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

SEVENTH QUARTERLY PROGRESS REPORT

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1.0 INTRODUCTION & SUMMARY

This report presents a summary of the work accomplished during the period January through March, 1966, on the study of the ultra-high vacuum frictional-adhesional behavior of silicates as related to the lunar surface. During this period studies were made to determine data reproducibility for various silicate pairs which had been run previously (reported in previous quarterly reports), runs were made for metal-silicate pairs not tried previously, the vacuum system was modified to permit vacuum cleavage, and two runs involving vacuum cleavage were made.

The data obtained to check reproducibility were for hypersthene (110) contacting orthoclase (001), albite (001) contacting orthoclase (001), and hornblende (101) contacting bytownite (001). It was found that the general behavior of the adhesion was quite reproducible, and that the adhesion magnitude was reasonably reproducible. Some of the differences in adhesion magnitude may be due, however, to the fact that the experimental conditions were not precisely the same between runs (see Table 1).

The additional silicate-metal runs were for aluminum alloy (2024), magnesium alloy (AZ31B), and nickel contacting orthoclase (001). It was found that the adhesional behavior was similar to that obtained previously for metal-silicate contact.

The initial vacuum system modifications, to permit vacuum cleavage, were installation of 1) an impact cleavage device, 2) a chain counterweight for the microbalance, and 3) a metal sleeve around the base of the sample to be cleaved. Two runs were made following these modifications. These were

for orthoclase cleaved along the (001) plane at 1 x 10^{-10} mm Hg. It was found that the adhesion forces were quite large, and that a relatively strong long range attractive force was present. It was concluded, tentatively, that the adhesion was caused principally by the normal silicate bonding forces; also that the long range force was due to surface charging produced by a statistical separation of charge during cleavage.

The adhesion forces, for vacuum cleavage, were sufficiently large to exceed the microbalance's capabilities for measurement. Accordingly, a second set of system modifications was made. These involved principally replacement of the microbalance by a Chatillon precision coil spring, and the use of a cathetometer to measure spring displacement. With this spring it is possible to measure adhesion forces as large as 50 gm and as small as 0.1 gm. Further runs will show whether this spring has a sufficient dynamic range. If it does not, it will be replaced by a spring that does.

2.0 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.

If, during the formation of a fresh surface on the moon an atmosphere is present, either as part of a general lunar atmosphere or generated as a transient phenomenon 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.)

Most of the studies conducted to date have involved measurement of adhesion between surfaces formed in air. These 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 lower bound lunar adhesion case. It was found, from these, that adhesion could indeed occur, but that it became significant only after the application of load force. The relatively high adhesion forces detected have been ascribed to the silicate bonding forces, and it appears that only with a previously applied load force can the residual surface contamination

be penetrated, and does sufficient distortion occur to make bonding sites available.

The recently begun vacuum cleavage studies represent the upper bound case. For these runs the surface demands are initially unsatisfied, and no contamination is present. The results obtained to date, discussed in following sections, indicate that for this case the magnitude of the adhesion can be very large, and that the problems posed to lunar missions can be quite serious.

3.0 EXPERIMENTAL

3.1 Vacuum System Modification to Permit Vacuum Cleavage

Three modifications to the experimental system were necessary in order to initiate 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 consists of a bellows-mounted tool steel chisel and a sample support bracket, also bellows-mounted, to provide support opposite the cleavage point. The details of these were given in a previous quarterly report. The sample to be cleaved is notched and the chisel tip inserted into this notch. Cleavage is obtained by impacting the chisel from outside the vacuum system. Following cleavage both the chisel and the sample support bracket are withdrawn from the vicinity of the samples. The samples are then rotated, to insure atomic mismatch, the microbalance zeroed, and the samples contacted.

Since it is not possible to anticipate the exact weight of the upper sample it was necessary to replace the 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 is then possible to zero the microbalance even when it is 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. This was sufficient to prevent unwanted fracture.

3.2 Experimental Data for the Air-Formed Surfaces

The experimental conditions under which the data were obtained are given in Table 1. Included in this table, for comparison purposes, are three runs made during previous quarters. The data for silicates contacting silicates are presented in Figure 1. Figure 2 presents the data obtained for silicates contacting metals. Roughness plots for the metal surfaces are given in Figures 3-5.

The general behavior of the adhesion is the same as that found for previous runs; 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.

3.3 Vacuum Cleavage Data

Vacuum preparation of the surfaces to be contacted has only recently begun. Two runs have been made as of this writing, and though neither has been 100% successful they are included here since they have provided important information regarding the effects of surface preparation upon adhesion phenomena, and have provided further insight into the general problem of silicate adhesion. Also, these runs represent, to the author's knowledge, the first silicate vacuum cleavage involving breakage of Si-O bonds.

The two runs were for orthoclase cleaved along the (001) plane. The evacuation procedure was similar to that for the air-formed samples. Cleavage, in both cases, was performed at a pressure of about 1×10^{-10} mm Hg, and at room temperature. For the first run, the metal sleeve used to prevent fracture in the zone of sample weakness around the cross-pin hole slipped so that though 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 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 quickly fell to the low 10^{-10} mm Hg range. Further impacts of the chisel device were made, with the chisel out of contact with the samples, and 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. $\gg\gg$ 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 bursts during cleavage, and during the search to determine the cause of these, 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.

The second run produced a single cleavage, but unfortunately during cleavage the steel bucket was knocked from the upper sample, hence it was not possible to zero the microbalance and only qualitative information could be obtained. During cleavage, a slight pressure rise occurred, again due to the bellows seal, but the pressure did not rise above the mid 10^{-10} mm Hg range. The observations were similar to those of the first run, the following points being particularly worth mentioning. 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 for 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.

4.0 DISCUSSION AND CONCLUSIONS

4.1 Air-formed Surfaces

4.1.1 Silicate-silicate data

The general behavior of the adhesion, Figure 1, can be seen to be similar to that found previously. However, there are significant differences in the detailed behavior between runs involving the same sample materials, these differences involving the magnitude of the adhesion force. It had been concluded previously that the adhesion at low load was produced through the action of dispersion forces. It is seen, from Figure 1, however, that the magnitude of this adhesion for a given sample pair differed between runs for two of the three cases shown. This appears to be due to roughness effects, since as can be seen from Table 1 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 the causal agent.

It had previously been concluded that the high load adhesion was caused by the normal silicate bonding forces. The magnitude of this adhesion appeared (previous quarterly report) to be independent of surface roughness. It is seen from the figure that for the runs obtained during this quarter the adhesion magnitude is larger than that obtained for the corresponding previous runs. Also, this behavior tends to appear at lower load forces than previously noted. From Table 1 it is seen that there is no correlation between surface roughness and the magnitude of the high load adhesion. There are,

however, two correlations of possible significance which can be noted from the table and figure: 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.

4.1.2 Silicate-metal data

The silicate-metal data are presented in Figure 2. The observed behavior of the adhesion, with the exception of the magnesium alloy run, is similar to that reported previously in that two distinct branches are present. The magnesium alloy behavior is 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 for the metals. The behavior is in this sense similar to that found for orthoclase (001) contacting alumina and Corning glass No. 1723.

4.2 Vacuum Cleaved Surfaces

Obviously it is premature to attempt a detailed discussion of the adhesion of vacuum cleaved silicate surfaces on the basis of only two, and these not completely successful, runs. However, the results obtained are of sufficient interest to warrant consideration of the implications involved, and the processes possibly responsible for the observed behavior.

In the previous section it was concluded that for Type A behavior (produced by the normal silicate bonding forces) to occur, a significant amount of

contamination penetration and surface distortion was required. For vacuum-cleaved surfaces, however, contamination is initially absent and the charge and coordination demands of the surface atoms unsatisfied (hence distortion is not required, though distortion under load may still act to increase the magnitude of the adhesion). As might be expected then, touch contact should be sufficient to bring the normal bonding forces into play and hence one would expect rather basic differences in the adhesional behavior of air-and vacuum-formed surfaces. The observations tend to substantiate this expectation.

The normal bonding and surface charging produced forces are the only ones which could be acting to produce the observed adhesion (hydrated and adsorbed layers can be excluded from consideration; dispersion forces can be ruled out due to the considerable surface roughness). Of the two, it appears that the normal bonding forces are the primary contributors. First, a threefold decrease in the adhesion force with time was observed, contrasted with the constancy of the magnitude of the long range force. Second, the long range force disappeared immediately upon admission of nitrogen to the system, but large adhesion remained for a short time (metal bucket to sample). Third, bucket material was transferred to the sample surface. Finally, due to the large number of atomic bonds broken during cleavage, it is difficult to believe that the normal bonding forces would not act strongly. The observations indicate that the adhesion between vacuum-produced surfaces can be quite large (though the only numerical data obtained give forces only in the 15-50 mg range, these particular surfaces were exposed directly to the observed gas bursts and were contaminated to an unknown degree). Final confirmation of this must await, however, the obtaining of quantitative data

for like and unlike surfaces.

The probable mechanism producing the long range force is worth note. 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 (first run) 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 $\cong 10^{8}$ elementary charges. Compared to the total number of bonds broken, $\cong 10^{12}$-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.

TABLE 1
EXPERIMENTAL CONDITIONS

Run No.	Upper Sample	Lower Sample	Orientation	Pressure (mm Hg)	Forepump Type	Surface Roughness
22	Hypersthene (110) Hs(//)NP	Orthoclase (001) O(//)2T	a-Axis trace/ a-Axis $\cong 40^{\circ}$	1×10^{-10}	Sorption	$\approx$ 3 microns Peak to peak
23	Albite (001) A(//)NP	Orthoclase (001) O(//)2B	a-Axis/ a-Axis $\cong 25^{\circ}$	2×10^{-10}	Sorption	$\approx$ 3 microns Peak to peak
24	Hornblende (101) Hb($\perp$)IT	Bytownite (001) B(//)NP	a-Axis trace/ a-Axis $\cong 20^{\circ}$	1×10^{-10}	Sorption	$\approx$ 5 microns Peak to peak
3	Hypersthene (110) Hs(//)NP	Orthoclase (001) O(//)2T	a-Axis trace/ a-Axis $\cong 10^{\circ}$	3×10^{-10}	Mechanical	$\approx$ 5 microns Peak to peak
4	Albite (001) A(//)NP	Orthoclase(001) O(//)2T	a-Axis/ a-Axis $\cong 10^{\circ}$	3×10^{-10}	Mechanical	$\approx$ 3 microns Peak to peak
6	Hornblende (101) Hb ($\perp$)IT	Bytownite (001) B(//)NP	a-Axis trace/ a-Axis $\cong 30^{\circ}$	3×10^{-10}	Mechanical	$\approx$ 3 microns Peak to peak
25	Aluminum (2024)	Orthoclase (001) O(//)4B	---	2×10^{-10}	Sorption	See Fig. 3
26	Magnesium (AZ31B)	Orthoclase (001) O(//)2B	---	1×10^{-10}	Sorption	See Fig. 4
27	Nickel	Orthoclase (001) O(//)1B	---	2×10^{-10}	Sorption	See Fig. 5

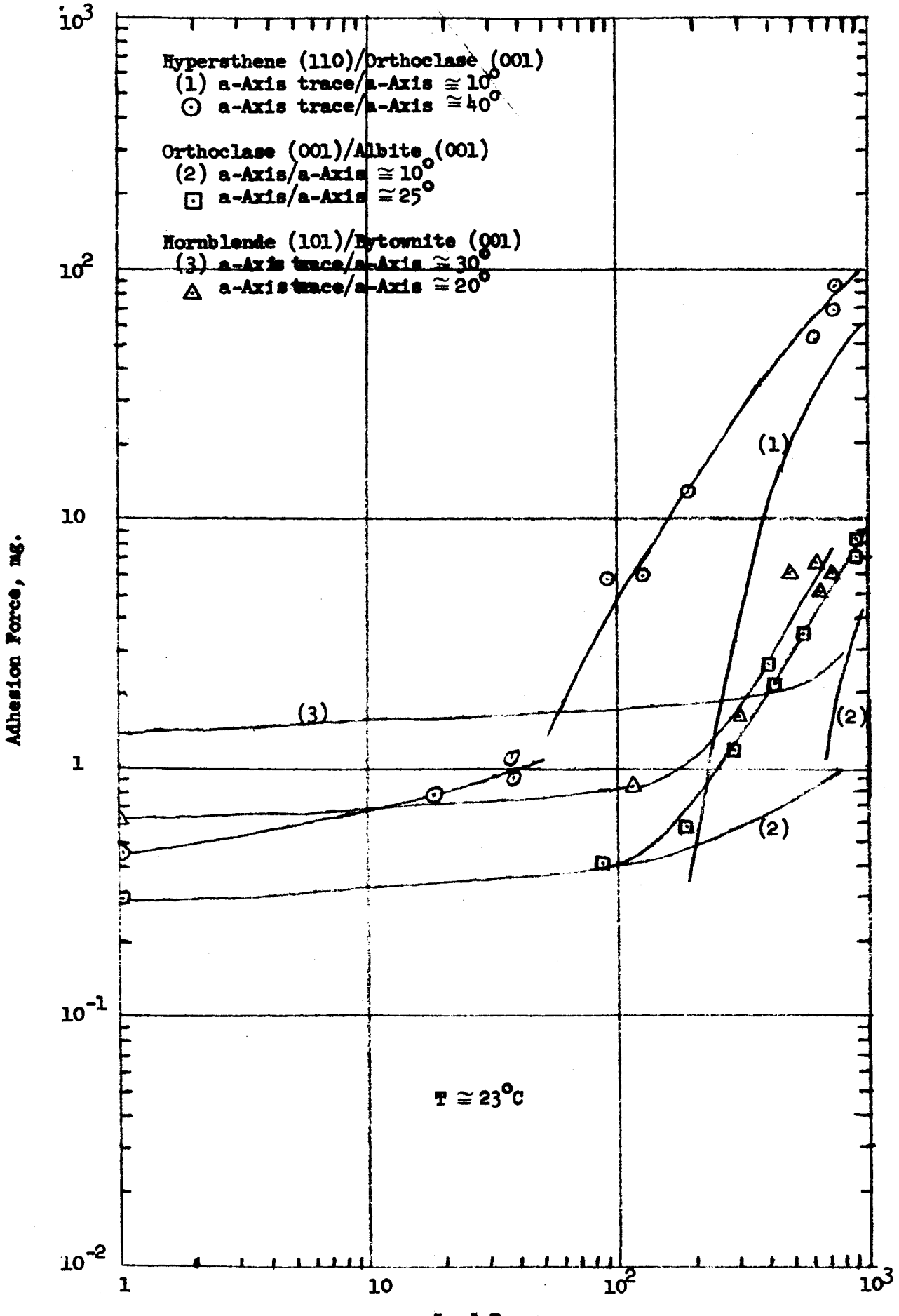

Hypersthene (110)/Orthoclase (001)
(1) a-Axis trace/a-Axis ≅ 10°
⊙ a-Axis trace/a-Axis ≅ 40°
Orthoclase (001)/Albite (001)
(2) a-Axis/a-Axis ≅ 10°
⊡ a-Axis/a-Axis ≅ 25°
Hornblende (101)/Bytownite (001)
(3) a-Axis trace/a-Axis ≅ 30°
△ a-Axis trace/a-Axis ≅ 20°
T ≅ 23°C
Adhesion Force, mg.
Load Force, gm.
10^3
10^2
10
1
10^-1
10^-2
1
10
10^2
10^3

Figure 1

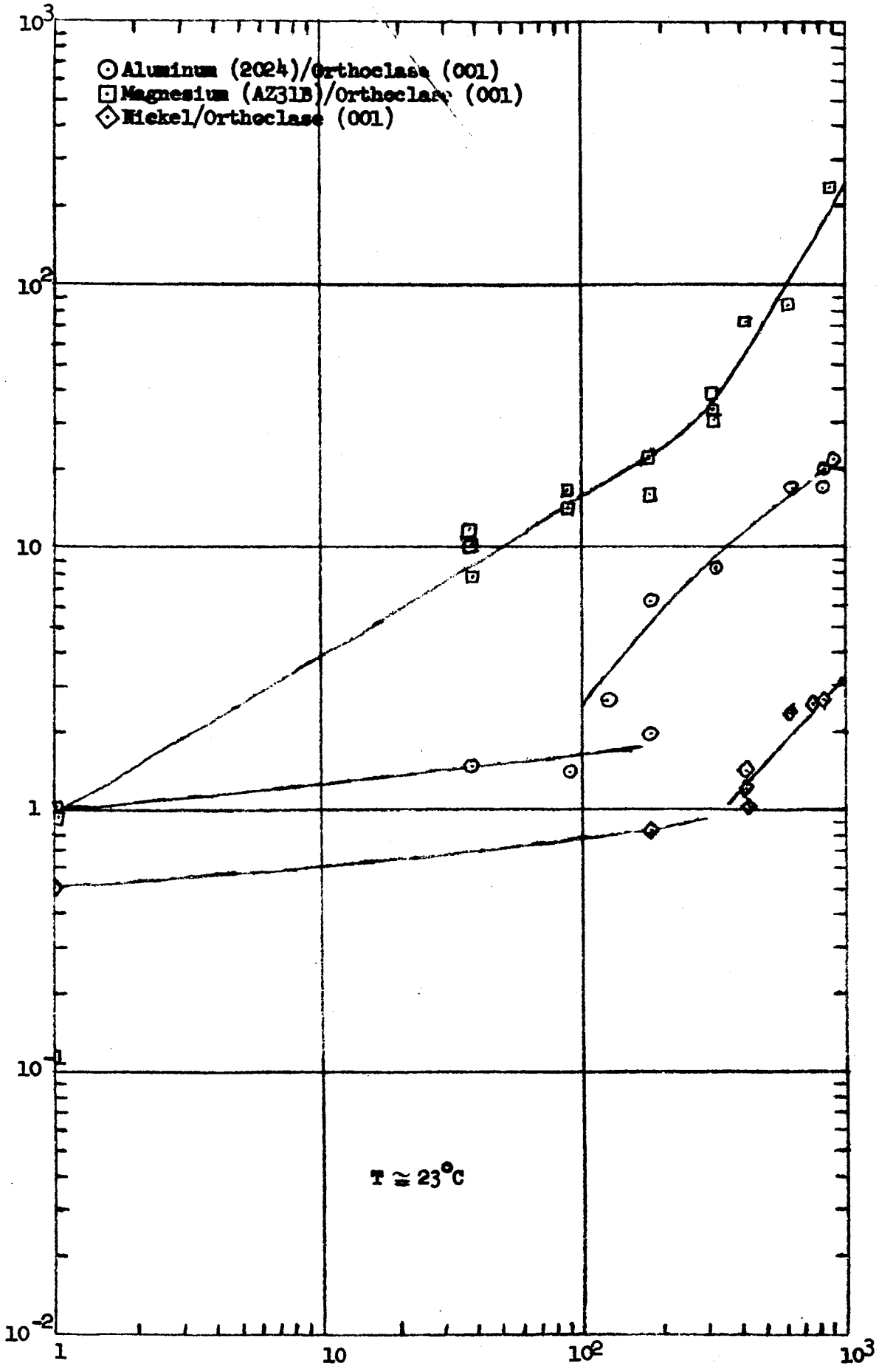
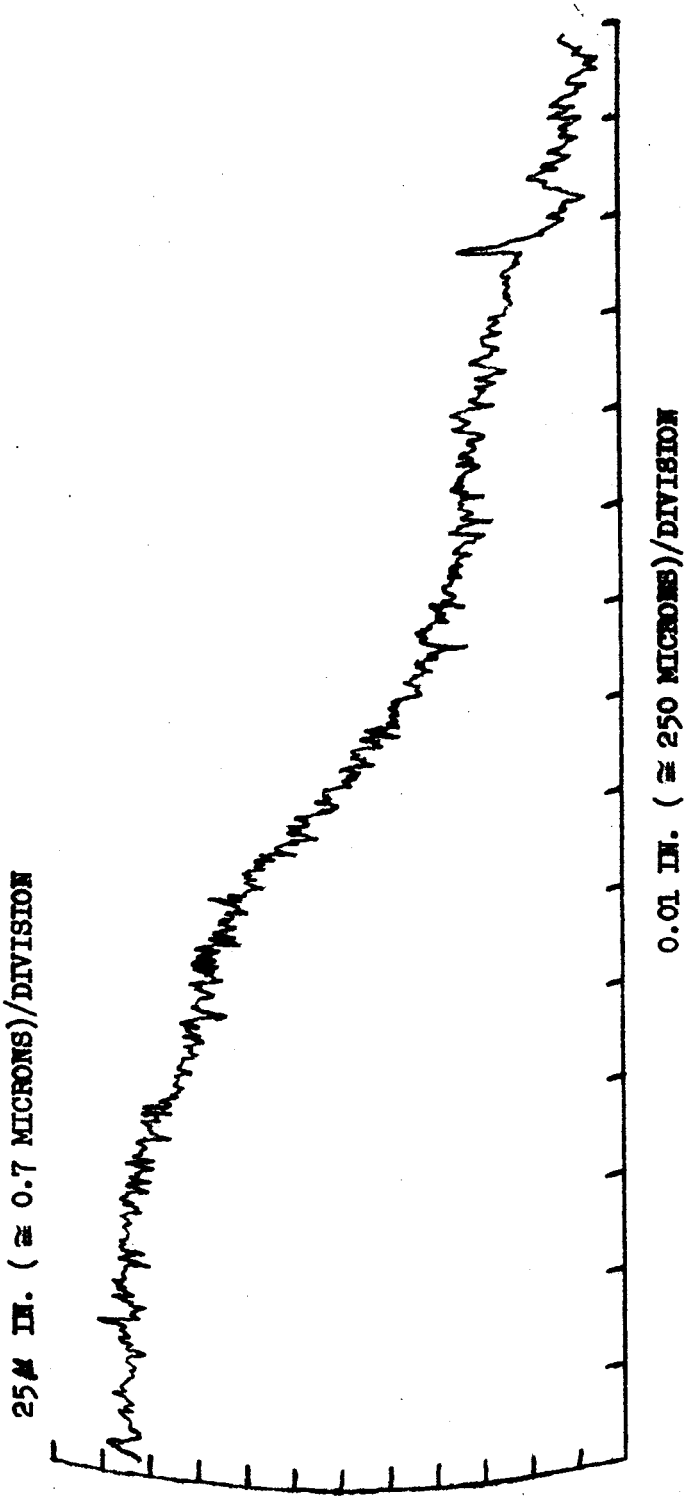

⊙ Aluminum (2024)/Orthoclase (001)
⊡ Magnesium (AZ31B)/Orthoclase (001)
◇ Nickel/Orthoclase (001)
T ≅ 23°C
Adhesion Force, mg.
Load Force, gm.
10^3
10^2
10
1
10^-1
10^-2
1
10
10^2
10^3

Figure 2

ALUMINUM
(2024)

SURFACE ROUGHNESS

Figure 3



MAGNESIUM
(AZ31B)

SURFACE ROUGHNESS

Figure 4

NICKEL

SURFACE ROUGHNESS

Figure 5