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ADHESION AND COHESION OF METALS

Final Technical Report

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TABLE OF CONTENTS

TABLE OF CONTENTS (Cont'd)

ABSTRACT

Apparatus used in previous work for studies of cohesion (bonding of similar metals) was modified to permit studies of adhesion (bonding of dissimilar metals) including indexing devices permitting tests on eight sample pairs for a single vacuum pumpdown operation were built and are described.

Modification of the spool piece which houses the apparatus was required to permit various cleaning techniques to be used.

Adhesion studies require a surface cleaning operation not required in the previous cohesion work in which specimens were fractured and rejoined. Two ion guns and a wire brushing device were constructed for such cleaning. The ion gun design is discussed herein, and the reasons for using guns rather than simple glow discharge are discussed. The ion gun was constructed and tested for cleaning ability using beam current and cohesion testing as a measure of design success. While the gun electrically performed well, no cohesion was obtained in several test series at an ambient 10^{-8} torr even after long cleaning times.

Rotary wire brush cleaning tests at various temperatures between 25° and 140°C gave cohesion between copper specimen pairs up to about one half the applied force. Adhesion tests between dissimilar metal pairs resulted frequently in significant adhesion, up to two thirds the yield stress of the softer material.

Some of the parameters found to influence adhesion between metal specimens are temperature, interface, stress, degree of cleanliness, and flow stress as measured by Brinell hardness number.

FORWARD

This is the final report on work performed under Amendment Number 1 of Contract NASw-734 between the National Research Corporation and the National Aeronautics and Space Administration.

The general objective of the program was to obtain additional data on the conditions under which metals and alloys of engineering importance would adhere to one another with sufficient tenacity to hinder their relative motion or separation of components of mechanical or electrical devices used in space exploration. Typical devices include bearings, hinges, solenoids, valves, etc.

The materials specified in the contract for study were:

Copper	
Copper Beryllium Alloy	440C Stainless Steel
SAE 1018 Steel	Titanium
SAE 4140 Steel	Coin Silver

Each of the above alloys was to be tested in both the fully hardened and fully annealed conditions. A major variable in the program was hardness although the effect of ion bombardment cleaning was compared to the effect of wire brushing (abrasion) as a cleaning method.

In addition to the materials specified above, these additional metals were tested:

Commercially Pure Aluminum . 1100 Alloy

Aluminum Alloy . . . . 2024-0 and T6

Aluminum Alloy . . . . 6061-0 and T4

Pure Soft Gold

This work was a continuation of work on the cohesion of copper to copper and of steel to steel performed under NASA Contract NASr-48.

Major contributors to this project were F. J. Brock, Program Director; John L. Ham, Research Associate; Paul L. Vitkus, Project Engineer; Lloyd R. Allen, Research Associate and Project Engineer; and George Reichenbach, Associate Professor and Consultant, Massachusetts Institute of Technology.

INTRODUCTION

Research on the tendency of clean metals to adhere in high vacuum has been carried on at National Research Corporation for the past five years under NASA Contracts NASr-48 and NASw-734 and under this extension as NASw-734,Amendment Number 1.

The general objective has been to obtain additional information as to the conditions under which metals and alloys of engineering importance for space applications will adhere to one another with sufficient tenacity to hinder the relative motion or subsequent separation of components of mechanical and electrical devices used in space exploration. Such devices include bearings, solenoids, valves, slip rings, mating flanges, conical rendezvous mating surfaces,etc.

Even a small amount of adhesion would be disastrous in many cases on a space vehicle. Power on such craft is ordinarily very limited and mechanical components must work freely and even a few ounces adhesion force may cause failure. There is, therefore, an important requirement for quantitative data on adhesion.

Techniques were developed in previous programs for evaluating the cohesion of metals at various temperatures by repeatedly fracturing and rejoining notched tensile specimens in ultrahigh vacuum. Two types of apparatus were used: 1) a differential expansion device, and 2) a screw drive device. The latter was found to be the better. The maximum cohesion obtained at room temperature was about 65% for copper, 19% for 1018 steel, and 0% for hardened 52100 steel. Time in contact appears to be an important factor for copper at 200°C and above. Both 1018 steel and 52100 steel were "self-cleaning" at 500°C, the former showing repeated readings near 100% cohesion, and the latter increasing in per cent cohesion with each successive break. Except for steel at 500°C, and copper at 350°C and 400°C, cohesion dropped on each successive test.

The apparatus was then modified to permit specimen positioning and thus the testing of eight specimen pairs with a single pump-down of the vacuum system. Arrangements were made for wire brushing in vacuum and/or for ion bombardment just before joining[(1)]. Both flat-faced and chisel-edged specimens were used, the rectangular faces or chisel edges being crossed. All tests were at room temperature and at pressures between 10^{-8} and 10^{-9} torr. Three runs (24 tests) were made with flat specimens and one run (8 tests) with chisel-edge specimens. Cohesion occurred only between flat-faced, soft copper specimens, wire brushed in vacuum. The cohesive force varied from 8 to 120 lbs. after a compressive force of 2000 lbs., and appeared to depend primarily on the thoroughness of wire brushing.

These results and the apparatus used are described in detail in the Final Report (Contract NASw-734), dated November 27, 1963, covering the period 3/1/63 to 11/1/63 and entitled, "Investigation

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(1) National Research Corporation, "Investigation of Adhesion and Cohesion of Metals in Ultrahigh Vacuum". First Annual Report, March 1, 1963, May 30, 1963, John L. Ham, June 12, 1963 (NASA Contract NASw-734), (NRC Project No. 81-1-0101).

of Adhesion and Cohesion of Metals in Ultrahigh Vacuum".

As indicated in that report, a number of important questions remained unanswered. In order to answer these, the contract was extended to cover the following tasks:

Task H Determine adhesion of pairs of similar metal specimens and cohesion of dissimilar pairs by measuring the force required to separate each pair after cleaning and joining by compressive loads in a vacuum or the order of 10^{-8} to 10^{-9}.

Task I Conduct tests after either wire brushing or cleaning by ion bombardment. Perform experiments with ion guns including various voltages, currents, and gas species, including xenon and hydrogen, to perfect this cleaning technique and develop methods of monitoring surface cleanliness.

Task J Test various material combinations at room temperature and at temperature of 200°C or above using the best xenon ion cleaning technique developed. Test a few combinations after proton bombardment cleaning if time permits.

APPARATUS

The apparatus consists of a stainless steel vacuum chamber with the accessories necessary to join and separate small metal specimens in ultrahigh vacuum and to measure the forces involved. The major components were developed and used in previous programs.

Figure 1 shows the loading and force measuring devices which communicate with the inside apparatus through a flexible metal bellows. Beneath the dome in Figure 1 hangs the apparatus shown in Figures 2 and 3. Sixteen specimens (eight pairs) can be mounted on the wheels shown in Figures 2 and 3, and positioned to bring different material combinations together or to expose a given surface for cleaning.

A twelve-inch long spool piece is now located between the dome and the bowl of Figure 1.

The spool pieces have windows for observing the specimens, and a bellows manipulator for positioning a motor-driven wire brush between specimens to clean the mating surfaces just before joining.

The entire assembly is mounted on a standard NRC ultrahigh vacuum pumping system with a 10-inch diffusion pump (HS10-4200) and a standard NRC Chevron-type liquid nitrogen trap. Concentric "O"-Rings cooled by a circulating refrigerant are used at the joints between the large flanges.

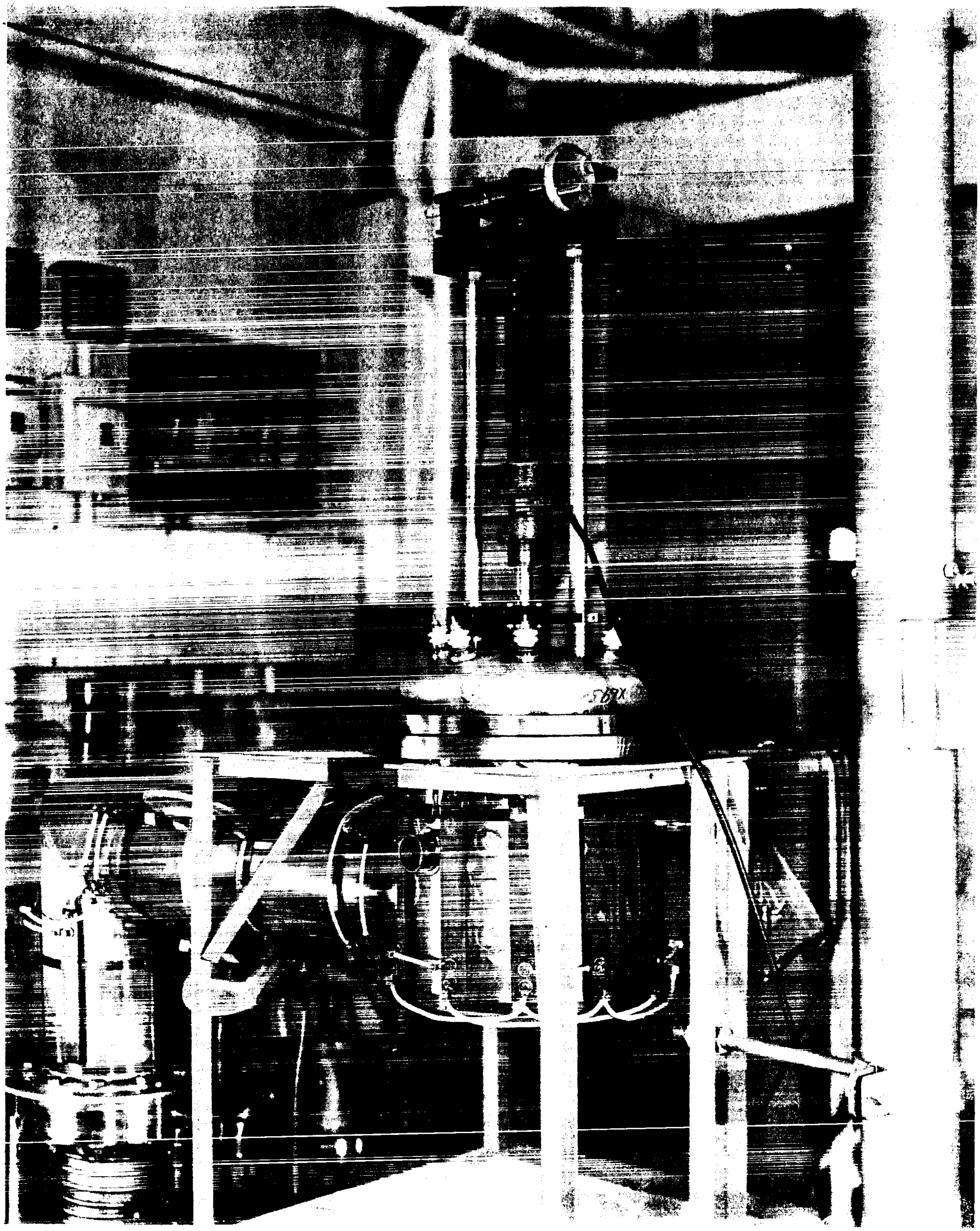

FIGURE 1

SCREW DRIVE COHESION TESTING APPARATUS (OUTSIDE)

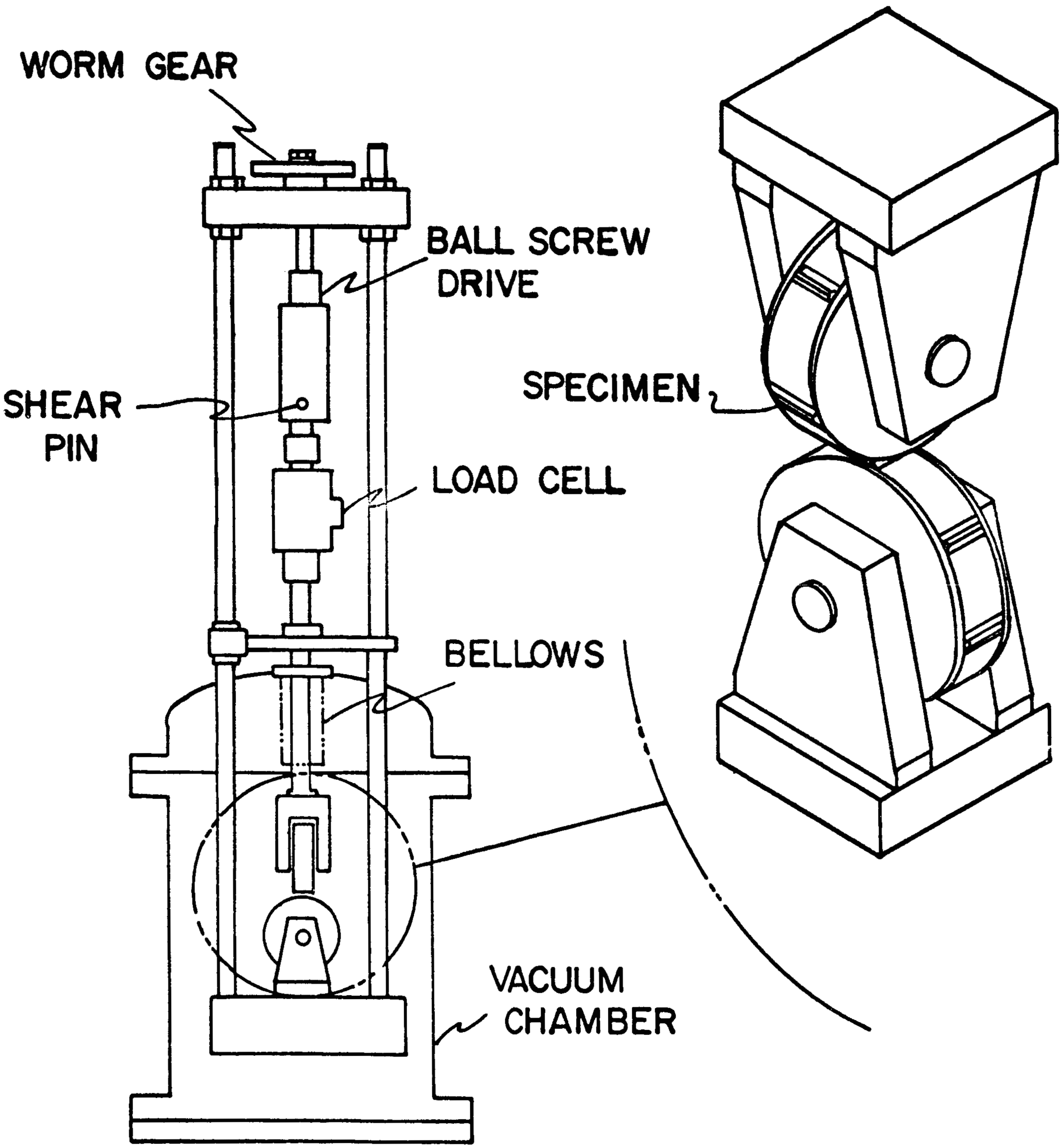

FIGURE 2 - COHESION TEST APPARATUS

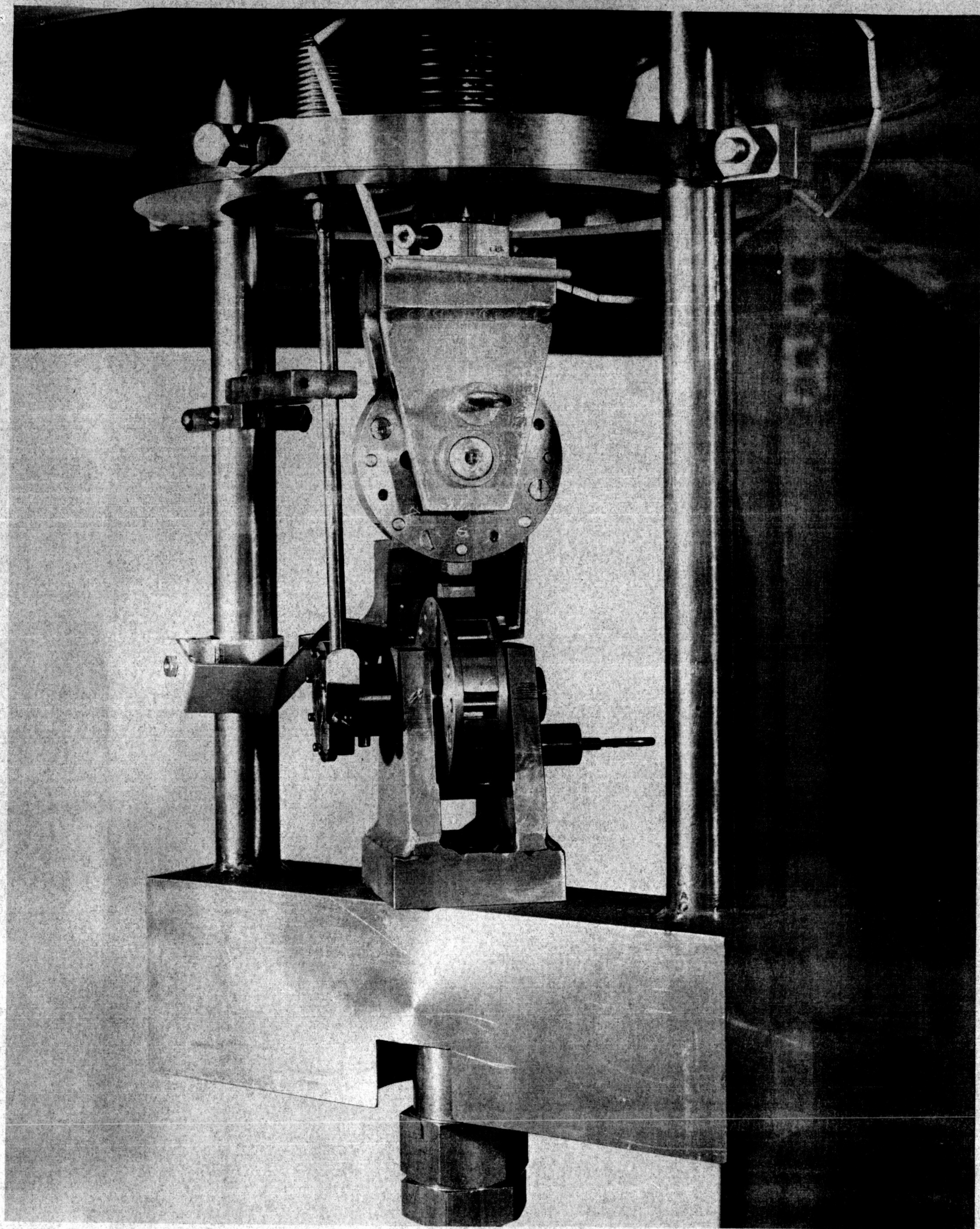

FIGURE 3

INDEXING SAMPLE HOLDER

SPECIMENS

The type of specimen used is shown in Figure 4. Eight of these fit into each wheel of Figures 2 and 3. They are held by hardened pins which permit either specimen of a pair to adjust itself parallel to the other. This minimizes the possibility of misalignment during either compression or tension.

The face widths of the specimens are adjusted to permit a few thousandths of an inch face depression with compressive loads of 2000 to 3000 lbs. The surfaces are prepared by mounting three specimens at a time in a special jig and polishing with successively finer grades of alundum or emery paper. The three specimens form a triangle in the polishing jig and a flat plat is used under the abrasive paper. This insures specimen flatness. A slightly different type of polishing jig was used for preparing the two flat faces of 90° chisel type specimens. Just before mounting in the test wheel, each specimen is given one rub on dry No. 000 paper.

The hard specimens of O.F.H.C. copper and 1018 steel were made directly from cold drawn 3/4 inch diameter bar stock. The soft copper and soft 1018 steel specimens were made from the same stock after annealing. The 3/4 inch diameter titanium stock could be obtained only in the condition referred to commercially as "annealed". Actually this condition is quite hard so that by annealing a portion of such a bar both hard and soft materials were obtained. The Cu-Be alloy (No. 25) was machined to size from 3/4 inch diameter bar stock in the solution treated condition and some of the specimens hardened by the standard aging treatment prior to finishing on the No. 000

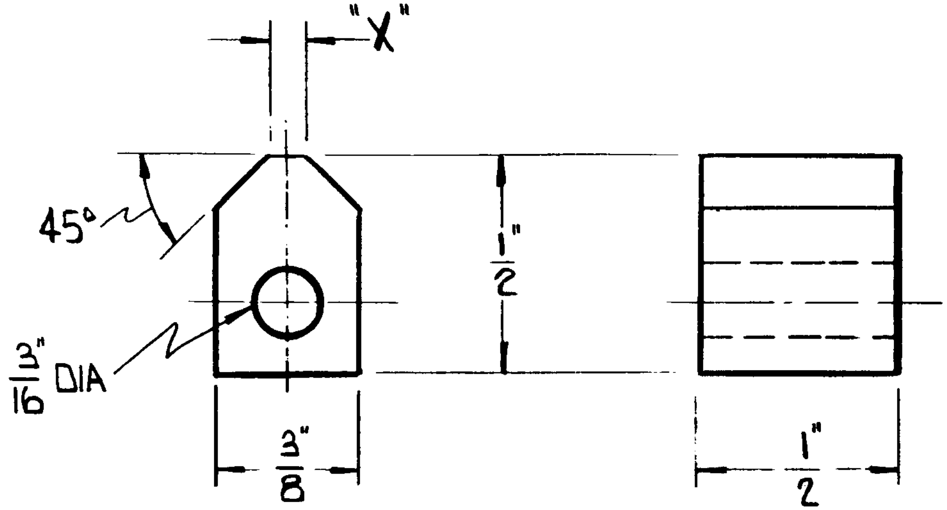

FIGURE 4 - Adhesion Specimen

paper and beveling by grinding. The 440C stainless steel and the 4140 steel specimen were machined to size from soft bar stock except for .010 inches to be removed from the face of the hard specimens by surface grinding to remove decarburized metal prior to finishing on the No. 000 paper. The 4140 steel was drawn to about 50 Rockwell C. The 440C steel and Cu-Be was left at maximum hardness.

The pure aluminum specimens were prepared from 1100-H18 and after annealing a bar section at 650°F and cooling to room temperature. The aluminum alloy 2024 was purchased in the T4 condition and a selected piece was annealed at 775°F for three hours while the 6061-T6 was used in both the T6 condition and after a three hour anneal at 775°F.

The gold specimens referred to were pieces of 0.015" gold foil silver brazed to an annealed copper specimen. It must therefore be considered that while the gold is truly in the annealed condition that its apparent penetration hardness is not that of a solid gold specimen and allowance should be made in interpreting the data.

Table 1. gives a summary of the material specifications of the samples used in this research program and their Brinell hardness number. This criterion for hardness was used as it is felt that it best correlates with flow stress. Figure 4, page 9 , gives the physical size and shape of the test specimens while Table II gives the value of the X dimension shown in Figure 4 for the face width and the number of specimens required for the test program.

TABLE I

SPECIMEN MATERIALS, ANALYSIS AND HARDNESS

MATERIAL	COMPOSITION (%)		HARDNESS BHN	
Copper (OFHC)	99 + Cu	Cold Worked	65	
		Annealed	55	1000°F Air Cool
Be-Cu	Cu+2Be, 0.35Ni	Heat Treated	415	
		Annealed	86	
Coin Silver	Cu+90Ag	Cold Worked	62	
SAE 1018	Fe+0.19±% Carbon	Cold Worked	176	
		Annealed	63	Furnace Cool from 1400°F
SAE 440C	Fe+0.10C+17Cr+0.6Mo	Heated Treated	601	Oil Quench 1500°F Temperature 400°F
		Annealed	285	Air Cool from 1400°F
SAE 4140	Fe+0.40C, 1Mn, 1Cr, 0.2Mo	Heat Treated	578	Oil Quench 1500°F Temperature 450°F
		Annealed	235	Furnace Cool from 1500°F
1100 Aluminum	99+% Aluminum	Cold Worked	44	
		Annealed	22	
2024	Al+4.5Cu, 0.6Mn, 1.5Mg	Hardened	120	T4 Condition at Start T86 at End
		Annealed	56	Then 775°F-Air Cool
6061	Al+0.25Cu, 0.6Si, 1.0Mg, 0.25Cr	Hardened	86	T6 Condition
		Annealed	52	2 hr. 775°F-Air Cool - Not fully soft
Ti-A40	0.2C maximum	Cold Worked	189	
		Annealed	167	1 hr. 850°F-Air Cool
Au	99.95 Gold Foil 0.015 Inches Thick	Annealed	(25 est.)	Used as Foil Soldered to Soft Copper Blocks

TABLE II

NUMBER OF SPECIMENS REQUIRED
OF EACH MATERIAL AND FACE WIDTH

MATERIAL	NOMINAL FACE WIDTH (INCHES)	NUMBER REQUIRED
Soft Copper	.25	35
Hard Copper	.25	2
Soft 1018	.18	4
	.25	2
	.18	16
Hard 1018	.25	2
	.13	5
Soft 440C	.25	2
	.13	5
Hard 440C	.25	2
	.13	2
	.08	4
Soft 4140	.25	2
	.10	5
Hard 4140	.25	2
	.10	5
Soft Cu-Be	.25	2
	.15	5
Hard Cu-Be	.25	2
	.15	2
	.10	4
Soft Ti	.25	2
	.22	5
Hard Ti	.25	2
	.22	2
	.14	4
Soft Coin Silver	.25	2
Soft 1100-Al	.25	5
Hard 1100-Al	.25	2
Soft 2024-Al	.25	3
Hard 2024-Al	.25	10
Soft 6061-Al	.25	3
Hard 6061-Al	.25	8
Soft Gold Foil	.25	7

OPERATION OF TEST APPARATUS

During a typical operation the system was baked out at +200°C under vacuum for times ranging up to four days, using a number of tungsten-quartz lamps inside the vacuum vessel as a heat source. When the pressure had stabilized at 10^{-8} torr or so, a refrigeration system was turned on to cool the vessel walls and gaskets. The pressure then fell to the 10^{-9} range, typically 4 to 5 x 10^{-9} at the end of 24 hours.

To maintain the temperature of the specimens, the heat input to the lamps was controlled and the temperature was measured by thermocouples. The individual specimens were wire brushed for measured times, typically between 30 seconds and 5 minutes (as will be discussed later). During brushing of the Cu specimens, pressure bursts occurred which yielded pressure transients up to 5 x 10^{-8} torr. This effect was not observed on 1018 steel or Ti metal specimens, nor was it connected with the operation of the motor itself when not brushing. The specimens were then positioned to face each other and the pair to be tested were forced into contact and loaded to a constant initial 2000 pound load. (Relaxation and creep later reduced this load as low as 1920 lbs. in one case.) The time between cleaning and contact was measured and found to be reasonably constant at about 25 to 35 seconds at 4 to 6 x 10^{-9} torr. This product, 2 x 10^{-9} torr minutes, is below the 1/10th monolayer formation time and thus assures contact between clean metal surfaces. The load was left on for 15 minutes for each pair and the measured breakaway load was then recorded on pulling the specimens apart using a Sanborn recorder and SR-4 strain gauges in a Baldwin load cell as shown in Figure 2.

Cleaning by Ion Bombardment

Ion bombardment cleaning is frequently accomplished in small glass systems by simply initiating an electrical discharge between the sample and an adjacent positive electrode with the whole system filled with the gas to be ionized at a pressure of about 10^{-2} to 10^{-1} torr. However, this requires isolation of the system from the diffusion pumps and in the system in use for the cohesion studies valving adds considerable complexity. There is also uncertainty as to the actual composition of the gas over extended periods of time, even though the relatively large volume of gas required is initially pure. Finally a finite time would be required to reduce the system pressure from 10^{-2} torr to the 10^{-8} to 10^{-9} torr range desired for the experimental environment and recontamination of surfaces might occur during this pumpdown period. Therefore, it was decided to use an ion gun which can direct a beam of ions at the specimen surface only thus permitting the system to operate at low pressures even during the cleaning period.

The ion gun can maintain the pressure of 10^{-2} torr within the gun cavity required to maintain the discharge. An ion beam is emitted through an orifice and is directed to the target specimen. Some idea of the pressure which can be maintained in the system can be obtained by comparing the amount of gas typically effusing from the gun with the pumping speed of the system. A gun with an internal pressure of 10^{-2} torr and an orifice of 0.010" diameter would effuse approximately 6×10^{-5} torr liters per second of air. If we assume a net pumping speed of 1000 liters per second at the exit from the test system then the system pressure would be 6 x 10^{-8} torr with the ion gun operating. The advantage of the ion gun over a local discharge to the specimen in a large chamber is that the ratio of the impingement rate of the desired species (Xe^+, for example) to that of undesired species (O_2, O^+, CO, CO^+, CO^{++}, etc.) is larger and more precisely known. Furthermore, the energy and species (Xe^+ or Xe^{++}) of the impinging inert gas ions is more controllable. The number of impinging nonionized inert gas atoms may be greater or less than in a local discharge but this is of little consequence.

It is important to control the energy of the impinging ions since ions even of the inert gases penetrate into the metal lattice if they impinge with too great an energy. This may be shown by curves of "sticking coefficient" versus voltage obtained by measuring the amount of inert gas given off on subsequent heating in vacuum. Apparently the ions actually form substitutional solid solutions with metals and diffuse out on subsequent heating according to the usual diffusion laws. However, the sticking coefficient curves indicate zero sticking (penetration) below a critical voltage for each type of ion. For example, argon ions penetrate into tungsten above 150 volts but Xe ions require 200 volts.

To remove inert gases from metals requires relatively high temperatures since the activation energies of diffusion appear to be comparable to that for self-diffusion of the metal itself. The apparatus used was not suitable for high temperature specimen outgassing, and since the magnitude of the effect of inert gas ions in solid solution on hardness and strain hardening coefficient was not known it seemed advisable to stay below the critical sticking coefficient voltages if possible. Data in the literature indicated that good "cleaning" (ratio of sputtered atoms or ions to impinging ions) was possible below these voltages but there was some question as to how efficiently the ion guns could be made to work at such low voltage. Obviously, the heavier the impinging ion the better, since a larger percentage of the energy was given up near the surface and higher voltage could be used without penetration. Therefore, xenon as well as argon was used. Actually, it was almost impossible to clean metal surfaces in vacuum at low temperature without changing the physical characteristics of the metal surface somewhat. Abrasion, even by a sharp wire brush undoubtedly work hardens the surface to some extent.

Ion Bombardment Cleaning Apparatus

Two cold cathode-type ion guns were assembled utilizing flanges and parts available from previous work together with new components. A stack of six barium ferrite magnets which had been previously tested for field uniformity and strength were used to provide the magnetic field for the ion source. A Granville-Phillips variable leak was attached to each gun for accurate control of gas flow. See Figure 5.

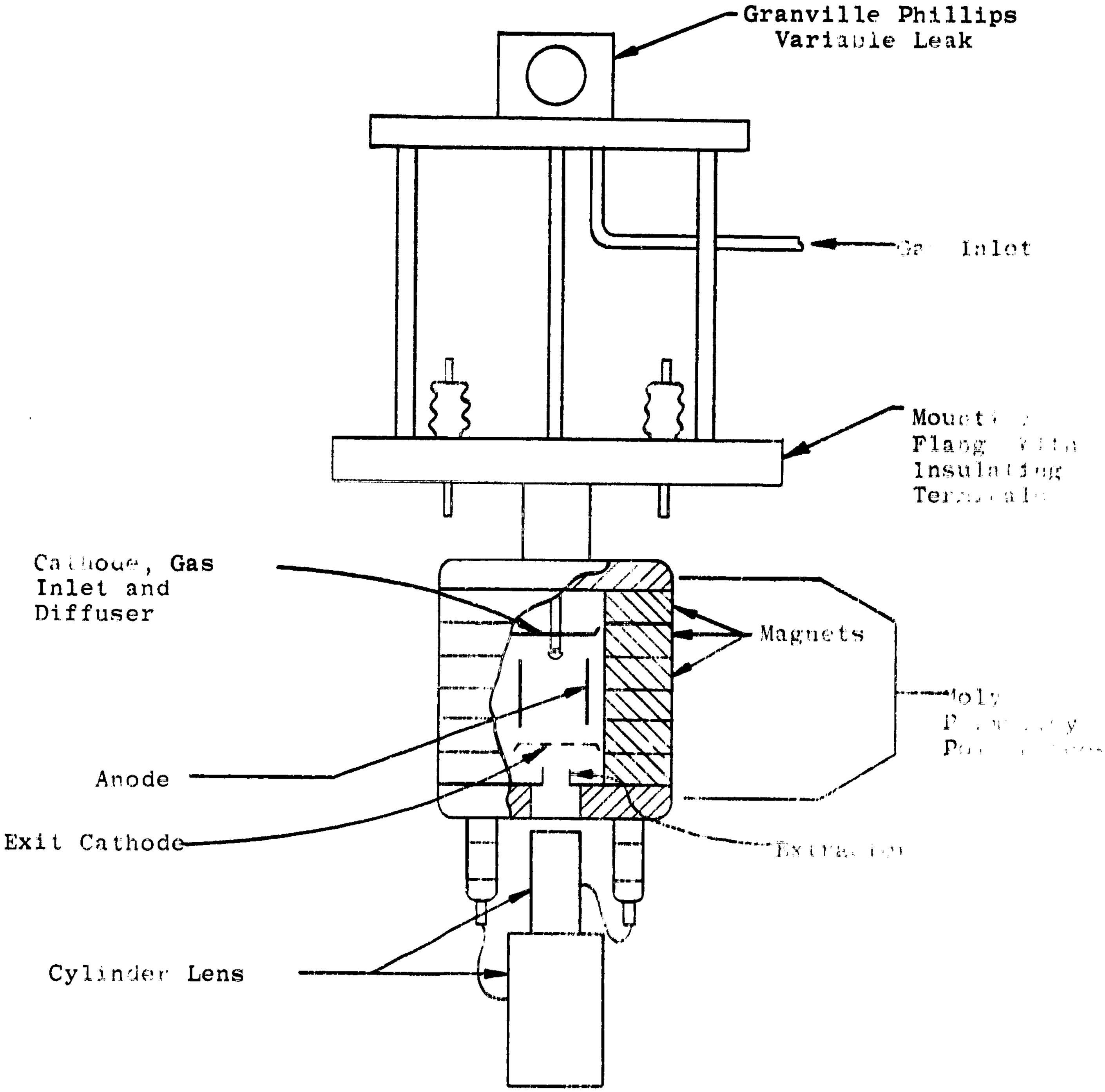

FIGURE [illegible]

ION GUN ASSEMBLY

The gun consists of two main sections, the cold cathode ion source, and the electrostatic lens system. The ion source is a modified magnetron gauge[(1)]. Auxiliary cathodes are omitted. One cathode contains a gas inlet tube and diffuser, the other containing an exit aperture for ions. Moly-permalloy pole pieces are used at each end of the magnet to obtain a uniform field distribution in the ionization volume. The focusing system consists of a conventional electrostatic cylinder lens ($\frac{D_2}{D_1} = 1.87$). The magnet pole piece nearest the exit cathode acts as an ion extractor.

The ion gun as orginally assembled contained a 1/8" diameter aperture in the exit cathode and in the extractor. An ion collector target was constructed and coated with a sodium silicate solution and placed a distance of approximately 20 cm from the ion source. This gun configuration was found to have low sensitivity at the target approximately 2 ma/torr under optimum conditions.

To improve focusing and to increase sensitivity at the target, the following modifications were made:

1. The exit cathode aperture was enlarged to 1/4" diameter.

2. The extractor aperture was also enlarged to 1/4" and an extension added to decrease the distance from the extractor to the aperture cathode.

3. A diverging screen lens[(2)] was constructed from the exit cathode and extractor by addition of a 94% transparent tungsten

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(1) Redhead, P. A. Can. J. Phys., 37, (1959), 1260

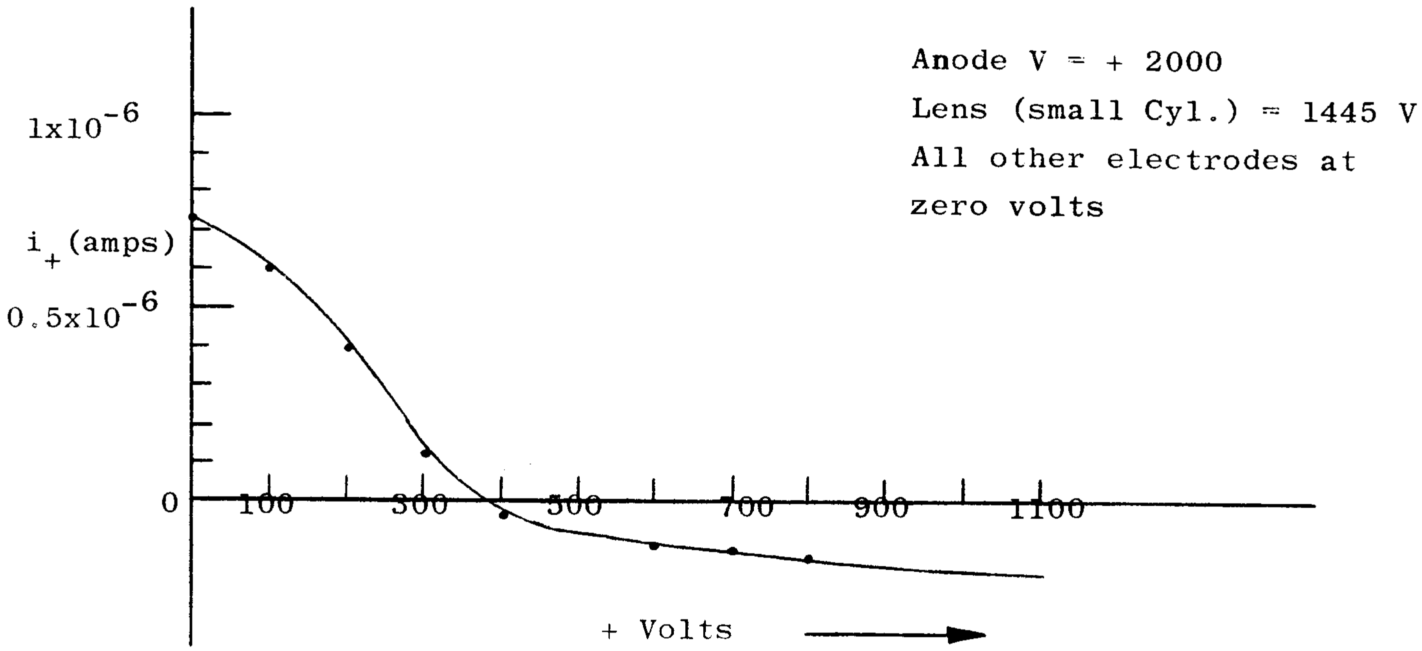
(2) Klemperer, O., Electron Optics, Cambridge University Press, 1953, p. 72

screen over the exit cathode aperture.

The above modifications increased the sensitivity of the device to 100 ma/torr at the target, and focusing could be achieved by variation of electrode voltages. It was necessary to deflect the electron component of the beam extracted from the ion source by a small permanent magnet in order to study the ion beam. Only by this procedure could a very faint ion induced glow be seen on the phosphor.

The effect of variation of the focusing voltage could also be seen by observing the exited residual gas glow produced by the ion beam.

Ion energy distribution curves were plotted for various voltage configurations. Typical ion energy distribution curves for a focused beam are shown in Figure 6. From these curves it can be seen that the ion energy is highly dependent on anode voltage. Typically, the highest ion energy is roughly one fifth the anode voltage. An electron deflecting magnet was not used in obtaining the energy distribution curves. Purified argon was used throughout on preliminary tests.



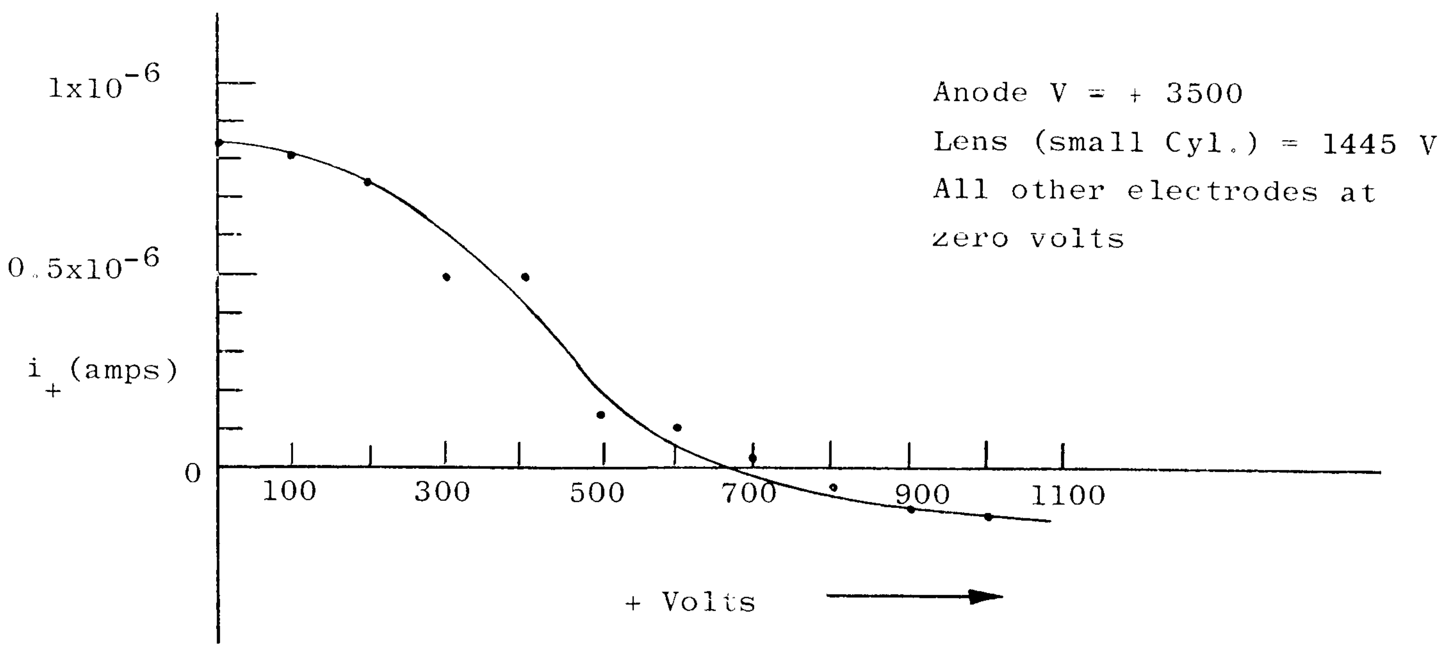


FIGURE 6
ION ENERGY DISTRIBUTION

EXPERIMENTAL PROGRAM

Ion Bombardment Cleaning

Following the initial tests, two ion guns were set up on the adhesion apparatus. Four pairs of OHFC copper samples were installed in the positioning mechanism. Two phosphor screens in the form of an open frame were placed approximately 1/8" in front of the samples to be bombarded. The phosphor screens were connected to insulated terminals so that ion current could be measured. To aid in beam positioning, four deflection plates were added to each gun. A schematic representation of the system is shown in Figure 7.

By visually observing the ion beam induced glow on the phosphor screen frame and by measuring the frame total current resulting from varying the voltage on the deflecting plates and the lens the beam could be focused and positioned on the sample surface satisfactorily. After adjusting the argon flow, two samples were bombarded for 70 hours. Pressure during bombardment was in the 10^{-6} torr range.

An attempt at cohesion was made with negative results. Inspection of the samples after removal showed both to be coated and discolored. This result was traced to a malfunction in the diffusion pump baffle liquid nitrogen level control circuit. The pump baffle had warmed and diffusion pump oil was evaporated into the system, coating the samples with oil. The oil was subsequently decomposed by ion bombardment.

In a second series of tests, three pairs of OFHC Cu samples were

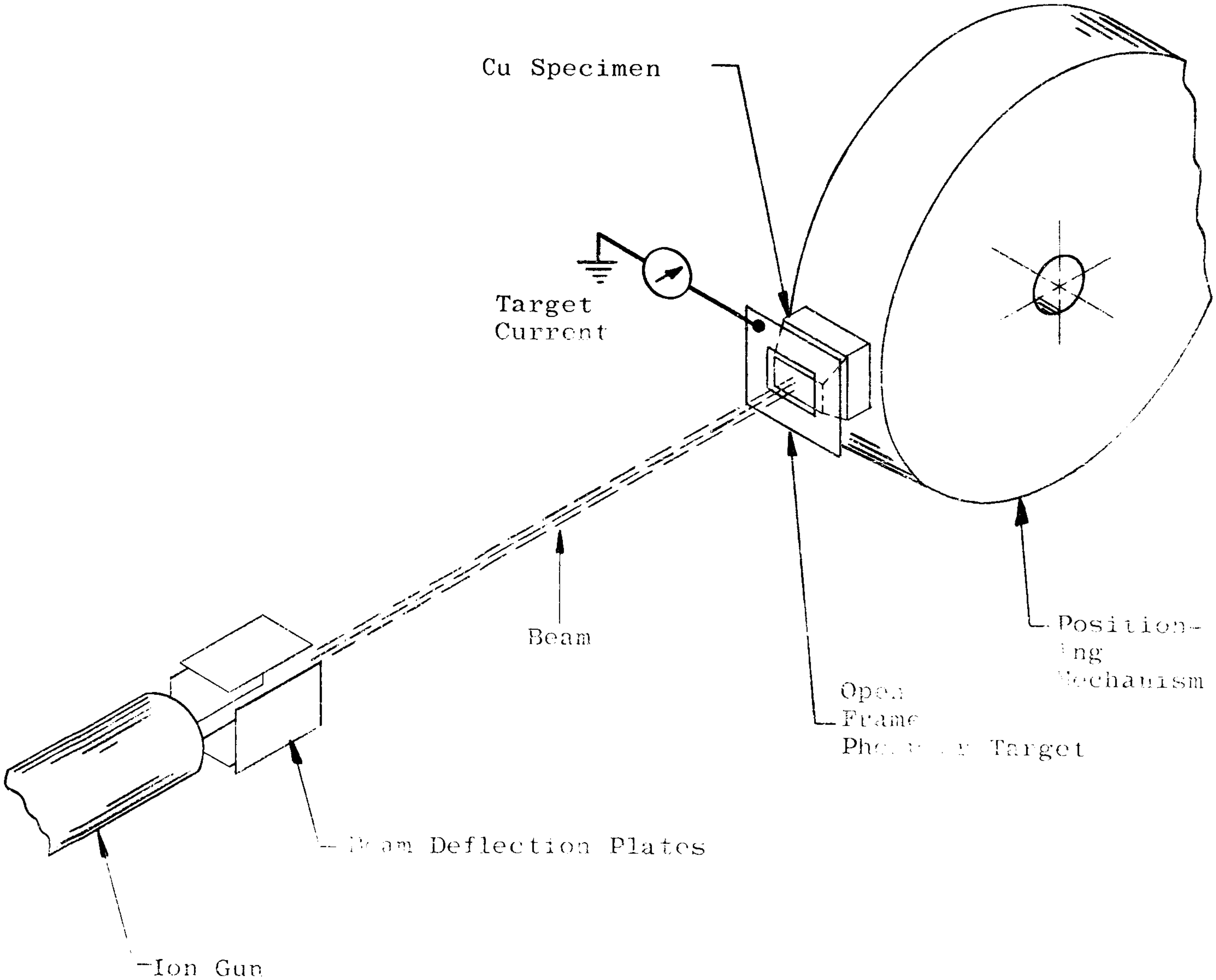


FIGURE 7

ION GUN AND SPECIMEN

SCHEMATIC REPRESENTATION

placed into the positioning mechanism in the adhesion-cohesion vacuum system for testing. A glass vial of spectroscopically pure xenon was attached to both guns by means of glass tubing. The vacuum system was cold trapped and baked by means of internal tungsten lamps. The glass tubulation between the xenon bottle and the guns was evacuated through the vacuum system and flamed to degas while the system was baking.

Prior to insertion in the vacuum system, all OFHC Cu samples were chemically etched in a solution of 80% distilled water, 10% hydrogen peroxide and 10% formic acid to remove organic surface films.

The first set of samples was bombarded 15 minutes at an ion current density of 10^{14} ions/cm^{2}sec. and then placed in contact and loaded to 2000 lbs. Approximately 2.5 minutes elapsed between termination of ion bombardment and surface to surface contact. The second set of samples was bombarded for 30 minutes at an ion current density of 10^{14} ions/cm^{2}sec. before applying the interface force. No adhesion occurred in either case. When the third set of samples was positioned for bombardment, the positioning mechanism locked and the test had to be discontinued.

Further work on adhesion testing after ion bombardment cleaning was discontinued for the following reasons:

1. The sample positioning mechanism is necessarily rather complex in order to successively position sixteen samples properly under vacuum conditions. A number of operations are involved in

moving the samples from position to position, some of which require rather exact termination. The accumulated time to move a sample from the cleaning position in front of the ion gun to the test position is therefore rather large (two to three minutes) since the sample must pass successively through six separate positions. Since the system ultimate pressure with all samples installed was of the order of 10^{-8} torr, several monolayers of gas can be adsorbed on the test surfaces before the samples come in contact.

2. Excessive expenditure of time and money would be necessary to modify the system for effective ion bombardment cleaning, and would preclude obtaining additional data using the wire brush method.

Throughout the above tests, both guns performed satisfactorily. Ion current density was sufficiently high to remove several hundred monolayers of material during the shortest bombardment time. The negative results on adhesion of the samples are attributed to the recontamination of the surfaces.

Wire Brush Cleaning

Modification of the vacuum vessel to permit translational motion of a motor driven wire brush for cleanup of the adhesion specimens was performed.

Rotary wire brushes about 1/2" diameter and 3" long and using a two strand twisted wire for the shaft were used for the cleaning operation. The wire bristles were made either of cold drawn mild steel, or cold drawn 304 stainless steel. The brush shaft was held in a collet which in turn was fastened to a small hysteresis motor (8-10 watts) operating at 3600 rpm (free speed). The motor was modified by opening the ends near the bearings to the vacuum chamber and by lubricating the bearings with dry MoS_2. These motors have operated as long as 100 hours in the 10^{-9} torr range in the past and no difficulty during the cleaning periods in this program was encountered.

The entire motor was encased circumferentially in a press fitted aluminum shell which was cooled by water flowing through the motor support rod. This rod was fastened through a flexible metal bellows to the outside of the vacuum vessel so that the brush (motor-rod) could be moved to clean both top and bottom specimens and to move the brush away from the test zone during cohesion tests.

A number of observations on brush usage and wear seem appropriate at this time. During the brushing operation on copper specimens there invariably occurred a pressure rise to $7 \cdot 10^{-8}$ torr, up almost a decade or so from the prior vacuum. When the motor and brush were turned on but not brushing a metal sample this pressure rise was not found, indicating that the pressure was in fact connected with the metal cleaning operation not with the operation as the motor alone.

The failure pattern of the brushes was interesting in that the mild steel bristle brush would last about 10-14 specimens before there were too few bristles to clean, while the stainless brush was debristled after 2 to 3 specimens regardless of material being brushed. Failure of the bristles in both cases meant a brittle fracture of the bristle wire at the point near the twisted wire shaft where the bristles were pinched. Almost always there was copper welded to the outer end of the bristle (away from the handle) put there during brushing a copper specimen.

Experimental Program

The first series of specimens to be tested in cohesion were four soft copper and four soft 1018 steel flat face specimens tested in pairs. The contact area approximated 1/16 square inches. For this series of tests each pair of the specimens were wire brushed for thirty seconds immediately before contacting. During the brushing operation, the chamber pressure rose to about $7 \cdot 10^{-8}$ torr from $5 \cdot 10^{-9}$ torr. After brushing, all specimens were loaded fifteen minutes at 2000 lbs., thus leaving only material and test temperature as the variables.

The second series of tests were made using soft copper specimens of the same 1/16 square inch face contact area. The deliberate variables in this test were temperature (25°, 100°, and 125°C) and wire brushing times between 0 and 5 minutes. In addition to the copper-to-copper cohesion tests, three adhesion pairs were included to evaluate the possible problems to be expected in later experiments.

These pairs were:

Cu - Be Cu (soft)
Cu - 1018 (soft)
Cu - Ti (soft)

and all were tested at room temperature. (These specimens were cleaned last at which time the wire brush was rather worn and the measured adhesion is probably lower than it would have been with a new brush.)

The third series of cohesion test specimens were copper-to-copper cohesion pairs and included cold worked as well as annealed copper. The variables for these tests were temperature, wire brushing time and material hardness.

It should be noted that for these tests the load was fixed at 2000 lbs., the compression holding time was 15 minutes and the ambient pressure was in all cases held between 2 and $8 \cdot 10^{-9}$ torr.

The fourth series of specimens were for the purpose of testing cohesion between similar metals at otherwise constant loads and cleaning times. One variation to be noted in the test series is a change in the composition of the wire brush bristles from stainless steel (304) to cold drawn mild steel.

The fifth series of tests were on adhesion between dissimilar metals using carbon steel wire brush cleaning at a constant load and somewhat shortened brushing time.

The three remaining test groups evaluated the effect of vacuum and loading on commercial aluminum alloys as well as the adhesion of gold to various materials. The variables were alloy composition, hardness; and to a lesser extent on the gold series, cleaning time.

Table 5, page 32, gives in chart form the sample pairs which were originally proposed and those pairs actually tested. In most cases where no adhesion was found between an annealed metal and a copper specimen the corresponding test using the same metals in the hardened condition was omitted. The specimens of aluminum alloys and the gold pairs were not required by the contract but were felt to be valuable either in an engineering or scientific sense.

The tabular presentation of the test data are given, overleaf in Tables III and IV with appropriate notations in the remarks column as to the variable being observed in the test series. A discussion of the test results follows in the next section of this report.

TABLE III

PRELIMINARY COHESION RUNS-SYSTEM CHECKOUT

No.	Test Pair	Test Load	Time of Loading (min)	Cleaning Time (min)	Test Vacuum (torr)	Test Temp. (°C)	Cohesion Load (lb)
1	Cu-Cu s-s*	2000	15	20	5.10^{-9}	25	8
2	Cu-Cu s-s	2000	15	20	6.10^{-9}	125	300
3	1018-1018 s-s	2000	15	20	5.10^{-9}	25	0
4	1018-1018 s-s	2000	15	30	6.10^{-9}	125	0

s* soft, annealed material

TABLE IV

ADHESION-COHESION TEST SERIES

No.	Test Pair	Test Load Lbs.	Loading Time Minutes	Cleaning Time Minutes	Test Vacuum Torr	Test Temp. °C	Test Area	Breakaway Load Lbs.	Remarks
5	Cu-Cu s-s*	2000	15	None	6.10^{-9}	125		0	
6	Cu-Cu s-s	2000	15	0.5	7.10^{-9}	125		240	Compares #2
7	Cu-Cu s-s	2000	15	0.5	6.10^{-9}	100		100	
8	Cu-Cu s-s	2000	15	0.5	6.10^{-9}	100		40	
9	Cu-Cu s-s	2000	15	5	7.10^{-9}	100		900	Note cleaning time compared to entry above.
10	Cu-BeCu s-s	2000	15	5	3.10^{-9}	25		25	
11	Cu-1018 s-s	2000	15	5	3.10^{-9}	25		190 ‡	Cu stuck to steel when pulled apart.
12	Cu-Ti s-s	2000	15	5	2.10^{-9}	25		50 ‡	
13	Cu-Cu s-s	2000	15	2	$1.2x10^{-9}$	25		300	
14	Cu-Cu s-s	2000	15	3	$1.3x10^{-9}$	25		220	
15	Cu-Cu h-h**	2000	15	2	$1.3x10^{-9}$	25		60	Hard copper
16	Cu-Cu h-h	2000	15	3	$5.8x10^{-9}$	100		250	Hard copper
17	Cu-Cu s-s	2000	15	2	$3.9x10^{-9}$	100		350	
18	Cu-Cu s-s	2500	15	2	$3.1x10^{-9}$	100		600	Note increased compression load
19	Cu-Cu s-s	2000	15	2	$5.8x10^{-9}$	140		600 ‡	
20	Cu-Cu s-s	2000	15	2	$5.0x10^{-9}$	140		600 ‡	
21	1018-1018 s-s	2000	15	3	$4.2.10^{-9}$	125	.060	0	
22	440C-440C s-s	2000	15	3	$4.0.10^{-9}$	125	.062	0	
23	Al-Al s-s	2000	15	3	$3.6.10^{-9}$	125	.055	320	Stainless steel brush.
24	CuBe-CuBe s-s	2000	15	3	$3.6.10^{-9}$	125	.023	0	Stainless steel brush.
25	CuBe-CuBe h-h	2000	15	3	$3.6.10^{-9}$	125	.058	0	Stainless steel brush.
26	Ti-Ti s-s	2000	15	3	$3.6.10^{-9}$	125	.039	0	Stainless steel brush.
27	Cu-Ti s-s	2000	15	3	$3.8.10^{-9}$	130	.062	48	Carbon steel brush.
28	[illegible]-Al s-s	2000	15	3	4.10^{-9}	130	.067	800	Carbon steel brush.
29	[illegible]-CuBe s-s	2000	15	3	4.10^{-9}	130	.042	400	Carbon steel brush.
30	Ti-Ti h-h	2000	15	3	4.10^{-9}	130	.023	0	Carbon steel brush.
31	CuBe-CuBe s-s	2000	15	2	4.10^{-9}	130	.023	0	Carbon steel brush.

s* soft, annealed material

h** full hardened by cold working

‡ brush worn and cleaning probably incomplete.

1 30 2

TABLE IV (Cont'd)

No.	Test Pair	Test Load Lbs.	Loading Time Minutes	Cleaning Time Minutes	Test Vacuum Torr	Test Temp. °C	Test Area	Breakaway Load Lbs.	Remarks
32	Cu-Cu h-s	2000	15	2	5.10^{-9}	125	.065	340	Carbon steel brush.
33	Cu-1018 s-h	2000	15	2	5.10^{-9}	125	.046	300	Carbon steel brush.
34	Cu-4140 s-h	2000	15	2	5.10^{-9}	125	.028	40	Carbon steel brush.
35	Cu-440C s-h	2000	15	2	$4.5.10^{-9}$	125	.024	40	Carbon steel brush.
36	Cu-CuBe s-h	2000	15	2	$4.5.10^{-9}$	125	.028	40	Carbon steel brush.
37	Cu-Ti s-h	2000	15	2	$4.5.10^{-9}$	125	.042	160	Carbon steel brush.
39	Al-Cu s-h	2000	15	2	$4.5.10^{-9}$	125	.044	300	Carbon steel brush.
40	CuBe-CuBe s-h	2000	15	2	$4.5.10^{-9}$	125	.025	0	Carbon steel brush.
41	2024-2024 h-h	1000	15	2	$3.1.10^{-9}$	125	.062	0	Differences in pressure in loading.
42	6061-6061 h-h	1000	15	2	$3.0.10^{-9}$	125	.065	40	Differences in pressure in loading.
43	6061-2024 h-h	2000	15	2	3.10^{-9}	125	.062	80	
44	2024-1018 h-s	2000	15	2	$3.5.10^{-9}$	125	.050	0	
45	6061-Cu h-s	2000	15	2	$3.2.10^{-9}$	125	.062	120	
46	6061-1018 s-s	2000	15	2	3.10^{-9}	125	.062	40	
47	6061-Ag s-s	2000	15	2	$3.5.10^{-9}$	125	.062	25	
48	6061-CuBe s-s	2000	15	2	3.10^{-9}	125	.029	0	
49	2024-2024 h-h	1000	15	2	$3.2.10^{-9}$	125	.062	0	Differences in pressure in loading.
50	6061-6061 h-h	1000	15	2	$2.8.10^{-9}$	125	.062	300	Differences in pressure in loading.
51	2024-2024 h-h	2000	15	2	3.10^{-9}	125	062	0	
52	Au-Au s-s	2000	15	0	$1.5.10^{-9}$	125	.073	0	No brush cleaning.
53	Au-Au s-s	2000	15	0.5	$1.5.10^{-9}$	125	.084	340	
54	Au-Cu s-s	2000	15	2	$1.8.10^{-9}$	125	.073	420	
55	Au-1018 s-s	2000	15	2	$2.5.10^{-9}$	125	.065	170	
56	Au-Al s-s	2000	15	2	$2.5.10^{-9}$	125	.090	800	
57	2024-2024 h-h	2000	15	2	$3.5.10^{-9}$	125	.0625	72	
58	6061-6061 h-h	2000	15	2	3.10^{-9}	125	.068	60	
59	2024-2024 s-s	2000	15	2	3.10^{-9}	125	.063	180	2024 annealed - 775°F 1/2 hour.

TABLE V

SAMPLE COMBINATIONS FOR 1964 COHESION TESTS

		Copper		1018		440C		4140		CuBe		Coin Ag		Ti		1100-Al	2024-Al		6061-Al		Al
		S	H	S	H	S	H	S	H	S	H	S	H	S	H	S	S	H	S	H	S
COPPER	S	X	X	X	X	X	X	X	X	X	X	O	O	X	X	X	X	X	X	X	X
	H		X																		
1018	S			X																	
STEEL	H				O																
440C	S					X															
STEEL	H						O														
4140	S							X													
STEEL	H								O												
CuBe	S									X	X										
COPPER	H										X										
COIN Ag	S											O									
Ag-Cu	H												O								
TITANIUM	S											X		X							
	H														X						
1100-0 ALUMINUM	S		X													X					
2024	S																X				
ALUMINUM	H																	X			
6061	S	X		X													X		X		
ALUMINUM	H																	X		X	
Au GOLD	S	X		X												X					X

X = Test performed

O = Test omitted

RESULTS OF TEST PROGRAM

Wire Brush Cleaning

The tabular data presented in the previous section of this report were analyzed for possible correlations which might allow prediction of the tendency for unknown pairs of metals to adhere to each other and for the effects of the experimental procedures upon the results obtained.

Certainly one of the most obvious questions lies in the validity of the wire brush cleaning procedure to obtain a clean surface, and if valid, under what experimental conditions. Figure 8 page 34, shows the effect of wire brushing time on the cohesion between two soft copper specimens at a constant load of 2000 lbs (100°C chamber temperature) for a fifteen minute compression cycle. The measured load to separate the specimens is recorded as it changes with the duration of the wire brushing times.

It may readily be observed that wire brushing does in fact increase cohesion between copper specimens in a vacuum Less clear however is the proper choice of time of cleaning. Although cohesion load seems to be increasing regularly up to five minutes cleaning time before becoming asymptotic, a five minute cleaning cycle for each of the eight specimen pairs proved impractical due to brush wear. It may be noted from the entries in the tabular run data that the last two or three specimens in a series of eight are usually marked "cleaning dubious" or "wire brush worn".

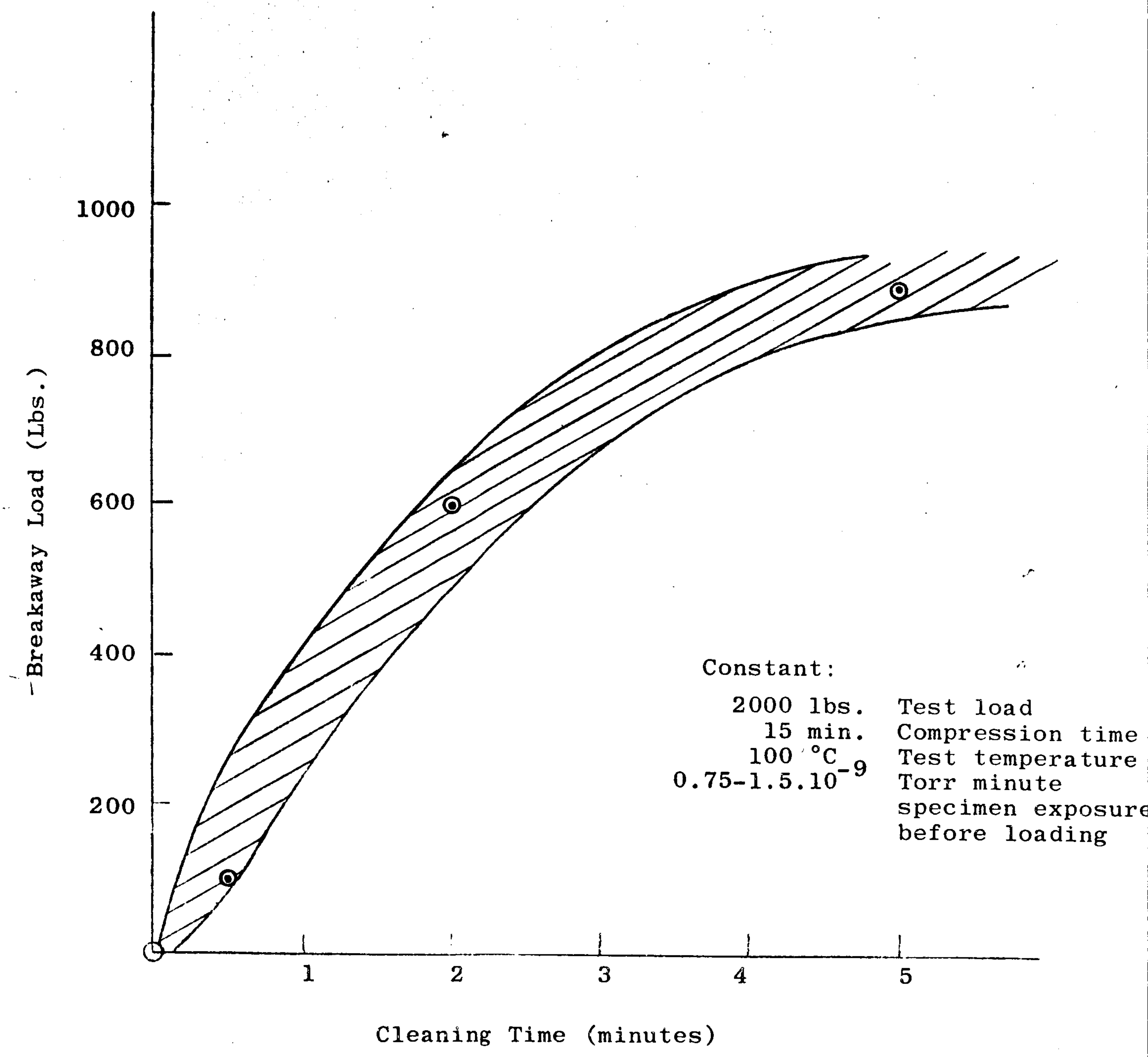


FIGURE 8

EFFECT OF WIRE BRUSHING TIME
ON COHESION OF SOFT COPPER

(Mild Steel Bristles)

Therefore a compromise cleaning time of two minutes was used in the great majority of the subsequent runs. This gave 30% or so less cohesion for copper specimen pairs and it is therefore entirely possible that some of the adhesion samples might have adhered provided longer cleaning cycles had been used.

Still another uncertainty lies in the grooving of the wire brushed surface found on all but the very hardest metal specimens. The cohesion/adhesion found may thus be influenced by mechanical interlocking of the metal surfaces although the grooves are at right angles on the two specimens.

Test Temperature

A possible variable in the cohesion/adhesion process is that of the specimen temperature. Intuitively one might expect from metallurgical considerations alone that the degree of adhesion would increase with increasing temperature, but simultaneously decrease due to the increased chemical reactivity at least until the temperature rises to the point where surface films are either absorbed or boiled off.

In normal metallurgical work with metals and alloys, one expects to find the onset of metallic softening prior to full recrystallization at about 0.30-0.35 Tm on the homologous temperature scale. This temperature is conventionally defined as the test temperature in Kelvin divided by the temperature in degrees Kelvin at the melting point of the metal. Thus, for pure Cu, one expects the onset of softening at about $T_H = 0.30$ (or 143°C). During this experimental series a number of separate tests were made to investigate cohesion between specimens of OFHC copper at various temperatures. These data, collected and plotted in Figure 9, page 37, show that as the temperature approaches the homologous temperature of 0.30 (143°C for Cu) the degree of adhesion of copper to copper rises extremely rapidly. It is further expected, although not proven, that the adhesion will become even stronger until full welding occurs at or near the recrystallization temperature, $T_H \sim 0.4$ or (272°C) for bulk copper.

Based upon these experimental results a standard test temperature of 125°C was chosen as a temperature which promotes but does not cause adhesion to such a level that all other effects are overshadowed.

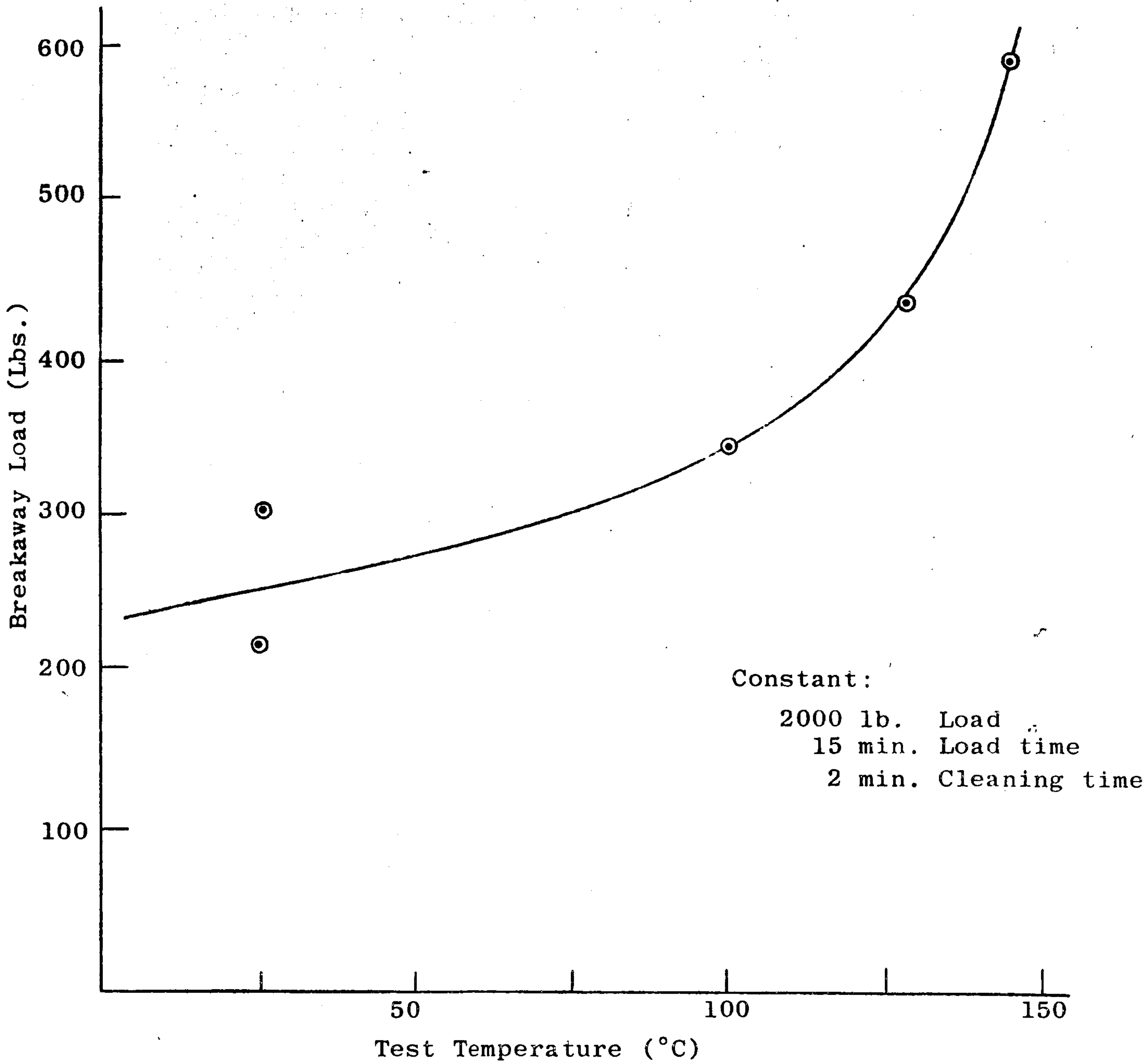


FIGURE 9

EFFECT OF TEST TEMPERATURE
ON COHESION OF SOFT COPPER

DISCUSSION

Cohesion between Similar Metals

It has been shown by John Ham[1] at the National Research Corporation and others that the major variables which determine the separating forces required after joining two similar metal specimens under vacuum conditions are:

1. Hardness
2. Prior cleanliness
3. Temperature
4. Compressive loading
5. Time of loading
6. Surface topography

This previous work emphasized pure copper and 1018 steel in most cases and leaves as an open question the effects of alloying, other metals, variations in hardness for similar compositions, etc.

The present work on the cohesion portion of the program was designed to study the sticking forces between a number of similar metals in both the hard and soft conditions. The metals tested are best reviewed at this time by referring back to Table I, page 11, of this report for a listing of the alloys tested and their gross properties. Table 5, already shown, gives in the diagonal line the cohesion tests performed. These tests were performed

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(1) Ham, J. L., Final Report, NASw-734, Nov. 27, 1963, Investigation of Adhesion and Cohesion of Metals in Ultrahigh Vacuum.

in the apparatus described earlier and cleaned by wire brushing, the specimen temperature was 125°C and the specimens were all loaded for 15 minutes at 2000 lbs force. After recording the breakapart force for the various specimens it was found that a correlation did in fact exist between cohesion in metals and hardness. Figure 10, page 40, shows the cohesive force measured plotted against the Brinell hardness number which is a measure of the compressive flow stress for the metals tested. The figure shows, albeit with scatter, that the cohesive force between similar metal specimens drops rapidly as the hardness increases, becoming zero or close thereto at BHN $\pm$ 150 regardless of the metal being tested. At the soft end of the hardness range cohesion force would seem to rise until plastic yielding of the gross specimen occurs to change the experimental conditions.

It should be remembered at this time, however, that the Brinell hardness number limit of 150 found in this work is a very specific number found as a result of a limited range of test variables and that generalization at this time is dangerous.

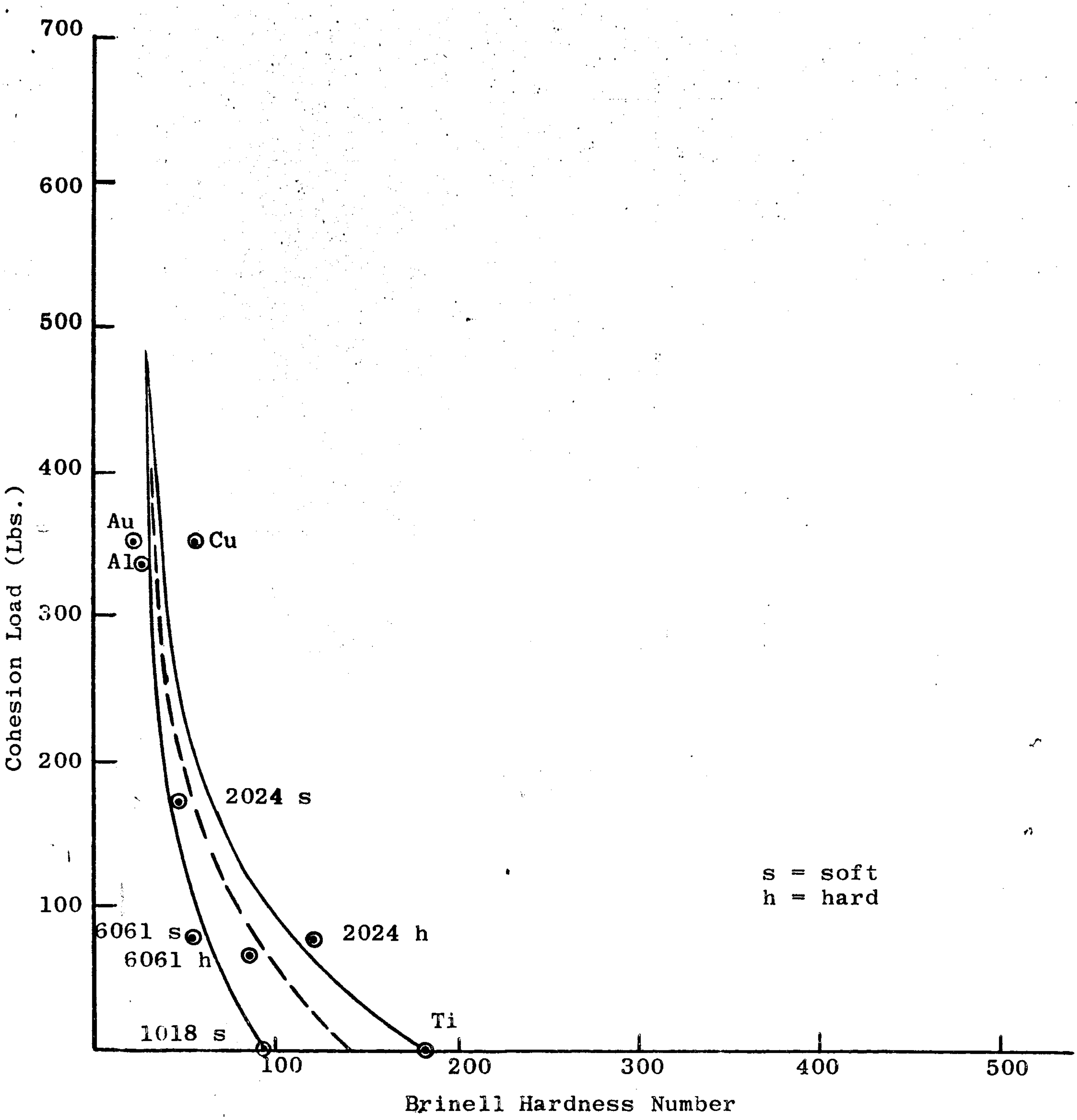

FIGURE 10

COHESION LOAD/BRINELL HARDNESS NUMBER
FOR VARIOUS METALS AGAINST EACH OTHER
2000 LB. APPLIED LOAD AT 125°C FOR 15 MIN.

Adhesion Experiments between Different Metal Pairs

When two dissimilar metals are cleaned and compression loaded in a vacuum, as performed during the cohesion tests previously described the process is called adhesion. A series of such tests were performed with the pairs indicated in Table 5, page 32, already shown. Essentially two sets of adhesion data were taken: 1) adhesion of OFHC copper to a variety of metals; and 2) a similar series using a commercial aluminum alloy (6061) and a few gold specimens against other metals.

Figure 11, page 42, is a plot of the adhesion of pure soft copper to other metals arranged in order of increasing Brinell hardness number. This series too was run off at 125°C under a 2000 lb. loading force for fifteen minutes. Inspection of the curve clearly shows a correlation between adhesion and hardness. Not shown on the curve is the fact noted under the microscope that in every case when the specimens were pulled apart, the softer of the two metals in the pair stuck to the harder of the pair. Spot tests and spectroscopy failed to show any material transfer of the hard to the soft sample.

Efforts to find any other direct correlation between adhesion and a variable similar to those shown for hardness failed. A list of the factors which were plotted against cohesion load or cohesive stress and failed to correlate are given below:

Modulus of elasticity

Modulus of shear

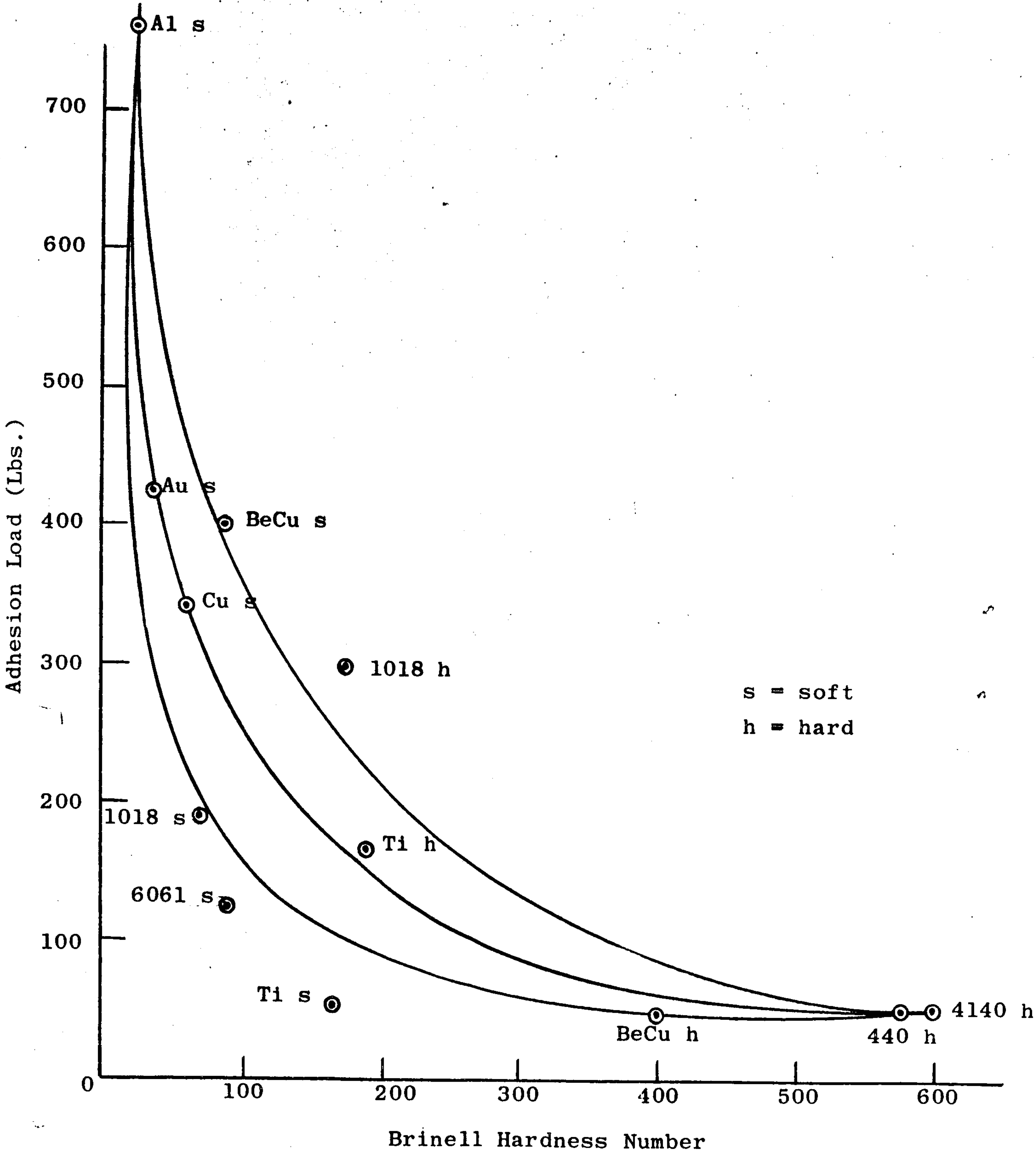

FIGURE 11

ADHESION LOAD VS. BRINELL HARDNESS FOR VARIOUS METALS AGAINST SOFT COPPER APPLIED FOR 2000 LB. LOAD AT 125°C FOR 15 MINUTES

Mutual solubility at test temperature (in atomic %)

Melting point in °K

Electronegativity differences

Electromotive potential of the couple formed

Mutual diffusion coefficient at test temperature

Stability of the oxide, nitride, etc.

Here again it should be emphasized that although a "standard" test was used it represents but one possible time, temperature, and cleanliness condition for the test series and generalization should be approached with skepticism.

CONCLUSIONS

From the data presented in the section on wire brush cleaning, it was found, as might be expected, between copper specimens:

1) cohesion increases with increasing test temperature

2) cohesion does not increase proportionally to the applied load when specimens are loaded over the yield stress

3) cohesion is strongly dependent upon surface cleanliness (reflected here in brushing time)

4) failure of the weld in adhesive processes occurs by plucking of the softer metal surface from the mass and its remnants are left on the harder metal.

Strong cohesion was found to exist for most but not all couples of identical metals and alloys having a Brinell hardness number less than 150 at 125°C after wire brushing in a vacuum of 10^{-9} torr. SAE 1018 (BHN 63), Be-Cu (BHN 86), 2024 T4 (BHN 120), failed to cohere in these tests.

Strong adhesion was found to exist between couples of dissimilar metals (when one of the metals was softer than BHN 150) by adhesion of the softer to the harder metal. The combinations SAE 1018 - Al 2024-T4, coin silver- 6061-0, and Be Cu-6061, failed to adhere in these tests.

Wire brushing is a satisfactory method of cleaning test couples for vacuum cohesion work at 10^{-9} torr although it produces a degree of surface roughening.

In spite of the known tendency for aluminum to cold weld the alloy 2024-T4 (BHN 120) failed to show significant adhesion to itself and to steel (SAE 1018) under test conditions. It would therefore seem profitable to re-evaluate the effects of alloying and dispersed phases on the cohesion process as it may be remembered that Be Cu also shows little tendency to stick to itself in the heat treated condition.

Adhesion/cohesion between metal couples becomes appreciable as the temperature approaches 0.30 of the melting point of the lower melting metal.