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# Adhesion of Materials in a Space Environment

## A status report

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**Summary**

*This survey is based on a symposium held jointly by ASTM-ASLE, May 1967 at Toronto. The present knowledge on the adhesion of metals and the cleavage of silicates is briefly reviewed, followed by a synopsis on the concept of low friction surfaces and recent advances in the testing of friction couples in a simulated space environment.*

### Introduction

The OECD-group of experts on wear of engineering materials has formulated several information projects [1]. One of these topics is: „The correlation between adhesion and frictional properties of materials". This theme was considered of particular interest in regard to the advances made in space research. The chairman of the group, R. L. Johnson, together with D. H. Buckley, has written a survey recently which covers the fundamentals of lubrication and wear in a high, a so-called „hard" vacuum [2]. The adhesion aspect of this new technology was discussed at another recent meeting, organized by ASTM and ASLE at Toronto. An attempt will be made in the following survey to summarize the main points of interest discussed at this symposium [3]. Related aspects formed the subject of later discussions held at several sections of the annual ASLE meetings [4].

### Adhesion

The term „adhesion" is used in the technology of joining operations to describe the formation of bonds between solids; this field has been surveyed elsewhere [5]. In friction studies cold welding of metals, but also of other materials, is frequently experienced. This type of „adhesion" leads to high friction and rapid wear. The aim of adhesion studies in mechanical engineering therefore is to find conditions of *low adhesion* in friction couples. Already of importance in conventional engineering, adhesion becomes a major obstacle in space engines, when „naked" metal surfaces touch. After nearly twenty years of space engineering research, the theoretical and experimental work can be subdivided in a number of topics, which will be discussed now.

### The surface of solids

The interaction of gases, vapours and liquids with solid surfaces is studied in many laboratories. The conventional textbook [6] approach, as Adamson called it, is sufficiently advanced to describe adsorption and adhesion in terms of thermodynamics and of molecular structures. However, such theoretical models are of little help to the student of friction. Ideal surfaces can be well defined in thermodynamics, but real ones, in the hard reality of engineering, are unsufficiently defined unless special precautions are taken in their preparation. R. W. Roberts has analyzed present concepts of clean surfaces [3,7]. A clean surface is taken to be one free of all foreign atoms, except a fraction of a single monolayer of impurities. There are then two general approaches to prepare such surfaces. One can either deposit material on a substrate, or clean a solid surface by removing impurities from it. Table 1 summarizes the principal techniques. It is seen that we are caught between two extremes: the preparation of large areas, which, however, are badly defined, or of well-defined but very small areas. In order to study the very first step of an adhesive process, one probably has to fall back on such extremely small surface areas. There are at present two methods, which hold some promise: „LEED" and „field ion microscopy".
LEED = „Low Energy Electron Diffraction" permits to follow the changes caused by the absorption of reactive gases in the surface of a single crystal. J. W. May illustrated the phenomena observed for a number of metal surfaces after contact with oxygen [3]. Similar examples have recently

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Table 1

Preparation of Clean Surfaces [7]

| Technique | Surface area ($cm^2$) | Range of materials | Surface „analysis" |
|---|---|---|---|
| Evaporation | $10^3$ | very wide | difficult |
| Sputtering | $10^3$ | very wide | difficult |
| Vapour plating | 10 | limited | difficult |
| High temperature heating | 1 | wide | easy |
| Chemical reaction | 1 | limited | difficult |
| Ion bombardment | 1 | wide | easy |
| Cleavage | 0.1 | limited | moderate |
| Field ion desorption | $10^{-10}$ | wide | easy |

been obtained in Cambridge [8]. Valuable as the method is, it does not seem very suitable for combination with friction measurements.

By far the most exciting advances were reported [3] by E. W. Müller and O. Nishikawa on the directed observation of adhesion effects with the field ion microscope. This technique has been mostly used to describe structural details observed on the surface of refractory metals. Figure 1 illustrates a typical observation from TNO laboratories. In order to expand the range of application, Müller and Nishikawa developed new techniques. An image of high quality was obtained at *low* temperatures by adding small quantities of hydrogen to the ionizing helium gas. The undisturbed surface structure of lower melting metals such as nickel, cobalt, carbon steel and iron were produced with the aid of this improved imaging field. The next step was the construction of a very delicate lever system. The radius of the metal tip, from which the image is produced, is only between 200 and 1500 Å. This tip was brought into contact with the surface of another material, by controlling the loads in the microgram range. The mechanisms could be operated either in air or in the camera of the microscope. Thus it became possible to observe the first steps of mechanical deformation on touch, leading to adhesion in a space environment. The significance of this advance is that the deformation of clean crystal faces on mechanical loading can now be studied in detail, and furthermore that the influence of surface impurities can be followed by controlling the atmosphere of the chamber. In the meantime, however, less sophisticated instrumentation remains useful in broadening our understanding of adhesion phenomena.

Table 2

*Autohesion of Copper*
„Hard" Vacuum at 20° C
Normal load 50-100 g

| UHV (torr) | Cu-crystal 99,999 % Cu | Coefficient of autohesion | Author | Year | ref. |
|---|---|---|---|---|---|
| $5.10^{-10}$ (?) | polycrystalline | 0.07 | Ham | 1963 | a |
| $1.10^{-5}$ | „ | < 0.1 | Burton | 1966 | b |
| $7.10^{-10}$ | „ | 1.0 | | | |
| $10^{-11}$ | „ | 1.00 | Buckley | 1967 | 2,3 |
| | single crystals | | | | |
| $10^{-11}$ | (100) | 1.02 | | | |
| $10^{-11}$ | (110) | 0.62 | | | |
| $10^{-11}$ | (111) | 0.30 | | | |

a. J. L. Ham, ASLE Trans. **6** (1963) 20

b. R. D. Brown and R. A. Burton, ASME preprint No. 66-Lub 3 (October 1966)

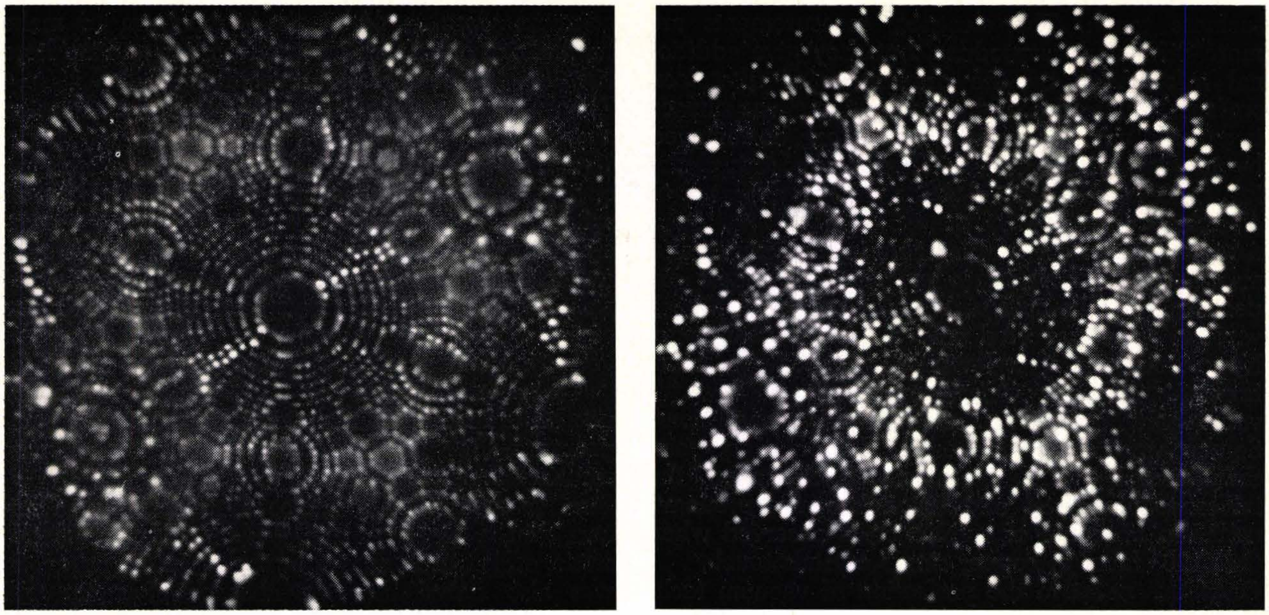

*Fig. 1 Application of Field Ion Microscopy. Pure molybdenum crystal at 4 x $10^{-9}$ torr. Left: clean; right: contaminated (Metal Research Institute TNO).*

**The adhesion of metals**

In any adhesion study a clear distinction should be made [5] between *autohesion,* the adhesion of two pieces of identical material, and *heterohesion,* the adhesion between different materials. Obviously, the second problem is much more complex than the first. With the advanced experimental facilities now available, autohesion studies can serve to follow the influence of impurities and that of crystal structure. D. V. Keller has produced much material in recent years on the strength of adhesion between metals in a hard vacuum [3]. Figure 2 illustrates the type of experience gained from such studies. The system consisted of crossed silver wires, adhesion was measured mechanically and at the same time the decrease in contact resistance was followed. The mechanical tests are in essence fracture experiments [3], and the highest values correspond to the tensile strength of the bulk material. It is obvious from Figure 2 that the absorption of gases inhibits the formation of an adhesive bond (under low loads). Only above $10^{-8}$ torr a strong bond is formed when the silver

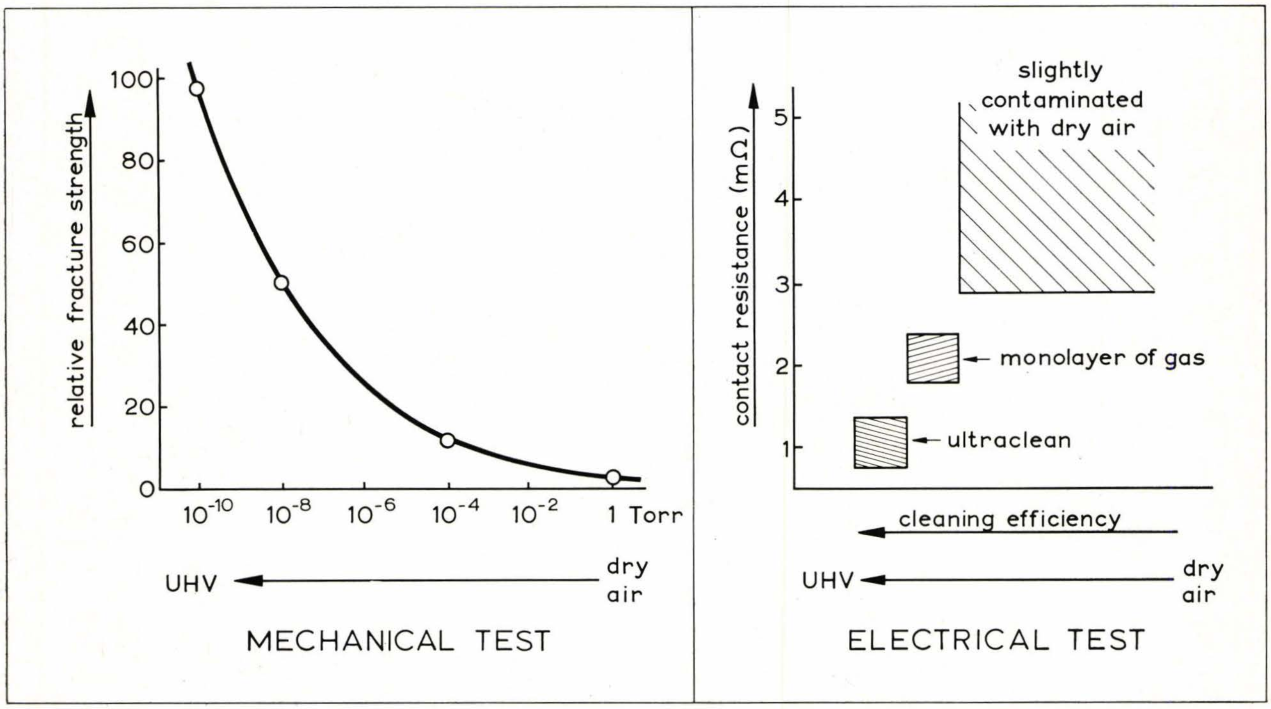


*Fig. 2 Autohesion of Pure Silver. Condensed presentation of results obtained by D. V. Keller [3] with crossed wire arrangement at light normal loading.*

surfaces touch. The electrical measurements are more difficult to reproduce, but they give additional information on the production of *ultraclean* surfaces. Even in a high vacuum the silver surface is covered with a gas layer, which can be removed by ion bombardment. The efficiency of this cleaning process is indicated by a further decrease in electrical resistance.

Another metal that is obtainable in high purity and also as single crystals is copper. The oldest experiments on the cold welding of copper in a high vacuum were reported less than five years ago. Now experimental techniques have been so much improved already, that much higher strength values are obtainable under similar conditions. Some data are summarized in Table 2. It is seen that, once the atmosphere is well controlled, the influence of crystal orientation becomes predominant. An optimum of autohesion is observed when the (100) faces of copper single crystals touch.

Once this point in the perfection of vacuum techniques is reached, many different experiments can be designed in order to learn more about the nature of adhesive bonding. H. Conraad and L. A. Rice [3] have treated the autohesion of polycrystalline copper as a cold welding problem. Time effects are expressed as „torr-seconds". It then becomes possible to compare the quality of bonds prepared either by loading at a moderate vacuum during a long time or by loading only briefly but in a hard vacuum. Flow processes under compression, the elimination of voids, influence of grain size, etc. are observed. In short, one can now follow the conventional path of metallurgical studies, but under simulated space conditions. The solid phase joining of very pure iron was reported by T. E. Johnson [3]. This process needs in fact a little heat (200-300°C) to produce good joints rapidly, but otherwise no serious difficulties are experienced in the „cold welding" of iron. Among the higher melting metals Keller studied the autohesion of titanium and of molybdenum. As might be expected, crystal structure and impurities influence the formation of a strong bond more in the cold welding of refractory metals than in that of the soft silver.

The next step then was taken by Johnson and Buckley, [2,3] NASA, who performed friction experiments in a vacuum of $10^{-11}$ torr. Indeed a high coefficient of friction was always accompanied by a high coefficient of adhesion. The systematic study of dissimilar metal couples led to the preparation of alloys with a fairly low coefficient of friction. Although heterohesion is a more complex phenomenon than autohesion, the qualitative understanding of the parameters involved in adhesion and friction was sufficiently advanced to predict the properties of certain „low friction" alloys.

## Cleavage and adhesion of silicate surfaces

The lunar surface probably consists of silicates. The question how these minerals will behave on contact with a spacecraft and with automatically controlled „spades" is asked with some urgency; attempts were made at the symposium to make predictions [3]. It will be convenient to state here first the European research position.

The cleavage of mica has been studied in Russian and British laboratories during the last thirty years. There is full agreement that the forces resisting cleavage are mainly electrostatic charges, unless sufficient water vapour is present to reduce the electrostatic effect. In that case the contribution made by Van der Waals' bonding can be measured and calculated [9]. In fact, these cleavage studies of mica are almost unique in demonstrating the validity of theoretical calculations. How much of this work is well-known in the United States could not be judged from the contributions to the symposium — in any case this earlier work was not mentioned explicitly.

Further theoretical evidence that cleavage of minerals in a high vacuum is governed by electrostatic effects was presented by J. J. Grossman. It was also stated that the absence of water will decrease the efficiency of creep and plastic flow in silicates. Consequently fracturing of silicates could be expected to be less laminar and more brittle in a space vacuum. Experimental work by J. A. Ryan and M. B. Baker [3] showed that fresh surfaces, formed in air and placed later in a vacuum, behave quite differently from the ultraclean surfaces produced by direct cleavage in a hard vacuum. These results confirm the earlier experiments performed by Miss Bailey in Cambridge [9]. One of the discussors remarked that a small quantity of water — brought along by a spacecraft — would be quite sufficient to alter the mechanical properties of the minerals on the moon surface, a conjecture no doubt of interest to space-atmosphere-control-commissions.

## Low friction surfaces

While the analysis of undesirable adhesion effects is still in progress, several approaches are followed to find friction couples with intrinsically low adhesion under static and dynamic conditions. The unique properties of some hexagonal metals were already mentioned [2]. Solid lubricants, gold and its alloys and plastics are also studied in this context.

Haltner had previously shown that molybdenite ($MoS_2$) has better friction properties in a hard vacuum than in an ordinary vacuum. In this respect $MoS_2$ differs notably from graphite. The latter lubricant functions properly only in the presence of gases or vapour. At this conference [3] Haltner reported the behaviour of synthetic $WS_2$. It appears that a hard vacuum has no beneficial effect on $WS_2$, quite on the contrary, small quantities of condensable gases reduce the friction. The position of this solid lubricant seems to be somewhat intermediate between that of $MoS_2$ and graphite. These results, but also subsequent discussions, made it clear that we are not yet sufficiently advanced to explain friction losses in terms of structural relationships. Many hundreds of compounds have been tested as solid lubricants. Some years ago the impression was gained that comparatively simple relations between a structure of chalcogenides and friction exist. It appears now, from the work of Haltner and others, that factors such as crystal imperfections and impurities are of great influence on the adhesion and friction of laminar solids. As these parameters are not yet sufficiently controlled in the synthesis of layer lattice compounds, the position of natural $MoS_2$ remains still unique.

The engineering applications of $MoS_2$ bonded films in high vacuum systems have recently been surveyed [10]. A committee of ASLE studies ways and means to promote the knowledge of bonded solid lubricants. One of the stumbling-blocks seems to be communication with designers. The application of a bonded film is indicated only by a „thin line" in design. Obviously the clearance of the system will depend on the thickness of this lubricant film. The designer, used to the behaviour of liquid lubricants, finds it difficult to make a choice between the original thickness of the film and that of the run-in system. His doubts are aggravated by the fact that no simple laboratory test, corresponding with conditions found in service, does yet exist.

Electro-deposited metal coatings are of great practical importance, *e.g.* in connector contacts for space systems. Reliability depends on the quality of the plated precious metal. M. Antler discussed at this symposium [3] his latest results with electro-deposits of pure gold. The main problem is here to avoid the initiation of adhesive wear, which leads to a rapid destruction of the contact. It appears that the metallographic properties of the substrate determines the structure of the deposit. There are other factors, such as roughness, pore formation, etc. On the whole, the understanding of frictional properties and adhesion with thin, plated metal surfaces seems still to be in the first, exploratory phase, notwithstanding the fact that millions of such contacts are used every month by the computer industry. More complicated combinations consisting of a porous soft metal matrix, containing a dispersed refractory metal, were discussed also at Toronto [11]. Such films may find some use in lubrication at higher temperatures and in the presence of air.

The application of plastics, more in particular PTFE (Teflon) in vacuum systems, is well-known. There are two principal limitations, the low heat conductivity and the mechanical weakness of this polymer. Both can be overcome to some degree by applying PTFE as a fibre- or metal-filled composite. Performance of such materials, however, frequently depends on the previous history, in other words, on the quality of the dispersion of fibres and the absence of microscopic voids. An entirely new approach was reported at the Toronto ASLE meeting [12] by the staff of the United States. Navy Marine engineering laboratory.

The purpose of their study was te develop a piston seal for high pressure air compressors (for obvious reasons the presence of atomized lubricating oil particles must be avoided in such constructions). This marine engineering group applied the technology of filament winding to the design of metal- or glass fibre sleeve seals filled with Teflon. While this development is not yet completed, the report makes it clear that closely controlled production methods can lead here to low friction machine elements with a good wear life.

## Testing

Space technology has led to improved equipment for testing the behaviour of friction couples; progress has been made along the following four lines.

The basic unit is equipment for producing an ultra high vacuum. The „ultra" limit has been gradually pushed from $10^{-8}$ torr to $10^{-11}$ torr. Less visible but even more remarkable is the fact that nowadays such equipment can function reliably after assembly. In the earlier period of laboratory activities one mostly found engineers busy searching for a leak or for an unexpected source of vapours.

The next step then is the design of instruments for more or less static loading to evaluate mechanical adhesion under normal forces. Of course results depend largely on the choice of conditions. If loading means a gentle touch between surfaces, elastic deformation will primarily take place. In that case, studied extensively by Keller, interference of monolayers of gases or other residual impurities can be measured. However, engineers will be inclined to use more drastic conditions, leading to plastic deformation of the substrate in the friction couple. Then solubility, crystal structure and other metallographic parameters come also into play.

The third approach is that of dynamic friction testing. Here not only the vacuum equipment has been improved, but also the design of friction couples. These are frequently used over a wide range of temperatures, loads and speeds. To make a comparison between different pairs of materials possible, geometry of design should be the same under a variety of conditions. Apparently small changes can, in fact, mean a considerable progress in friction testing. Until recently the Falex tester was the most widely used cheap instrument. Here a rotating pin is pressed between V-blocks. The efficiency of a solid lubricant is evaluated by the onset of plastic deformation of the pin (a condition not likely to be experienced in well-designed machines).

A more reasonably designed tester has recently come into use in the United States. It is called a „dual rub shoe test configuration" and the principle is shown in Figure 3. The geometry is comparable to the pin and ring arrangement of the classical Timken tester and the stability is that of the Falex tester. As rubbed off, lubricant particles fall down and cannot heal the film, conditions of rapid destruction can be readily evaluated. Most of this new development has appeared in reports scarcely known in Europe. An unfortunate factor is that standard committees in the United States are in the process of transforming the Falex tester into an official standard. Perhaps Europeans, lagging behind a bit, can avoid this mistake and

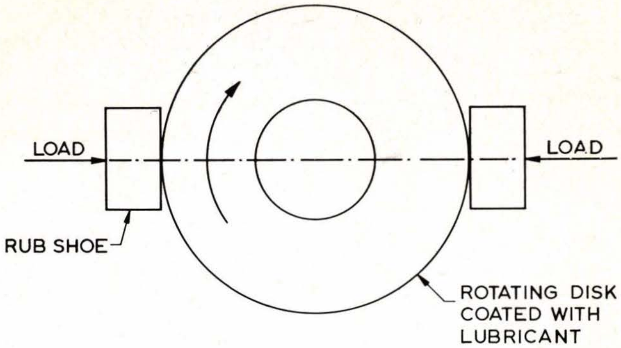


*Fig. 3. The Dual Rub Shoe Test Configuration.*
*A new testing device developed in USA for the evaluation of solid lubricants.*

concentrate in future on adapting the „dual rub shoe test" or even more advanced test configurations.

The fourth approach in testing is the actual evaluation of engineering materials sufficiently accurate to base *design specifications* on space simulation testing. As might be expected, results from different laboratories are still conflicting (J. B. Rittenhouse) [3]. The ultimate but costly solution is therefore to equip small experimental spacecraft with friction couples and observe their behaviour over periods of months or years in instrumented, non-manned spaceflights. The design of some of these testing devices is astonishingly simple, comparable to the testing of an ordinary screen-wiper of cars. It appeared from the lively discussion on this point that sufficient experience is already accumulating to predict reliability in actual spaceflight over periods of a few years. The component testing for a nuclear reactor operating in space was crowned by the start up and operation in orbit of SNAP 10 A (L. G. Kellogg) [3].

## Future developments

The European observer, not specifically engaged in space research, will ask now whether any obvious gap exists between the European and the North-American research effort. This possibility, I believe, can be denied. As indicated above, much of the theoretical background actually stems from Europe. The same can be said of the origin of the powerful analytical tools used in surface analysis: microprobe analysis, field ion microscopy and the most recent major instrument, the stereoscan electron microscope. Equipment to produce an ultra-high vacuum is commercially available. The screening of materials done on a gigantic scale in the United States needs no repetition and advantage can be taken in future of the new alloys developed already to this purpose [2].

The real problem is not the „ultra-high" vacuum, but the „ultra-clean" surface. We are only at the beginning of an era when the friction properties of metal, ceramic and plastic coatings will be defined in terms of structural parameters. The new instruments help us to understand the significance of impurities, voids and other structural irregularities, caused by the adsorption of vapours. At present several research centres in Europe study the production of hard surface layers for friction couples in instruments, e.g. watches. There is no indication that this type of research is lagging behind similar developments in other parts of the world.

## Acknowledgement

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