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EXPERIMENTAL INVESTIGATION OF ULTRA-HIGH VACUUM ADHESION AS RELATED TO THE LUNAR SURFACE

EIGHTH QUARTERLY PROGRESS REPORT AND 2ND YEAR SUMMARY

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ABSTRACT

This report presents the results obtained during the past year on a study of the ultra-high vacuum adhesion of silicates as related to the lunar surface. Silicates, such as may exist at the lunar surface, were contacted with silicates and engineering materials which may be used at the lunar surface. The silicate contacting surfaces have, in some cases, been formed in air; in other cases, they were formed at ultra-high vacuum (10^{-10}mm Hg) by cleavage. The engineering material surfaces were, in all cases, formed in air. Adhesion force was measured as a function of load force, crystalline orientation, surface roughness, and type of forepump used.

Two types of adhesional behavior were found for the air-formed surfaces. The first appeared only under load, increasing rapidly with increasing load; was of relatively large magnitude (up to about 0.4 gm); and was present only at ultra-high vacuum. In addition, when this type of adhesion was present, extensive surface damage and material transfer were noted. It is concluded that this type of behavior is caused by the action of the normal silicate atomic bonding forces. The second type was present at zero load, showed little load dependence, was of relatively low magnitude, persisted in dry nitrogen (at atmospheric pressure), and did not produce surface damage or material transfer. It is concluded that this type of behavior is most probably caused by the action of the dispersion forces.

For the vacuum-cleaved samples, the adhesion was found to be much larger than that observed for the air-formed samples. In addition, a strong, long-range attractive force, indicative of considerable surface charging, was noted. The results indicate that the primary contributor to the observed adhesion is the normal silicate bonding forces, and also that the long range force is caused by a statistical charge separation produced during cleavage.

The results obtained indicate that silicate adhesion can be the major factor in determining the behavior of lunar materials and that its precise role is critically dependent upon the nature of the contacting surfaces.

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1.0 INTRODUCTION

1.1 General

This report presents a summary of work accomplished during the period July 1, 1965, through July 1, 1966 (second year of the program), on the study of the ultra-high vacuum frictional-adhesional behavior of silicates as related to the lunar surface. This work is being conducted for the Office of Advanced Research and Technology, National Aeronautics and Space Administration, under contract NAS7-307.

1.2 Purpose and Importance of the Program

The primary purpose of this program is to obtain quantitative experimental data concerning the ultra-high vacuum adhesional-frictional behavior of the materials which may presently exist at the lunar surface (believed to be primarily silicates), and to obtain similar data for behavior between those lunar surface materials and other engineering materials which may be placed upon the surface. Additional purposes of this program are to analyze these data with regard to the possible reactions of granular lunar materials to engineering operations, and to investigate means by which any problems posed by these reactions may be minimized.

The importance of this program is that adhesional-frictional phenomena may pose serious problems to lunar surface operations.

1.3 Approach

The approach used during the first year of this study was to obtain quantitative data relating to the adhesion force as a function of load force, temperature, type of silicate, and crystalline orientation. All surfaces to be contacted were formed in air.

During this second year of the study, the following approach has been used:

1) obtain data, in the manner of the first year's program, on additional materials;

2) investigate the effects of forepump type and surface roughness on the adhesion of air-formed surfaces;
3) investigate, further, orientation effects for given crystal faces in contact;
4) check data reproducibility;
5) study the adhesion between vacuum formed surfaces in order to place possible bounds on the range of adhesional phenomena which may occur at the lunar surface.

2.0 NATURE OF SILICATE SURFACES AND ADHESION

The physico-chemical nature of silicate surfaces has been considered by a number of investigators. Most of this work has been summarized by Eitel (1964). Studies of particular interest are those by Weyl (1955) and De Vore (1963). Weyl has approached the problem from the classical electrostatic and polarization viewpoint, rather than from the quantum mechanical homopolar-bond-resonance viewpoint. Although the validity, or desirability, of approaching the problem from an essentially classical basis can be questioned, Weyl's treatment has been successful in providing reasonable explanations for a number of phenomena associated with silicate surfaces, and, hence, it appears that his approach is, at present, as good as any for use as a starting point in the understanding of silicate adhesion.

A freshly produced silicate surface must adjust to the resulting changed conditions, these changed conditions being the unsatisfied charge and coordinations produced, and exposure to the external environment. According to Weyl, there are, in general, three possible ways in which a solid surface can adjust: through polarization of surface ions, through distortion of surface structure with formation of an electric double layer, and through adsorption of materials from the surrounding environment. For silicates, the first possibility can be disregarded because of the low polarizability of the Si^{4+} (also Al^{3+}) ions. Obviously, if the new surfaces are formed in a perfect vacuum, distortion alone is possible. Surface distortion, according to Weyl, is caused by the necessity for cation screening. Hence, the anions (0^{2-}) tend to displace toward, and the cations away from, the surface. This forms oriented dipoles (electric double layer) with the negative parts at the surface, and reduces the role of the cations in surface phenomena.

De Vore (1963) has considered further the changes occurring to a freshly produced silicate surface. He notes that in almost all cases, Si-0 bonds, as well as metal-oxygen bonds, must be broken. If surface formation occurs in air, the exposed silicon (or metal) ions will satisfy their charge and coordination demands by attaching oxygen, while the exposed oxygens (initial and attached) will satisfy their demands through the addition of water (as H^+ to form hydroxyl groups and as adsorbed polar molecules). Hence, essentially

all silicate surfaces formed in air can be considered to be hydrated. For a surface formed in vacuum, the charge and coordination demands remain unsatisfied, and even though surface distortion occurs in an attempt to improve cation screening, the surface will remain highly reactive.

There are a number of processes (or forces) which may contribute to solid-solid silicate adhesion in vacuum. These are the action of the normal silicate lattice bonding forces (ionic-covalent), the action of dispersion forces (London--Van der Waals), surface electrostatic charging, and the action of adsorbed surface films (including hydrogen bonding).

The silicate bonding forces are highly directional and their range of effectiveness is the shortest of all forces which could act. Hence, their effectiveness should be highly sensitive to the atomic structures of the contacting surfaces, to the degree of surface contamination present, and to the degree to which charge and coordination demands are unsatisfied. These forces are, in general, the only ones of sufficient strength to produce surface disruption.

The dispersion (London--Van der Waals) forces can also contribute significantly to silicate adhesion, as evidenced by the work of Bradley (1932), Lowe and Lucas (1953), Jordan (1954), and Derjaguin et al. (1954). These forces have a range of effectiveness much greater than that of the ionic-covalent forces, but less than that for the forces produced by electrostatic surface charging. Also, unlike the ionic-covalent bonds, these forces are essentially nondirectional. The force-distance relationships for these dispersion forces have been calculated theoretically, and verified experimentally for surface separations greater than about a tenth of a micron. Harper (1955) has shown that contact of quartz surfaces can produce a surface electrostatic charge. In general, this charge produces long-range forces, the longest range of any which may act. This phenomenon is herein called "homogeneous" surface charging, as it was by Overbeek and Sparnaay (1954). Another type of surface charging denoted as "mosaic charging" has been postulated by Derjaguin et al. (1954) to explain the anomalously high attractive forces detected by Overbeek and Sparnaay. According to Derjaguin et al., because no surface is perfect with respect to atomic arrangement, lack of localized impurities, etc., a mosaic distribution of charges (of opposite signs) could

be generated, with the net surface charge remaining zero or near zero. If a certain amount of surface mobility of these charges is allowed, then forces could act between two dielectrics brought into close proximity or contact. Because of the mosaic distribution of these charges, the range of effectiveness of the forces produced would be much less than those produced by homogeneous surface charging, but greater than the range of effectiveness of the ionic-covalent and dispersion forces.

The action of adsorbed surface films, particularly H_2O (OH), in producing solid-solid adhesion has been known for some time, a considerable amount of work having been done on this problem in the field of clay mineralogy. Grim (1953) notes that chemisorbed hydrogen (OH) bonding between the silicate units is a major contributor to the strength of clays, and that adsorbed water also can contribute. As far as vacuum adhesion is concerned, however, the adsorbed water can be removed by heating to 100-150°C, possibly also by exposure to the vacuum alone. However, the chemisorbed hydrogen (OH) requires bakeout at least to 400°C, and possibly to 700°C. Hence, for surfaces produced in air, this remains a possible adhesion producing mechanism in vacuum.

Hydrogen bonding, which in certain aspects is similar to that found in clays, is also postulated to cause adhesion through surface charging. According to Weyl's views (see Gruver, 1956), transfer of hydrogen ions (such as will become attached to silicate surfaces formed in air) can occur between contacting surfaces if the degree of cation screening of the two surfaces is dissimilar, the hydrogen ions moving from the surface having the more poorly screened cations. This will result in the formation of an electric potential between the surfaces and, hence, cause adhesion. This is a possible adhesion producing mechanism in vacuum, provided surface cleaning techniques are insufficient to remove hydrated surface layers. It should be noted that this charging mechanism should not act, however, for similar materials in contact.

Since a number of mechanisms could act to produce adhesion between contacting silicate surfaces in vacuum, the problem of understanding the nature of the adhesion becomes somewhat complicated. Additional complications arise from present uncertainties regarding the nature of silicate surfaces, the specification of what a "clean" silicate surface is and when it is "clean", and the determination of the proper method for removal of surface contamination.

3.0 LUNAR SURFACE ADHESION

The exact nature of the surfaces of lunar materials is not known. However, reasonable bounds can be placed upon their nature as pertains to the resultant adhesional behavior. The lower bound would be for surfaces whose charge and coordination demands are satisfied, and which have some degree of adsorbed material present. The upper bound would be for surfaces whose charge and coordination demands are unsatisfied (actually, this is probably a minimum upper bound since time in contact effects, on the lunar scale, have not as yet been considered).

If, during the formation of a fresh surface on the moon, an atmosphere were present, either as part of a general lunar atmosphere or as a transient phenomenon generated by the mechanism causing fresh surface production, then the charge and coordination demands could be satisfied, and some degree of surface contamination could persist. Additionally, even if a significant atmosphere is not present at generation, it is conceivable that the surface demands could be satisfied over a period of time, either by the remnant lunar atmosphere, or by de-gassing from the lunar interior. Such surfaces could exist below the lunar surface, but, if the solar wind strikes the lunar surface, it is unlikely that they could exist at the surface.

On the other hand, if a fresh surface is produced in the absence of an atmosphere, the charge and coordination demands can remain unsatisfied. Alternatively, a contaminated surface exposed to the solar wind can be "cleaned" to the extent that its demands are no longer satisfied. Such surfaces can exist at, and below, the lunar surface, being produced through the action of the solar wind and micrometeorite impact. An additional future production mechanism would be through the operations of man (drilling, coring, sample taking, experiment implacement, locomotion, etc.).

The studies conducted to date have involved measurement between surfaces formed in air, and between surfaces formed in vacuum. The air-formed surfaces initially have their charge and coordination demands satisfied. Exposure to ultra-high vacuum suffices to remove gross surface contamination, but it

is likely that the surface demands remain to a large degree satisfied. These studies, hence, are representative of the possible lower bound lunar adhesion case. The vacuum formed (cleaved) surfaces represent, on the other hand, a possible upper bound for lunar adhesion (excluding time in contact effects). For these surfaces, the charge and coordination demands are initially unsatisfied, and no contamination is present.

4.0 INSTRUMENTATION

4.1 Experimental System in Use at Start of Second Year

Details of the basic experimental apparatus used during the first year of this study, and during the early phases of the second year, have been given by Ryan (1965). Briefly, the vacuum system consisted of a mechanical forepump with liquid nitrogen cold trap, a 200 l sec^{-1} ion pump, and the experimental chamber. Pressure was monitored by a "nude" Bayard-Alpert ionization gage which was cross-checked against pump current.

The samples consisted of disks with the flat faces (≈ 0.4 cm^2) being contacted. A steel bucket was suspended from the upper sample to allow load force to be applied to the contacting surfaces. Load force, up to 1000 gms., was applied by bringing an electromagnet up from outside the system and passing current through it. Adhesion force, after withdrawal of the electromagnet, was measured by means of a torsion microbalance. With this microbalance, forces as small as about 20μg could be detected.

4.2 Major Experimental System Modifications During Second Year

4.2.1 Replacement of Mechanical Pump

All data from the first year of this study were obtained with a trapped mechanical pump as the forepump. Great care was used to ensure that no oil from the mechanical pump back-streamed into the ultra-high vacuum part of the system, since oil on the sample surfaces could affect the adhesion (in particular, small amounts of oil are generally believed capable of "killing" adhesion, whereas large amounts, if the surfaces are sufficiently flat, can possibly cause adhesion. The particular precautions used were to 1) insert a liquid nitrogen cold trap-baffle between the forepump and the UHV system, 2) keep the samples in contact during the roughing cycle (lasting about ten minutes), 3) degrease the low vacuum part of the system prior to every pump-down, and 4) conduct all system bakeouts with the valve separating the low and UHV parts of the system in the closed position. These precautions appeared to achieve the desired objective. The evidence for this was first,

the variations in adhesional behavior obtained between the various sample pairs; second, the lack of detectable adhesion during pumpdown and the disappearance of high magnitude adhesion at moderate vacuum, nitrogen, and air after UHV had been obtained; third, the negative results obtained from attempts to detect hydrocarbons on the sample surfaces by means of infrared techniques (these studies placed an upper limit of ≈1000Å for film thickness); fourth, the lack of detectable oil deposits in the UHV chamber after many pumpdown cycles; and finally, the presence of adhesion after sample bakeout to temperatures in excess of 500°C. Though the evidence obtained weighed heavily against oil contamination, it was decided to replace the mechanical pump by a sorption pump bank.

The bank consists of three standard sorption pumps with associated heaters and liquid nitrogen dewars, a Pirani gage for pressure monitoring, three valves for individual pump control, and a fourth valve for admittance of dry nitrogen to the system. The manifold for the system was constructed of 304 stainless steel, and the entire assembly was mounted on a cart permitting the assembly to be detached from the ultra-high vacuum part of the system during the experiment. With these sorption pumps, it was found that the system could be pumped from atmospheric pressure to ion pump start pressure in about 30 minutes.

4.2.2 Experimental Chamber Changes For Study of Adhesion Between Air-Formed Surfaces

The experimental chamber used during the first year of the study was, during this second year, considerably modified. The primary purpose of these changes was to permit utilization of an electron gun for surface cleaning. The basic modifications were to (1) expand the vertical height of the chamber and add additional bellows so that the samples could be separated sufficiently for the electron gun to be inserted between them, and (2) provide additional ports for the electron gun. Other modifications were (1) the addition of more view ports to aid in the observation of the samples and the microbalance, (2) the improvement of the micrometer screws attached to the tilt stage to reduce the problems associated with obtaining sample parallelism, and (3) polishing the interior chamber walls to aid in obtaining better vacuum.

This new chamber has operated successfully during this year. With it, lower base pressures have been obtained (8×10^{-11} - 2×10^{-10} mm Hg) than were obtained with the previous chamber.

4.2.3 Cleavage Device

Three modifications to the experimental system were made in order to begin vacuum cleavage studies. These were (1) installing a cleavage device, (2) providing sufficient support for the sample to be cleaved to prevent unwanted breakage during the cleavage process, and (3) permitting the microbalance to be zeroed even if initially greatly out of balance after the cleavage. The cleavage device consisted basically of a tool steel chisel and a sample holder. The purpose of the chisel was to produce the cleavage. It is wedge-shaped and formed to fit into a pre-cut small slot in the sample. The chisel was mounted on bellows so that it could be brought into contact with the sample for cleavage and then withdrawn prior to measurement of adhesion. Cleavage was produced by impacting the chisel from outside the chamber. The purpose of the sample holder was to hold the sample rigidly during cleavage. It was designed to fit around the metal bucket (used to apply load force) so that the load force dependence of the adhesion could be measured. The holder could be removed (by means of bellows) from the vicinity of the sample after cleavage was accomplished.

Since it was not possible to anticipate the exact weight of the upper sample after cleavage, it was necessary to replace the microbalance counterweight with a chain loop, one end of this loop being held to the chamber wall by means of a magnet located outside the system. By moving the magnet up or down, it was then possible to zero the microbalance even when it was initially considerably out of balance.

It was found, from air tests, that during cleavage the sample also tended to break near its base due to a zone of weakness in the region of the slug and crosspin holes. Accordingly, a metal sleeve was inserted around the sample in this region.

These modifications did not prove to be sufficient to insure a high reliability for the success of the experiment. The principal problems encountered were:

1) the adhesion force exceeded the capacity of the microbalance
2) the metal bucket tended to be dislodged from the sample during cleavage
3) the metal sleeve inserted around the sample at its base to prevent fracture tended to slip out of place during system bakeout
4) the control mechanisms for the placement of the cleavage chisel and sample holder were not sufficiently sensitive, hence, a number of samples were lost during placement, by fracture.

Accordingly, the following modifications were made during the fourth quarter:

1) The microbalance was replaced by a precision mechanical spring, spring extension being measured with a cathetometer. This allowed adhesion forces as small as a few tenths of a gram, and as large as fifty grams, to be measured
2) the metal bucket was replaced by a metal slug attached rigidly to the top of the sample. With this slug, the maximum load force which could be applied was about 200 gm.
3) the sample forming procedure was altered so that the diameter of the sample base was greater than that of the top. The sample was then inserted into a tapered hole in a large copper plate.
4) a new chisel and holder were fabricated, incorporating much more sensitive means for implacement.

5.0 EXPERIMENTAL DATA

5.1 Adhesion Between Air-Formed Surfaces

The experimental conditions under which the data were obtained are given in Table I. Included in this table, for comparison purposes, are five runs made during previous quarters. The data for silicates contacting silicates, also calcite contacting calcite, are presented in Figures 1, 2 and 3. Figure 4 presents the data obtained for silicates contacting metals.

The general behavior of the adhesion is the same as that found previously (Ryan, 1965): the higher magnitude adhesion persisting only at UHV, disappearing rapidly in nitrogen (atmospheric pressure), and the lower magnitude, low load, adhesion remaining in nitrogen; also, surface damage and material transfer were evident whenever the higher magnitude adhesions were present.

5.2 Adhesion Between Vacuum-Cleaved Surfaces

Eleven vacuum cleavage runs have been made to date. Of these one was essentially completely successful, eight were partially successful (in that though experimental difficulties developed some useful information was obtained) and two were entirely unsuccessful. The pertinent experimental conditions are given in Table II.

Run #28: Cleavage Along Orthoclase (001) Plane

Cleavage was performed at a pressure of 1×10^{-10} mm Hg. The metal sleeve used to prevent fracture in the zone of sample weakness around the cross-pin hole slipped so that although the desired cleavage was produced, fracture occurred in this area also. During impact of the chisel to produce cleavage, a brief burst of gas entered the system (see discussion on Run #38) raising the pressure momentarily possibly into the 10^{-8} mm Hg range (exactly how high the pressure rose is not known since the protective relay on the ionization gage control tripped). The pressure then fell to the mid 10^{-10} mm Hg range. It was found that the cause of the gas burst was a slight leak in the bellows seal which opened momentarily during impact. Following cleavage,

the upper sample rotated about 10° (the support wire had been purposely twisted to ensure such rotation), displaced about 1 mm with respect to the bottom sample, and then, within 1-2 seconds after cleavage, recontacted the bottom sample. Upon contact, the samples adhered strongly and the microbalance was unable to separate them. A number of impacts of the chamber base plate immediately beneath the samples sufficed to cause separation of the lower sample into two sections with the upper half remaining firmly affixed to the upper sample. Estimates of the force required to cause this separation indicated that the force of adhesion was orders of magnitude greater than the pulling capacity of the balance (i.e. $\gg$ 0.4 gm). A number of unsuccessful attempts were made to separate the upper two samples. It is of interest to note that the cleavage surface produced was good except for a ridge at one edge. The upper sample was resting on this ridge so that the adhering surfaces were canted at an angle to each other.

The adhering samples were then used to contact the remaining half of the lower sample. Initial contact (no external load force applied) resulted in an adhesion force of about 50 mg (it should be noted that these surfaces were quite irregular so that no attempts to obtain sample parallelism were made; also, first contact was made about 15 minutes after initial cleavage, and with the observed gas burst during cleavage it must be assumed that a significant amount of contamination was already present on the surfaces). This adhesion force decreased over a period of 21 hours to about 15 mg at which time dry nitrogen was admitted to the system. The upper samples immediately separated (possibly due to wedging action of the adsorbed nitrogen in the potentially highly strained regions of true contact) and the newly exposed face contacted the steel bucket. It adhered to the bucket, and tapping of the base plate was required for separation. Recontact indicated a much smaller adhesion force, and all indications of adhesion disappeared shortly. Optical study of this surface revealed that a considerable amount of metal from the bucket was present on, and adhering firmly to, the surface.

An additional observation at vacuum was the presence of a relatively strong long range attractive force. This force, indicating a very great amount of surface charging, was of sufficient magnitude to pull the samples into contact

for surface separations less than 0.5 mm. The force remained constant over the entire 21 hour period at vacuum. It disappeared immediately upon admission of nitrogen to the system.

Run #29: Cleavage Along Orthoclase (001) Plane

Cleavage was performed at a pressure of 1×10^{-10} mm Hg. During cleavage the steel bucket was knocked from the upper sample, so it was not possible to zero the microbalance and only qualitative information could be obtained. The observations were similar to those of the first run, the following points being particularly worth mention. After initial cleavage, the upper sample recontacted the lower sample (prior to rotation). Adherence was immediate and it was necessary to use the cleavage chisel to separate them. The upper sample then rotated about 20°. The linear motion feedthrough (the microbalance is attached to this) was used to bring the samples into and out of contact, and it was estimated that the adhesion force was considerably in excess of that noted for the first run (it was found on later inspection that the required pulling force was sufficiently great to severely damage the microbalance). The long range force was also somewhat greater than that of the previous run, the samples being pulled together at separations less than 1.0 mm. This long range force remained constant over a period of 18 hours, disappearing immediately upon admission of nitrogen to the system.

Run #30: Cleavage Along Orthoclase (001) Plane

For this run the microbalance was replaced by a Chatillon spring. Spring extension and hence adhesion force were measured with a cathetometer. This arrangement allowed adhesion forces as small as about 0.2 gm, and as large as 50 gm, to be measured.

Cleavage was performed at a pressure of 2×10^{-10} mm Hg. During cleavage the metal bucket tipped and the sample came loose from the copper support sleeve. The metal bucket was then removed from the sample vicinity, using the cleavage device for manipulation. Upon cleavage the upper sample rotated about 10°, recontacted the lower sample, and lifted it out of the support sleeve. The lower sample was then reinserted into the sleeve and held

in place with the chisel. The first measurement was taken fifteen minutes after cleavage. This gave a force of adhesion of 8 gm. A strong long range attractive force was noted. This was sufficient to pull the samples together when separated by about 1 mm, giving a force of about 2 gm. A second measurement of adhesion force was made five minutes after the first. This gave an adhesion force of 3 gm, and a pull-down force again of 2 gm. The third measurement gave a force of adhesion and pull-down force both of 2 gm. Subsequent readings maintained this equivalence of forces. The system was kept at vacuum for 234 hours. During this time, the magnitude of the adhesion, and the long range force, decreased slowly to about 0.4 gm. The data are plotted in Figure 8.

Upon admission of nitrogen to the system, all indications of adhesion immediataly disappeared.

Run #31: Cleavage Along Orthoclase (001) Plane

The metal bucket was replaced by a rigidly attached metal slug for this run. Cleavage was performed at a pressure of 1.5×10^{-10} mm Hg. No long range force was detected and only a slight indication, below measurement capability, was noted. Study of the cleaved surfaces revealed that the cleavage plane was almost perfect, much more so than for any previous run. The slot to initiate cleavage in this particular sample was cut much deeper than for the previous samples and it is felt that perhaps during cutting, some of the cutting fluid may have penetrated into the subsequent cleavage plane. More work is required before a firm conclusion can be reached in this regard.

Run #32: Cleavage Along Orthoclase (001) Plane

The sample attachment wire failed during cleavage and no data were obtained.

Run #33: Cleavage Along Orthoclase (001) Plane

Cleavage was performed at a pressure of 8×10^{-11} mm Hg. During cleavage the sample fractured at the crosspin hole. The upper sample rotated 45°, recontacted the upper segment of the lower sample, and lifted it into the air. This indicated an adhesion force greater than 0.1 gm (the weight of the

adhering segment). Attempts were made to separate the samples with the chisel. Upon separation the lower sample fell to one side and hence no further data could be obtained.

Run #34: Cleavage Along Orthoclase (001) Plane

Cleavage was performed at a pressure of 2×10^{-10} mm Hg. The same difficulties arose as in Run #33. The observations were also similar to Run #33.

Run #35: Cleavage Along Orthoclase (001) Plane

The sample attachment wire failed during cleavage and no data were obtained.

Run #36: Cleavage Along Orthoclase (001) Plane

The sample shape was changed for this run to permit its being held to the base plate by a press fit, and hence avoiding the problem of unwanted fracture. During cleavage a leak developed in the bellows supporting the chisel, raising the pressure to 6×10^{-7} mm Hg. No adhesion or long range force could be detected.

Run #37: Cleavage Along Microcline (001) Plane

A new chisel and anvil were installed for this run. These allowed more precise alignment and control than was possible with the previous set. Unfortunately, the anvil contained a small leak and the base pressure reached was only 9×10^{-10} mm Hg. It was decided to cleave away, in order to determine what would happen at this relatively high pressure.

Following cleavage, a slight indication of a long range attractive force was detected, but its magnitude was too small for measurement.

Run #38: Cleavage Along Labradorite (001) Plane

Cleavage was performed at a pressure of 2×10^{-10} mm Hg. During cleavage the Bayard-Alpert gage indicated a pressure surge into the 10^{-8} mm Hg range. The gage indication then dropped rapidly into the low 10^{-9} range, more slowly to about 7×10^{-10} mm Hg, and henceforth very slowly in the course of an hour back to the mid 10^{-10} range. No indications of adhesion were detected.

Pressure surge indications by the Bayard-Alpert, associated with impacting the system, have been observed a number of times. In most cases the pressure reading quickly returns to its initial value; however, in a few cases, noted in the run descriptions, it either remains at the higher value or decreases very slowly. The latter behavior has occurred only during operation of the cleavage device, and indicates that gas has entered the system either through a transient or permanent leak produced during the operation. The former behavior has been found to occur essentially any time the vacuum system is impacted, regardless of where. There appear to be two possible explanations for this. First, the vibrations introduced could cause relative motion of the ion gage leads, hence affecting its signal; or second, the vibrations could cause rapid desorption of gas from the chamber walls, particularly in the vicinity of the ion gage (this latter possibility was suggested by Mr. George Neff who had experienced similar effects of vibration upon ultra-high vacuum systems). Regardless of which of these may be correct, it now appears evident that an alternative method for producing cleavage is desirable. It has been decided that a wedging technique should eliminate (or much reduce) the problem, and this will be applied to the next run.

6.0 DISCUSSION OF DATA

6.1 Air-Formed Surfaces

6.1.1 Silicate-Silicate Data

In the previous annual report (Ryan, 1965), it was found that in general there were two types of adhesion observed. The first type, designated Type A, was characterized by a very rapid rise of adhesion with load force, with the adhesion being of relatively large magnitude at high load; by the presence of the adhesion only in ultra-high vacuum; and by the production of observable surface damage (and material transfer). The second type, designated Type B, was characterized by a much less notable load dependence, and relatively low magnitude even at high load; by the tendency of the adhesion to remain in dry nitrogen after evacuation first to ultra-high vacuum; and by the lack of observable surface damage (and material transfer). It was concluded that Type A behavior was most probably caused through the action of the normal atomic bonding forces, whereas the dispersion forces were possibly responsible for Type B behavior. The results obtained during this year are in general agreement with these conclusions. This pertains not only to the silicate runs but also to the calcite, which represents the only non-silicate mineral investigated.

Though the general behavior is similar there are differences in the detailed behavior. The principal difference involves variation in the adhesion force magnitude between runs involving the same sample materials. It is seen first, from Figures 1, 2 and 3, that the magnitude of the Type B adhesion differs in some cases for a given sample pair. This appears to be due to roughness effects since as can be seen from Table I the adhesion magnitude increases as roughness decreases, and remains the same (albite-orthoclase runs) when no difference in roughness is evident. This type of behavior is to be expected if dispersion forces are, as believed, the causal agent of Type B behavior.

Differences appear also in the adhesion magnitude for Type A behavior. This type of adhesion should be essentially independent of surface roughness if

caused by the atomic bonding forces. Comparison of Figures 1, 2 and 3 with the roughness data in Table I indicates that there is no correlation between adhesion magnitude (Type A) and surface roughness. There are, however, two correlations of possible significance which can be noted from the table and figures: the higher magnitude adhesion for each pair of runs was obtained when sorption pumps were used for roughing, also for the runs where the system pressure was lower. Further work is required before it can be concluded which of these factors may be responsible for the observations, alternatively whether these differences are inherent in the experimental technique.

6.1.2 Silicate-metal Data

The silicate-metal data are presented in Figure 4. The observed behavior of the adhesion, with the exception of the magnesium alloy run, is similar to that reported in the previous annual report in that two distinct branches are present. The magnesium alloy behavior is somewhat different in that, though there is a steepening in the curve at higher loads, the rate of increase at lower loads is significantly greater than previously noted. The behavior is in this sense similar to that found for orthoclase (001) contacting alumina and Corning glass No. 1723 (Ryan, 1965).

6.1.3 Effects of Forepump Type

The data obtained this year using sorption pumps for the forepump are generally similar to those obtained previously with the mechanical pump. The only possible detailed effect noted, see Section 6.1.1, was the slight increase in adhesion magnitude with the sorption pumps. The results indicate that mechanical pumps can be used for adhesion studies provided proper precautions are taken.

6.1.4 Roughness Effects

The data obtained to determine the effects of surface roughness upon adhesion are shown in Figure 5. Curves (1) and (2) were obtained from the roughest surfaces (Runs #2 and 1 of Table I); Curves (3) and (4) were obtained from

surfaces of intermediate roughness (Runs #20 and 19); while Curve (5) was obtained from surfaces which approached optical flatness (Run #21).

Since different crystalline orientations were used in the runs, not much can be said at present about the effects of roughness upon the high load adhesion (Type A) behavior, though as previously noted no correlation between roughness and adhesion magnitude is evident. The effect of roughness upon the low load behavior (Type B), on the other hand, is quite evident. For the roughest surfaces, as load force decreases, the magnitude of the adhesion rapidly drops below detectable. For the intermediate roughness surfaces measurable adhesion remains even at zero load. For the "optical flat" surfaces adhesion also remains at zero load, and in addition its magnitude is considerably greater than for the intermediate roughness surfaces. This behavior is precisely what one would expect if the low load adhesion is caused through the action of the dispersion forces.

6.1.5 Orientation Effects

The studies to date relating to orientation effects are summarized in Figure 6. Of interest are the Type A sections of the curves since the forces producing this type of behavior may show an orientation dependence.

Data have been obtained for the (001) face of orthoclase with various orientations of the respective a-axes (e.g. 10°, 40°, 80°, 90°, 100°, and 190°). The highest magnitude adhesions are found for the 10° orientation, i.e., for the orientation closest to atomic match across the interface. The lowest adhesions are found for the two runs where the a-axes are 90° and 100° out of match. As the orientation approaches 180° the adhesion increases again (190°>150°>90° and 100°), though not to the level attained for the 10° orientation. This behavior is consistent with an orientation effect associated closely with crystal structure.

There is, however, one serious problem remaining. This pertains to Curve (2), for the 80° orientation. To fit with the picture outlined above, this curve should fall close to the 90° and 100° curves. However, the curve falls above all the other curves except the 10° curve. The reason for this is not

presently clear. Hence, it will be necessary to obtain additional curves. In particular, all orientations should be run using a given sample pair. The curves shown here were obtained with four different sample pairs having different surface roughnesses.

6.1.6 Material Transfer

In the previous annual report it was noted that surface damage and material transfer were associated with Type A behavior. This was used as an argument for the atomic (normal bonding) nature of the causal forces.

One difficulty with this argument was that this disruption and transfer could conceivably be caused simply by the effects of surface roughness (even though only normal load was applied; lateral movement on a microscale due to the roughness could occur, resulting in breakage of surface asperities). Accordingly, attempts were made to reproduce this damage and transfer in air by applying normal load and rotating the samples while in contact. No damage or transfer, remotely approaching that found in vacuum, could be obtained. This provided strong evidence that surface roughness was not responsible.

However, it could be argued that roughness was not as effective in air due to the lubricating effect of the air and associated impurities. In order to remove this uncertainty it was decided to make a vacuum run with a pair of "optical flats" (Run #21 of Table I).

High magnitude adhesion was found for these samples (Figure 5). Study of the contacting surfaces showed that considerable surface damage had occurred. This is shown in Figure 7 (the lighter areas are the areas of surface damage and material transfer). In fact, more damage was evident than for any previous run (this may be, however, an observational effect since, due to surface smoothness, damage was more easily visible). In addition, indications were present that extensive material transfer had occurred. This was determined by matching the surfaces to the orientation they had in vacuum and searching for (and finding) correlation across the surfaces between pits and deposits. It is concluded that surface roughness does not play a significant role in producing the observed surface damage and material transfer.

6.2 Vacuum-Cleaved Surfaces

In order for the normal atomic bonding forces to act in producing adhesion between air-formed silicate surfaces, it appears necessary that a significant amount of penetration of the surface contamination must occur, and that sufficient surface distortion must be produced to make bonding sites available. For vacuum cleaved surfaces, however, contamination is initially absent and the charge and coordination demands of the surface atoms are unsatisfied. Hence, distortion is not required (though distortion under load may still act to increase the magnitude of the adhesion) and a large adhesion force should be observed even without prior load. The observations tend to substantiate this expectation.

The only forces which could act to produce the observed adhesion are the normal atomic bonding forces, dispersion forces, and forces produced by surface electrostatic charging. The data obtained to date, particularly from Runs #28 and #30, provide information concerning which of these may be acting. Dispersion forces can be ruled out as being the primary contributor to the adhesion on the basis of Run #28, due to the considerable surface roughness involved on this run. The primary contributor, highest magnitude adhesion, appears to be the normal silicate atomic bonding forces. First, a large decrease in adhesion force with time, principally within the first hour, was observed, contrasted with the constancy of the magnitude of the long range attractive force during the same period (Runs #28 and 30, also Figure 8). Second, the long range force disappeared immediately upon admission of nitrogen to the system, but adhesion remained for a short time (metal bucket to sample, Run #28). Finally, bucket material was transferred to the sample surface. The observations indicate that the adhesion between vacuum-produced surfaces can be quite large. The largest force measured was 8 gm (Run #30). This was measured fifteen minutes after cleavage and as can be seen from Figure 8 the adhesion prior to this would have been much greater.

The long range attractive force present after cleavage must be due to surface charging. None of the other possible forces can have such a great range of effectiveness. It is of interest to consider the possible origin of this charging.

First, piezoelectric or associated effects can be excluded since the crystals used do not possess a center of symmetry. Second, the charge possesses very little or no mobility. It remains for the duration of each run, see Figure 8, and repeated contacts with the chisel and copper base plate had no effect on its magnitude (the samples in all cases were, not unexpectedly, attracted to any metal in the vicinity). The most probable production mechanism appears to be a random or non-random charge separation associated with the breakage of the atomic bonds. If, during cleavage, the bond breakage is a non-random process, that is, one type of ion remains with one surface, then a net surface charge will result. The magnitude of this charge would be highly reproducible for cleavages along a given crystal plane. On the other hand, the breakage may be a random process, and indeed study of the orthoclase structure across the (001) plane indicates this to be the case, then the tendency would be to end up with equal numbers of positive and negative ions, i.e., maintain a net charge neutrality. However, there will be a statistical distribution of possibilities about this null point, so that one surface may find itself with a slight excess of positive charge, the other surface having an equal negative excess. Using the microbalance (Run #28) it was possible to obtain an estimate of the magnitude of the long range force, and hence of the excess charge present.

This excess charge was determined to be $\approx 10^8$ elementary charges. Compared to the total number of bonds broken, $\approx 10^{12} - 10^{13}$, it is seen that the deviation from the null point is extremely small. This hypothesis predicts that the magnitude of the long range force, for cleavages along a given crystal plane, should be highly variable. Both hypotheses indicate that if two simultaneous cleavages are performed, and one face from each cleavage contacted, a repulsive long range force may be evident.

One final point should be noted. Several runs were made when the system pressure, due to leaks, was not in the low 10^{-10}mm Hg range. These were Run #37 at 9×10^{-10}mm Hg, Run #38 with the pressure in the 10^{-9}mm Hg range, and Run 36 with the pressure in the 10^{-7}mm Hg range. No adhesion was detected for either of the latter two runs; a slight indication of adhesion

was observed for the first run. This indicates that system pressure is a critical factor in adhesion, and that large adhesion does not appear until the system pressure enters the low 10^{-10} mm Hg range. Further work will be done on this question.

7.0 SUMMARY OF CONCLUSIONS

The principal conclusions reached during the past year for the air-formed surfaces are:

(1) Two types of adhesional behavior are present, in agreement with the results of the first year's study. The first, Type A, appears to be due to the action of the normal atomic bonding forces; the second, Type B, appears to be due to the action of the dispersion forces. The additional confirmatory data obtained pertain to surface roughness effects (Type A behavior appears independent of surface roughness; Type B is highly dependent upon this), and to the surface damage produced on the optical flats (indicating that surface roughness is not responsible for the surface damage).

(2) Type A behavior appears to have a crystalline orientation sensitivity for given faces in contact but more work is required to confirm this.

(3) On only rare occasions is there any indication of surface charging, and when present the magnitude of the adhesion produced is quite small.

The principal conclusions reached for the vacuum cleaved surfaces are:

(1) The adhesion is orders of magnitude greater than for the air-formed surfaces.

(2) The primary contributor to the adhesion appears to be the normal atomic bonding forces.

(3) A large degree of surface charging is produced, and this is most probably due to charge separation associated with the bond breakage.

(4) Relatively large adhesion persists for a considerable length of time; the total time it will persist is, however, not as yet known.

The principal implications of these results to the Moon are:

(1) Atomic adhesion can be the major factor in determining the strength of the lunar surface material.

(2) The degree to which it will act is critically dependent upon the precise nature of the contacting surfaces; hence, that if environmental differences, particularly the amount of gas which has been released from the lunar interior, exist over the surface the degree to which atomic adhesion can contribute to surface strength will vary greatly.

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Table I (page 1 of 3)

EXPERIMENTAL CONDITIONS FOR AIR FORMED SAMPLES

Run No.	Upper Sample	Lower Sample	Orientation	Pressure (mm Hg)	Forepump Type	Surface Roughness (peak to peak)
15	Calcite $(10\bar{1}1)$	Calcite $(10\bar{1}1)$	a_2-axis/a_2-axis $\cong 180°$	2×10^{-10}	Mechanical	≈1.5 microns
16	Tektite	Tektite	--	2×10^{-10}	Mechanical	≈1 micron
17	Orthoclase (001) [0(//)1T]	Orthoclase (001) [0(//)1B]	a-axis/a-axis $\cong$ 40°	2×10^{-10}	Mechanical	≈4 microns
18	Orthoclase (001) [0(//)1T]	Orthoclase (001) [0(//)1B]	a-axis/a-axis $\cong$ 150°	2×10^{-10}	Mechanical	≈4 microns
19	Orthoclase (001) [0(//)4T]	Orthoclase (001) [0(//)4B]	a-axis/a-axis $\cong$ 100°	4×10^{-10}	Sorption	≈0.5 microns
20	Orthoclase (001) [0(//)4T]	Orthoclase (001) [0(//)4B]	a-axis/a-axis $\cong$ 190°	4×10^{-10}	Sorption	≈0.5 microns

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Table I (page 2 of 3)

EXPERIMENTAL CONDITIONS FOR AIR FORMED SAMPLES

Run No.	Upper Sample	Lower Sample	Orientation	Pressure (mm Hg)	Forepump Type	Surface Roughness (peak to peak)
21	Orthoclase (001) [0(//)F1]	Orthoclase (001) [0(//)F2]	a-axis/a-axis ≅ 90°	3×10^{-10}	Sorption	≈300 Å
22	Hypersthene (110) [Hs(//)NP]	Orthoclase (001) [0(//)2T]	a-axis trace/ a-axis ≅ 25°	1×10^{-10}	Sorption	≈3 microns
23	Albite (001) [A(//)NP]	Orthoclase (001) [0(//)2B]	a-axis/ a-axis ≅ 25°	2×10^{-10}	Sorption	≈3 microns
24	Hornblende (101) [Hb(⊥)IT]	Bytownite (001) [B(//)NP]	a-axis trace/ a-axis ≅ 20°	1×10^{-10}	Sorption	≈5 microns
3	Hypersthene (110) [Hs(//)NP]	Orthoclase (001) [0(//)2T]	a-axis trace/ a-axis ≅ 10°	3×10^{-10}	Mechanical	≈5 microns
4	Albite (001) [A(//)NP]	Orthoclase (001) [0(//)2T]	a-axis/ a-axis ≅ 10°	3×10^{-10}	Mechanical	≈3 microns

Table I (page 3 of 3)

EXPERIMENTAL CONDITIONS FOR AIR FORMED SAMPLES

Run No.	Upper Sample	Lower Sample	Orientation	Pressure (mm Hg)	Forepump Type	Surface Roughness (peak to peak)
6	Hornblende (101) [Hb(⊥)IT]	Bytownite (001) [B(//)NP]	a-axis trace/ a-axis ≅ 30°	3×10^{-10}	Mechanical	≈3 microns
25	Aluminum (2024)	Orthoclase (001) [0(//)4B]	--	2×10^{-10}	Sorption	≈0.6 microns
26	Magnesium (AZ31B)	Orthoclase (001) [0(//)2B]	--	1×10^{-10}	Sorption	≈3 microns for orthoclase ≈0.6 microns for magnesium
27	Nickel	Orthoclase (001) [0(//)1B]	--	2×10^{-10}	Sorption	≈4 microns for orthoclase ≈1 micron for nickel
1	Orthoclase (001) [Ȯs(//)IT]	Orthoclase (001) [Os(//)1B]	a-axis/a-axis ≅ 80°	4×10^{-10}	Mechanical	≈5 microns
2	Orthoclase (001) [0(//)2T]	Orthoclase (001) [0(//)2B]	a-axis/a-axis ≅ 10°	$2\text{-}3 \times 10^{-10}$	Mechanical	≈4 microns

Table II (page 1 of 2)

EXPERIMENTAL CONDITIONS FOR VACUUM FORMED SAMPLES

Run No.	Sample Type	Cleavage Plane	Pressure (mm Hg)	Comments
28	Orthoclase	(001)	1×10^{-10}	Fracture at base.
29	Orthoclase	(001)	1×10^{-10}	Metal bucket falls off.
				No quantitative data.
				Microbalance damage.
30	Orthoclase	(001)	2×10^{-10}	Replace microbalance by spring.
				Sample loose, but able to obtain quantitative data.
31	Orthoclase	(001)	1.5×10^{-10}	Replace bucket by slug.
				No indication of adhesion; used deep slot in sample; most perfect cleavage surface obtained to date.
32	Orthoclase	(001)	2×10^{-10}	Sample attachment wire fails.
33	Orthoclase	(001)	8×10^{-11}	Fracture at base; sample falls to side.
34	Orthoclase	(001)	2×10^{-10}	Fracture at base.
35	Orthoclase	(001)	1×10^{-10}	Sample attachment wire fails.

Table II (page 2 of 2)

Run No.	Sample Type	Cleavage Plane	Pressure (mm Hg)	Comments
36	Orthoclase	(001)	3×10^{-10}	Leak develops; cleave at 6×10^{-7}; no detectable adhesion.
37	Microcline	(001)	9×10^{-10}	Slight indication of adhesion; not measurable.
38	Labradorite	(001)	2×10^{-10}	Gas burst in system, pressure rises into high 10^{-9} range; no detectable adhesion.

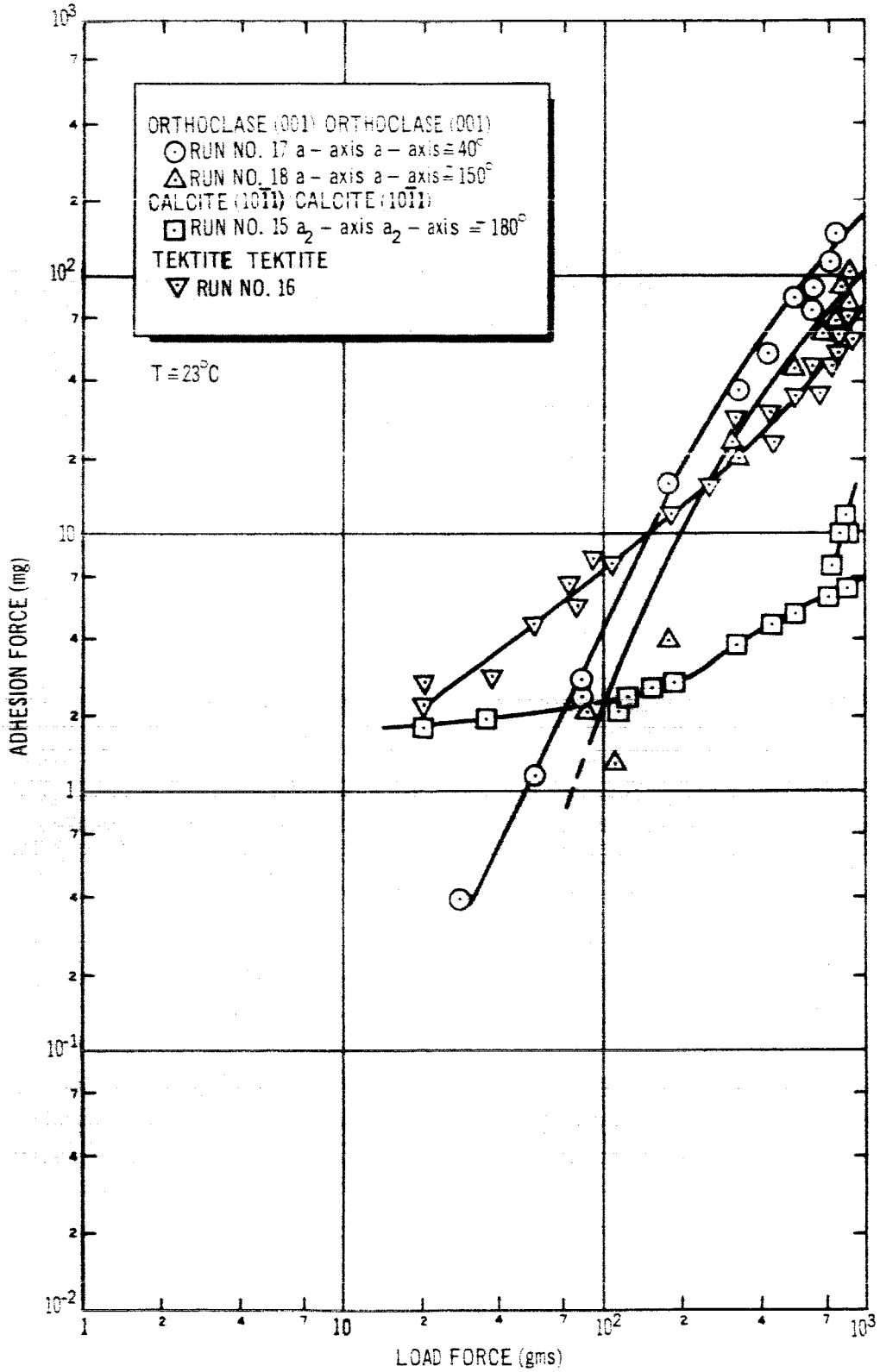

Figure 1. Adhesion Between Various Air-formed Silicates, and Calcite, at Room Temperature and Ultra-High Vacuum

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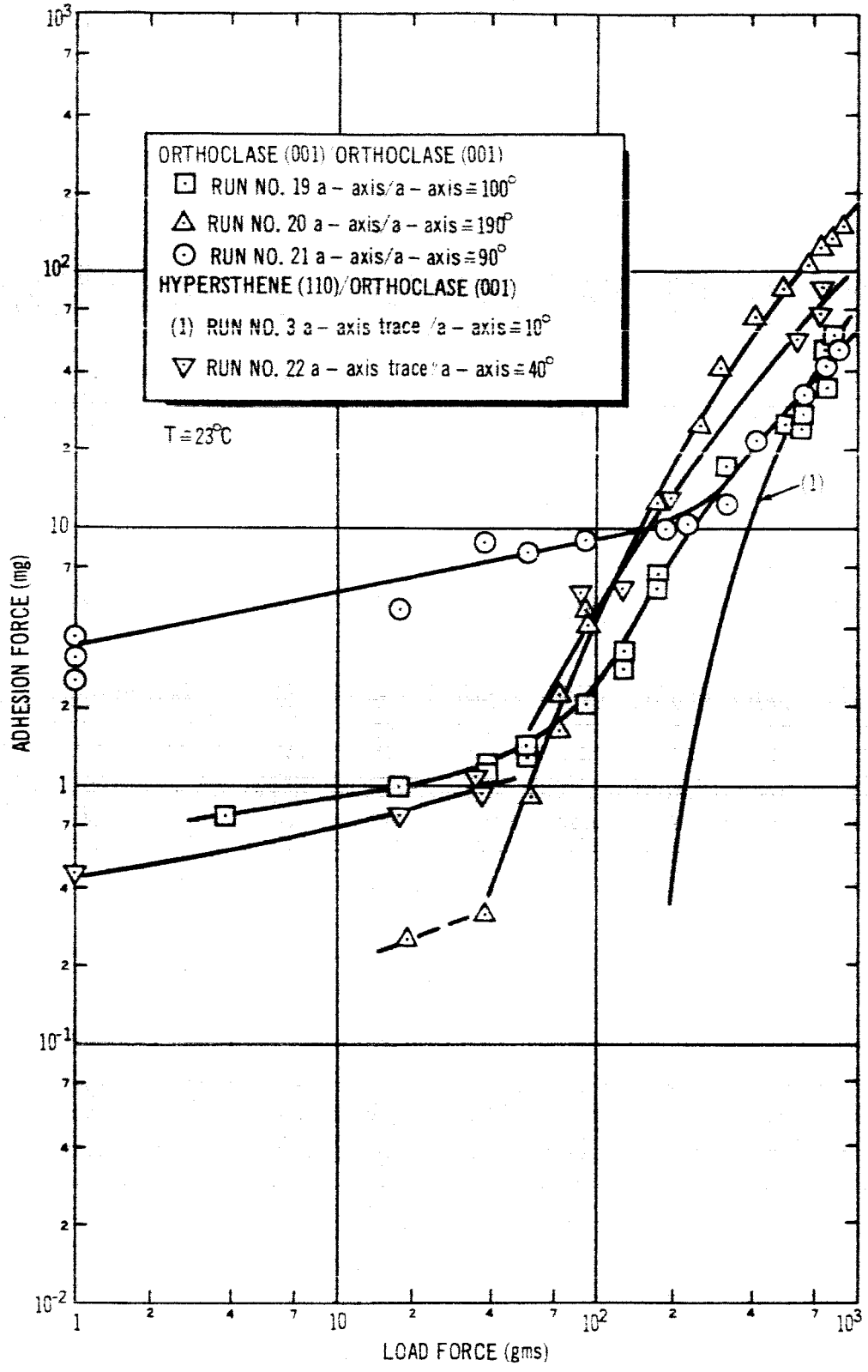

Figure 2. Adhesion Between Various Air-formed Silicates at Room Temperature and Ultra-High Vacuum

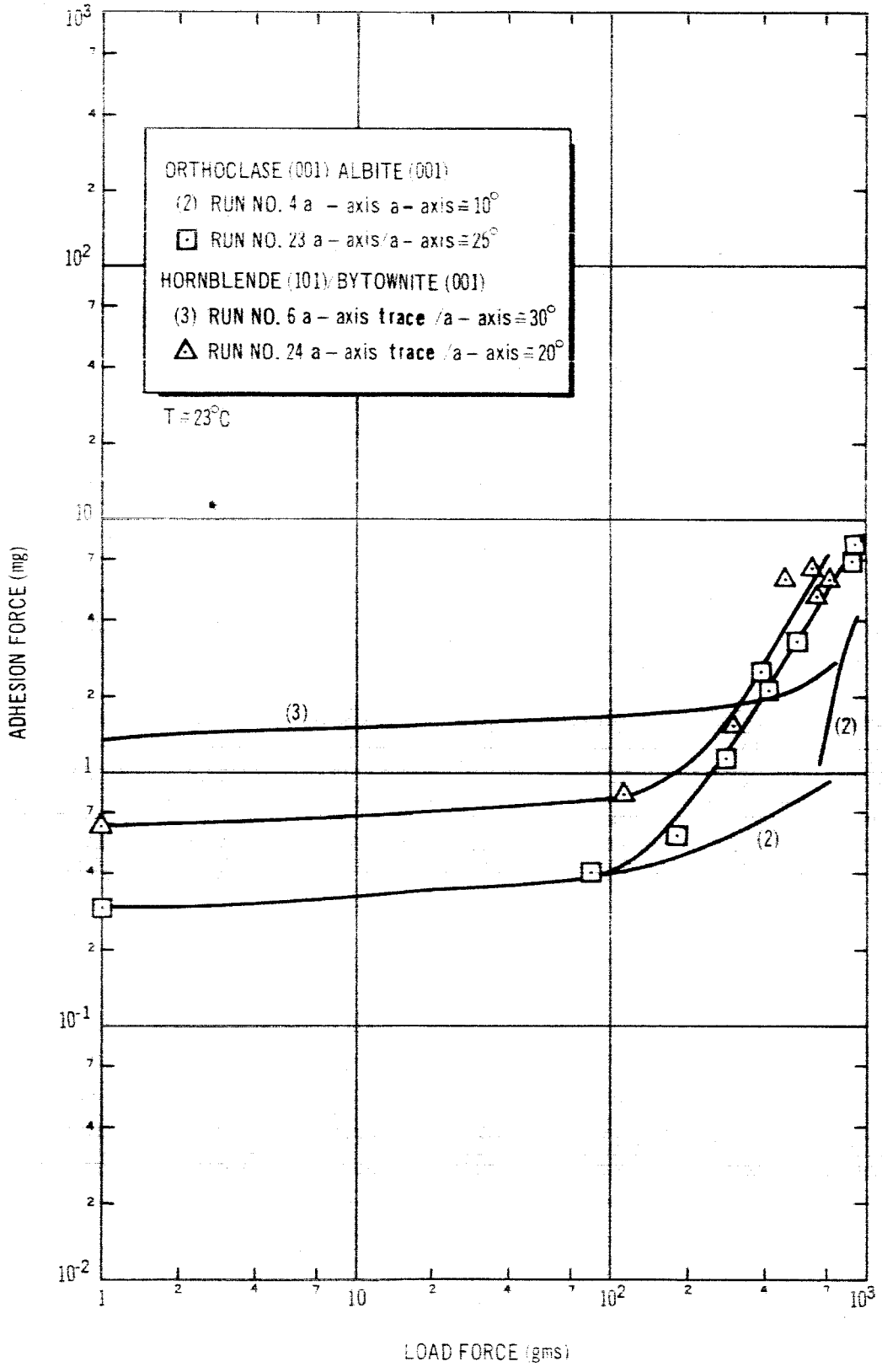

Figure 3. Adhesion Between Various Air-formed Silicates, at Room Temperature and Ultra-High Vacuum

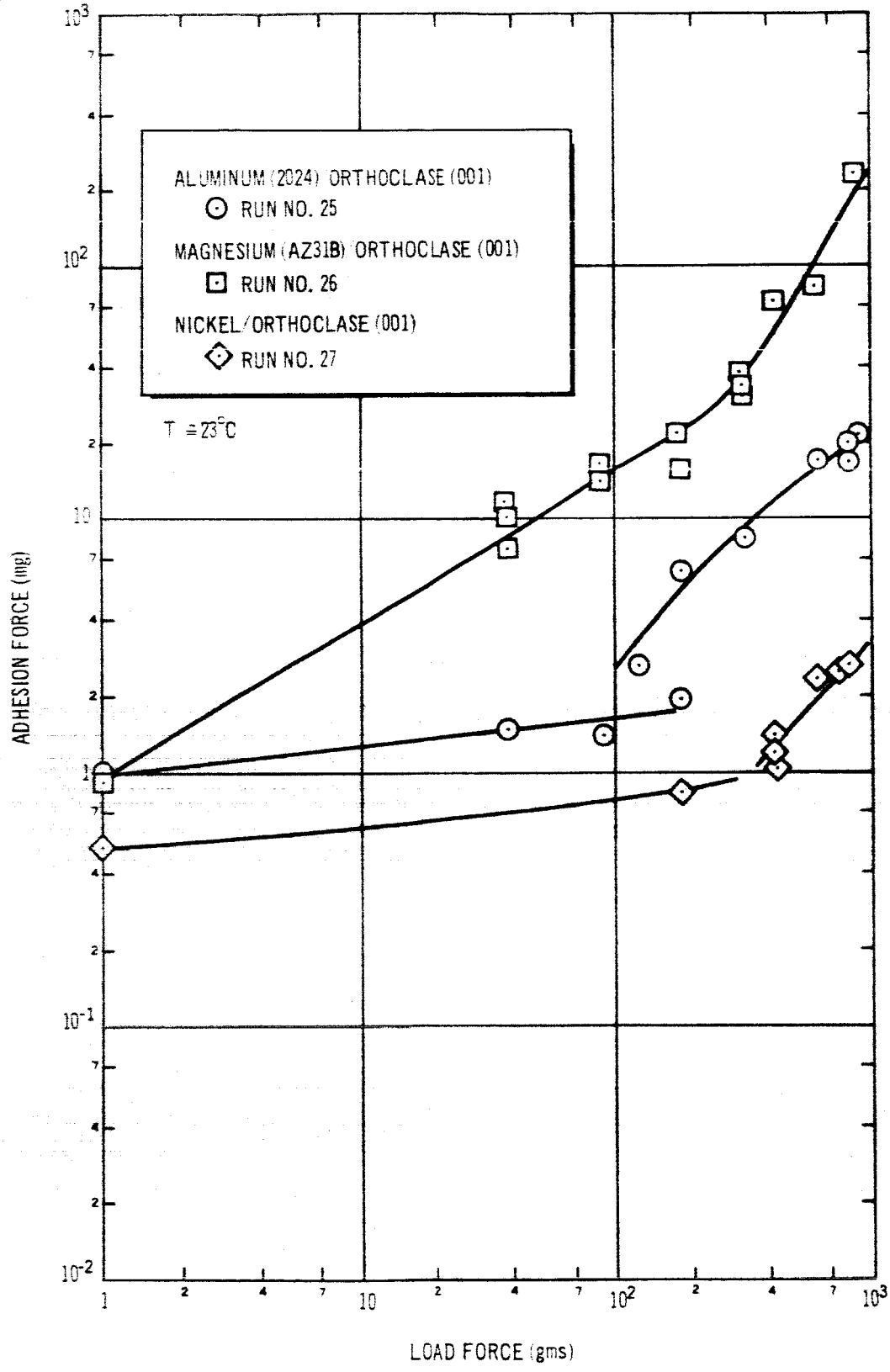

Figure 4. Adhesion Between Various Air-formed Silicates and Metals at Room Temperature and Ultra-High Vacuum

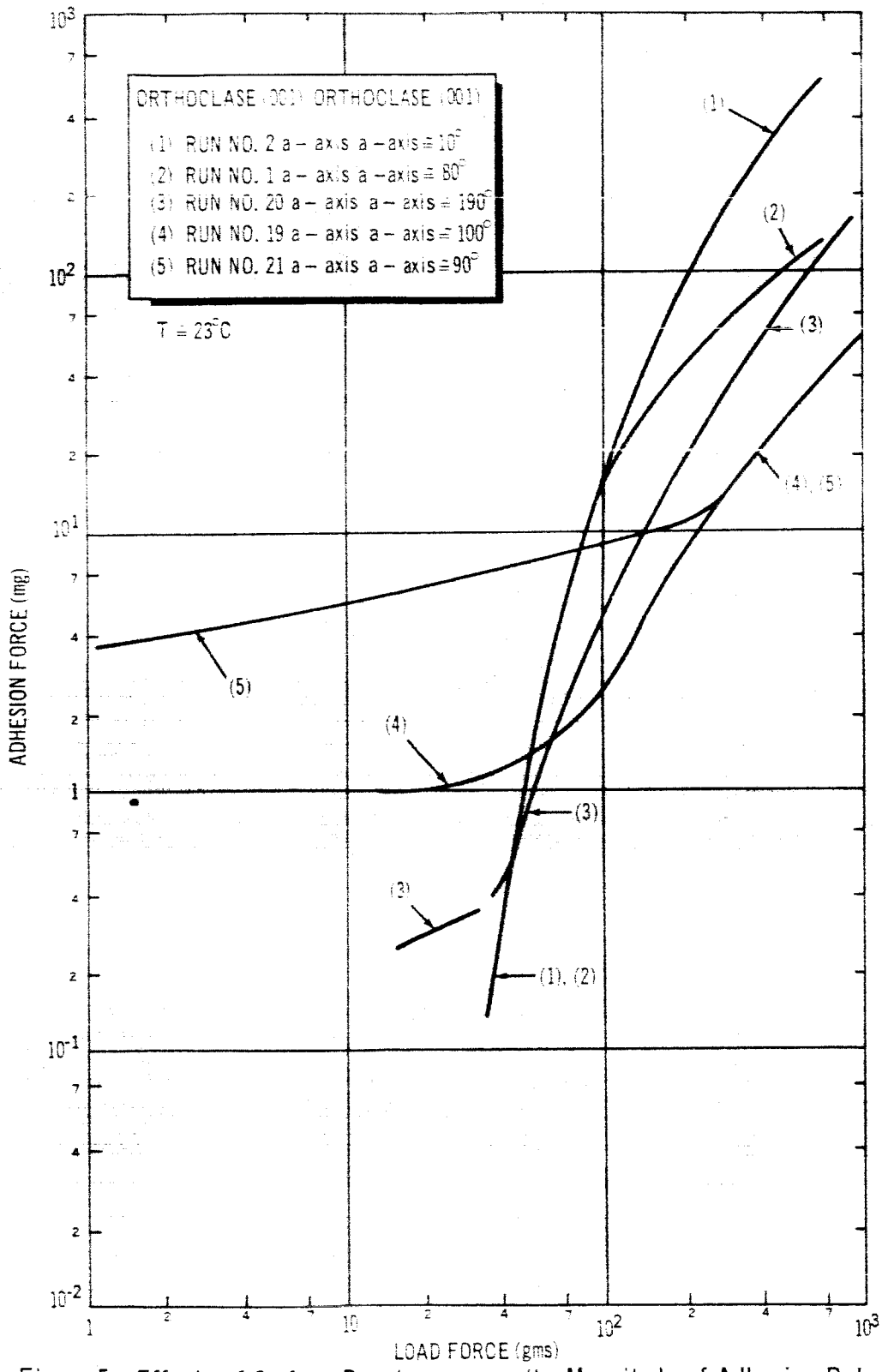

Figure 5. Effects of Surface Roughness upon the Magnitude of Adhesion Between Air-formed Orthoclase at Room Temperature and Ultra-High Vacuum

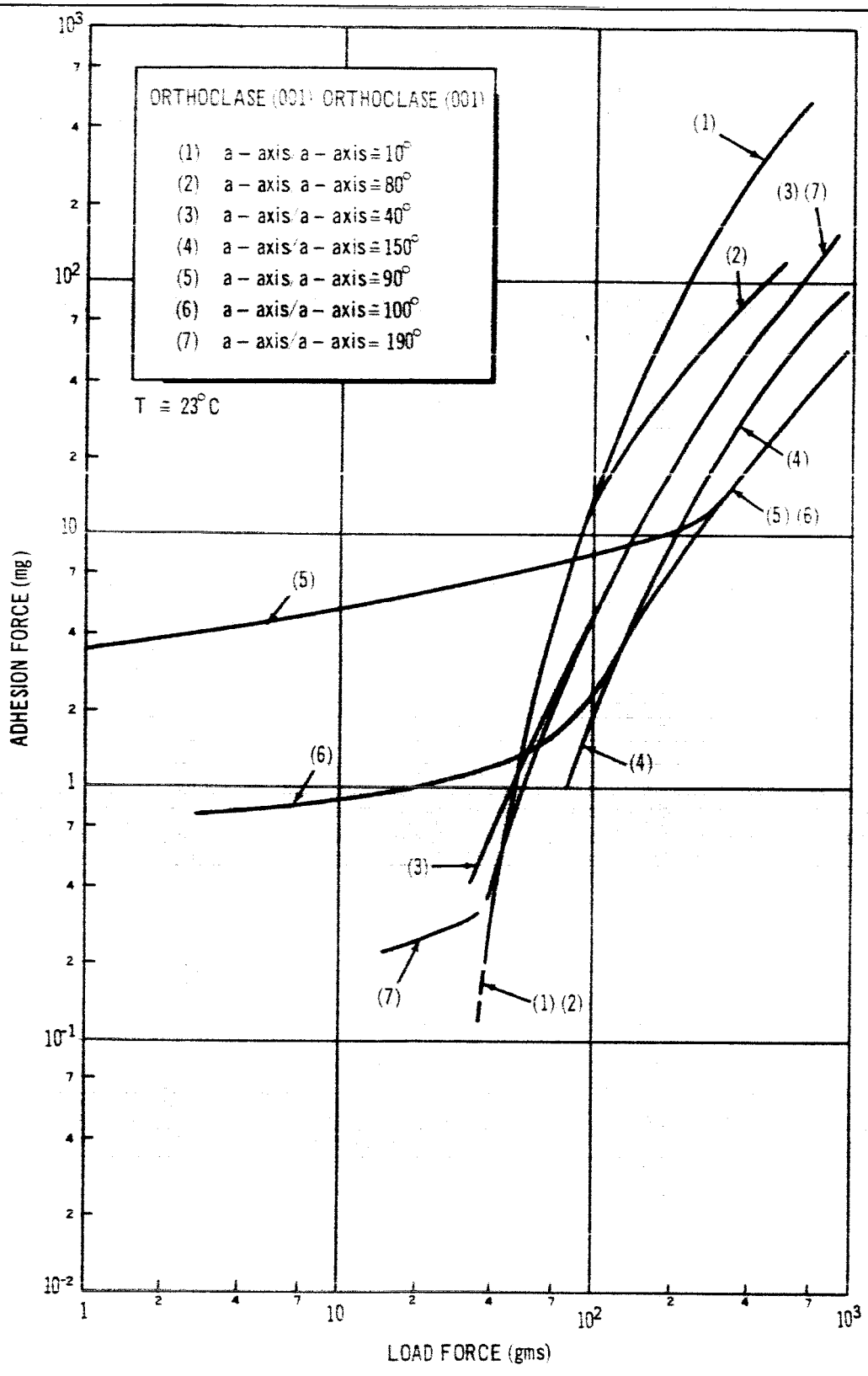

Figure 6. Effects of Crystalline Orientation upon the Magnitude of Adhesion Between Air-formed Orthoclase at Room Temperature and Ultra-High Vacuum

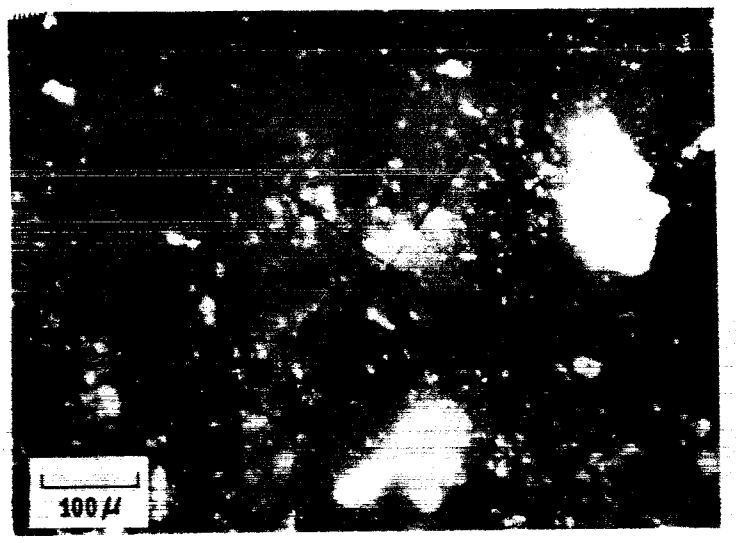

Figure 7. Micrograph of Damage to Optically Flat Orthoclase Surface after Contact with Other Orthoclase Flat at Room Temperature and Ultra-High Vacuum

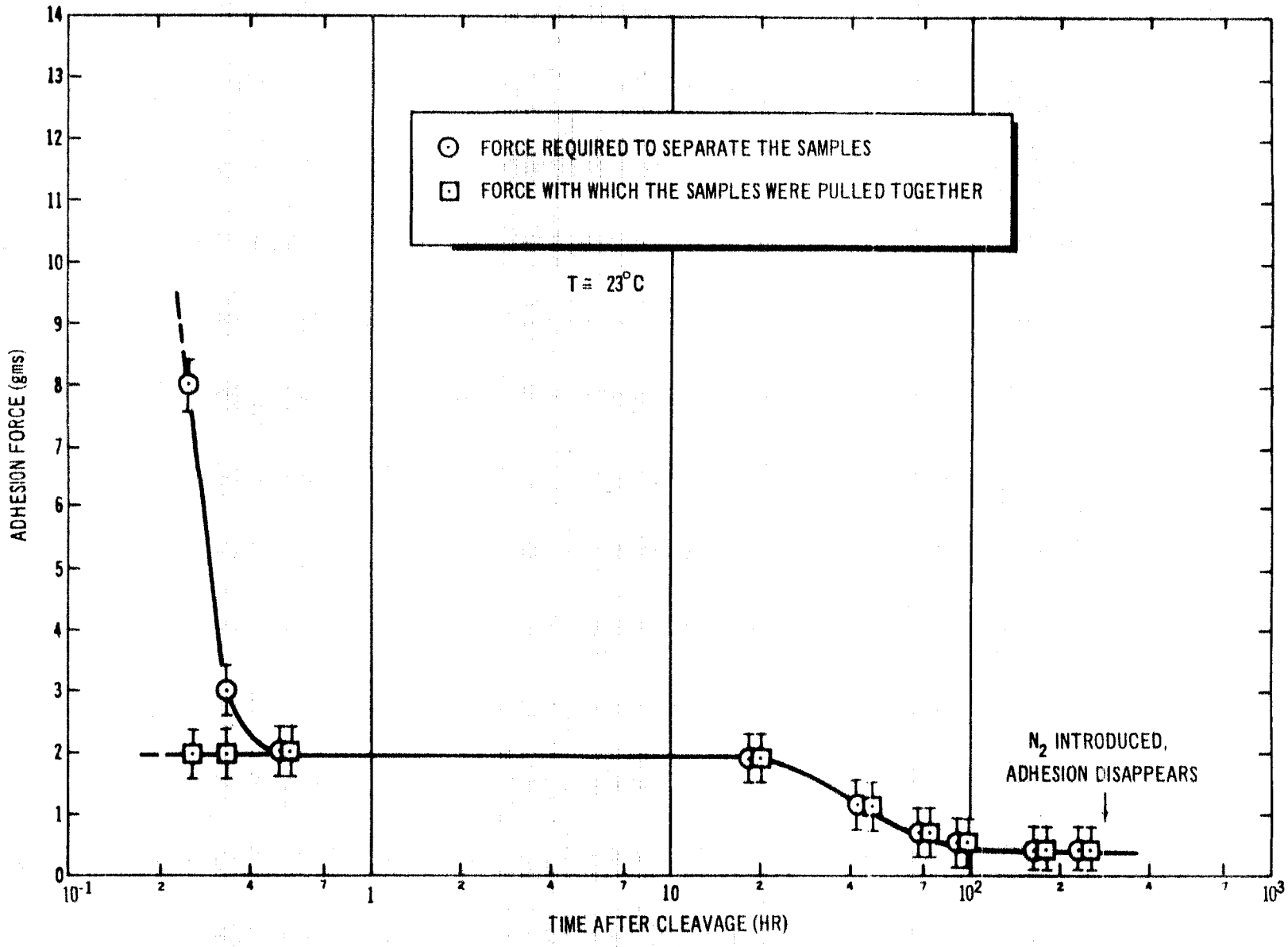

Figure 8. Effect of Time of Exposure to Vacuum (2×10^{-10} mm Hg) on Adhesion Between Vacuum Cleaved Orthoclase (001) Surfaces; Orientation ≅ 10° from Atomic Match in Angle