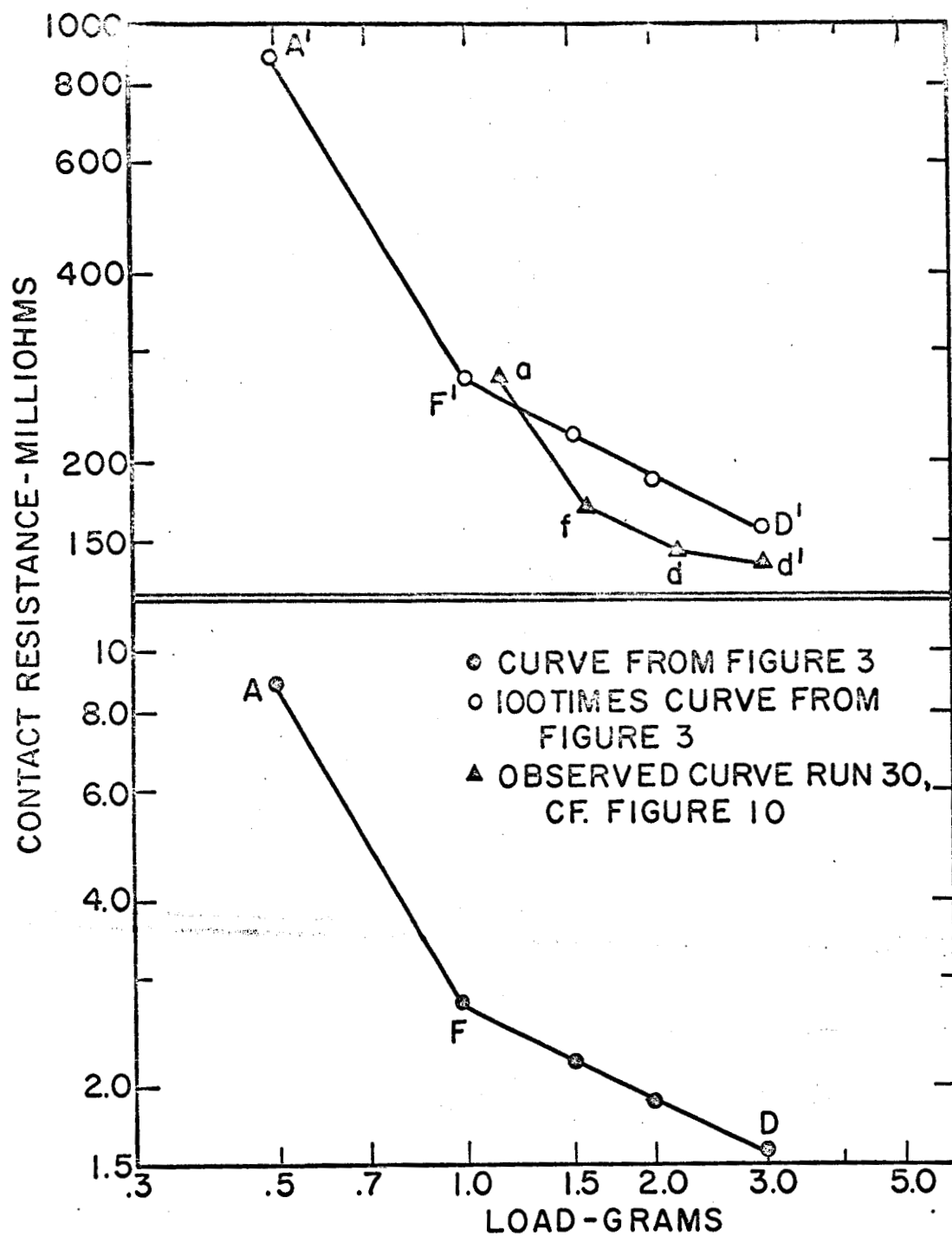


Figure 12 . Comparison of Theoretical Ultra pure iron curve AFD with Run 30 afdd'. Curve A'F'D' is curve AFD displaced two orders of magnitude.

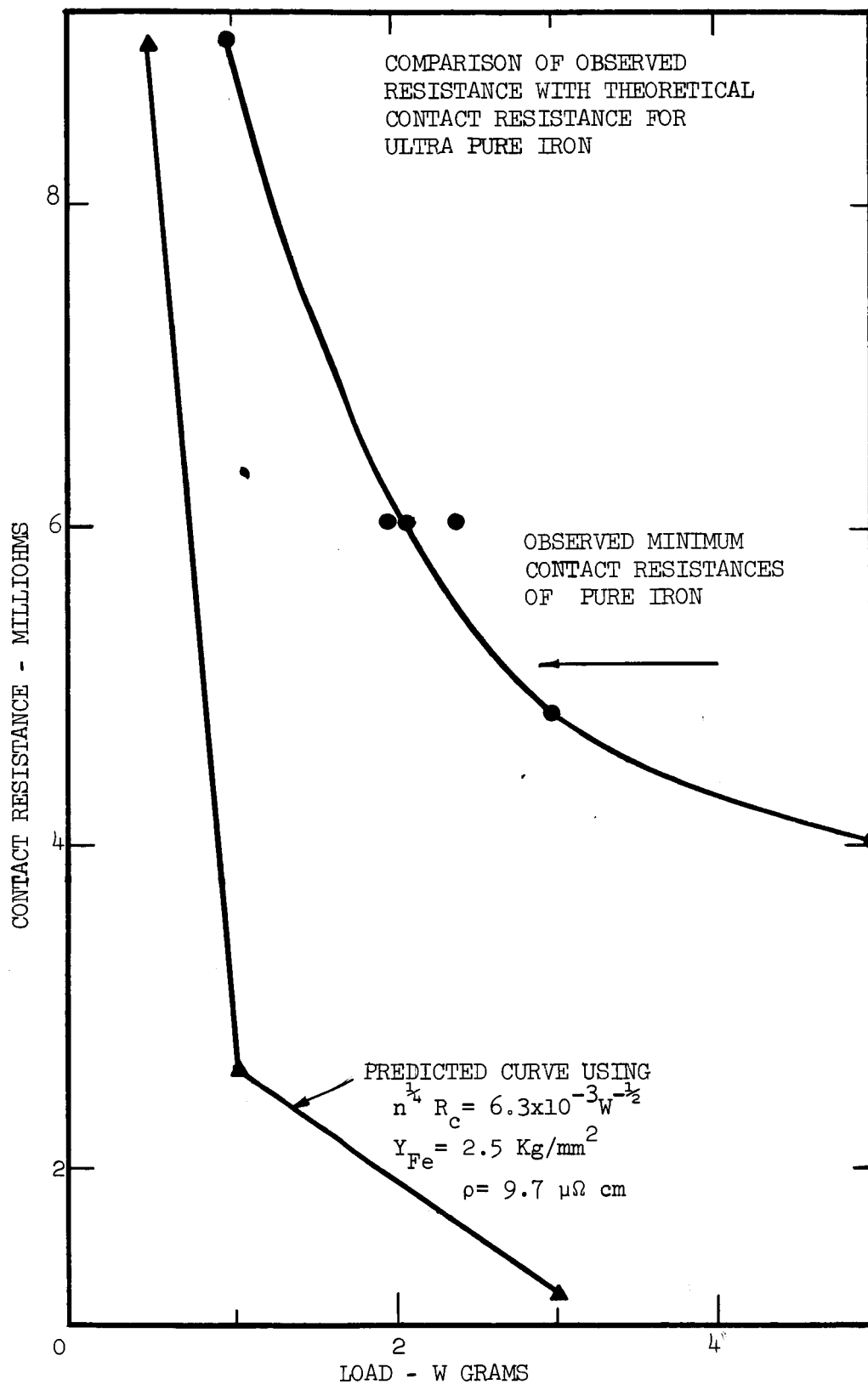


FIGURE 13 THEORETICAL CURVE FOR ULTRA PURE IRON (PREDICTED)
COMPARED TO OBSERVED VALUES FROM ULTRA PURE
IRON DATA

A number of further significant features ought to be considered in the characteristic shape of the runs 30 - 33 (Figure 10). The deformation process at light loads seems to have a slope (log-log plot) in the range of -2 which changes to about -0.4 as the load is increased to about 1.6 - 1.7 gms. At some point greater than 2.5 gms the slope again changes to a value of -0.15 or less. The degree of scatter for about 50 cycles can be shown approximately

Stage I -2 ± 0.5 slope

Stage II -0.4 ± 0.1

Stage III 0.15 ± 0.05

During unloading, R_0 remains constant to a point of about 1.3 grams contact force, at this load the stable junction disintegrates to a contact resistance value approximating the loading value of R_0 as measured during the loading cycle. This aspect will be discussed later based on the load deformation mechanisms.

A comparison of the theoretical curve shown in Figure 3 is made with run 30 from Figure 10 in Figure 12, where afdd' is a reproduction of the observed run 30. Also plotted on Figure 12 is a curve A'F'D' which represents a two order of magnitude displacement of curve A-F-D to a higher resistance. For now, this displacement can be considered as simply the change in bulk resistivity of the iron concerned from $9.7 \mu\Omega\text{-cm}$ to $97 \mu\Omega\text{-cm}$ and a change in yield point of from 2.5 Kg/mm^2 (3.5 Ksi) to 10.0 Kg/mm^2 (14. Ksi) which will account for the two orders of magnitude. Since the shift in position of A-F-D to A'F'D' does not effect the slope of the theoretical curves, let us compare the slopes with the observed curve afdd'.

The slope of the theoretical curve between A-F is approximately -2 or that of the observed data between a-f; furthermore, very high slopes were observed under very light loads for nearly all of the 150 runs conducted in this particular investigation. Since the change from a few asperity contacts (A') to many (F') was arbitrarily chosen as 0.5 gms. and 1.0 gms, this could also have been equally well chosen to coincide with the observed curve (a-f). The slope between fd on the observed curve lies in the range -0.4 which is somewhat less than predicted in F'D' (-0.5). From the previous equations and the discussion of Figure 12, it is evident that if the number of asperities in contact were increasing, the deformation slope will also increase in a similar continuous manner. Since the observed slope (f-d) is less than that calculated at -0.5 and is linear, the difference cannot be attributed to a continuous change in the number of contacting asperities. Since resistivity does not vary significantly with pressure, the variation between f and d might be accounted for by breakthroughs in a contaminant layer; however, such breakthroughs would abruptly decrease the contact resistance also causing non-linear increase in slope, again not in accord with that observed. Variation in the yield point could shift the curve in the proper direction; however, one would again expect a loss in linearity which is not observed.

The three most likely explanations for the smaller slope are:

- a. That the deformation process of the asperities is a mixture of elastic (slope -0.33) and plastic (slope -0.50) processes.
- b. That surface creep is superimposed on the deformation curve.
- c. Surface contaminants have modified the overall deformation process.

Creep was observed to expand the contact area over a significant period of

time, as is reported later.

The small slope (-0.15) observed between d and d' is also a characteristic observed in Figure 10. Stage III may signify the beginning of bulk phenomena, i.e. where the interaction between asperities has ceased except for creep and bulk elastic support of the load has ensued. It is interesting to note that the slope of the creep curves varies between 0.05 and 0.18 depending on the work hardening of the area tested. The slope of the last portion of the adhesion curve (Figure 10) also varies between 0.05 and 0.15 .

Although the prime purpose of these investigations was not intended to include the process of creep in the formation of an interface, preliminary studies were initiated. A significant amount of creep was observed in the loaded interface at room temperature, e.g. $0.17 T_{mp}$ between iron-iron 65 ppm carbon couples. This process corresponds to what has been described as "junction growth" of two contacting surfaces subjected to a load for a period of time.

The creep process also lends credence to the proposed model of rough surface contact phenomena, i.e. plastic deformation of asperities as micro-deformation and elastic or plastic macro-deformation. Creep had not been considered previously for two bodies in elastic (macro) contact since it was thought that the plasticity of the material must be involved in the creep process. We can now consider the creep process of the asperities which were plastically deformed, even though the bulk elastic point had not been exceeded.

A brief analysis of this process will illustrate the possibilities of a study of creep in a more detailed manner. Consider a relationship similar to that

developed in equation 4. If the true area (A_n) is studied as a function of time, Tabor (16) has shown that

$$A_n = \frac{W}{\bar{P}} \quad \text{Equation 12}$$

where W is the load and P is the yield pressure in hardness studies.

Furthermore, P is related to time (t) by

$$P = A_1 t^{-1/m}$$

where

$$A_1 = A_5^{-1/m} \exp(-Q/RT)^{-1/m}$$

A_5 = system constant

Q = Activation energy of creep

R = Universal constant

T = Absolute temperature

M = Mechanical deformation constant

by substitution

$$A_n = W/A_1 t^{-1/m} \quad \text{Equation 13}$$

By proceeding as illustrated in the development of equation 4 and making use of the time dependent equation we arrive at a relationship between R_c and t .

$$R_c = A_2 t^{-1/2m} \quad \text{Equation 14}$$

where

$$A_2 = \frac{\pi \rho^2 W A_1}{5.6n^{1/4}} = \text{constant}$$

= Bulk resistivity

n = Number of contact points

W = Load

By choosing the load in the creep experiment to exceed the load (1.3 gm) where the number of contact points become constant, time becomes the only major variable in the expression. The observed slopes of numerous creep curves range from -0.2 for the new contact points and -0.05 for the multi-contact points. This suggests that the value of m must vary between 2.5 for the ductile deformation process and 10 for the work hardened process. These values are consistent with values suggested by Tabor.

The creep investigations were not intended to be exhaustive; and therefore, the only valid conclusion that can be drawn from the data and correlations is that a technique has been developed which shows much promise for the study of interfacial deformation phenomena. The technique could also possibly develop the mechanism of contaminant layer - real surface deformation processes, as well as the activation energy for creep in the surface layers of various states of contamination.

In conclusion the evidence presented certainly demonstrates that a technique has been devised which aids in the characterization of surficial phenomena including deformation processes, adhesion and adhesion junction fracture. The analysis of the observed data strongly indicate that contact resistance measurements can be considered in detail in a manner relative in one adhesion cycle but a great deal of caution must be used to extrapolate these analyses to absolute values even though such an extrapolation would be most desirable. Certainly more intensive studies would supply the security necessary to make these extrapolations and provide even more details of surficial behavior. For example, the solution of the creep problem in the contact zone at zero load is generally

recognized as "neck growth" in powder compacting problems and has never been examined under conditions of limited or controlled contamination. Surface diffusion is the fundamental parameter which is involved in this process. Certainly other processes will become involved as the exploration of this research area expands.

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