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SURFACE PHYSICS AND CHEMISTRY OF ELECTRICAL CONTACT PHENOMENA.(U)
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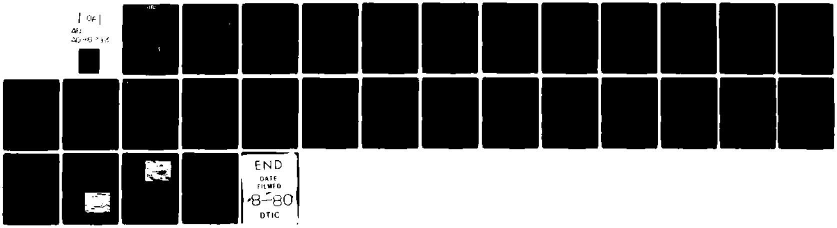

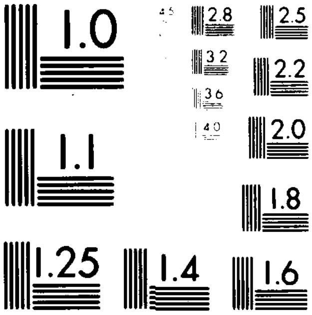
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The vacuum surface segregation of impurities on copper slip ring materials was studied by Auger electron spectroscopy. In addition the surface chemical composition, electrical contact resistance, and friction were measured in situ for a copper slip ring rotating in vacuum against two copper wire brushes running in separate tracks. The results are embodied in reprints and preprints of 4 papers attached as appendices to the report.

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October 1, 1979 - August 31, 1980

SURFACE PHYSICS AND CHEMISTRY OF ELECTRICAL CONTACT PHENOMENA

by

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Research supported by the Office of Naval Research on
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June 1, 1980

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SUMMARY

The work carried out on the present research program dealing with the surface physics and chemistry of electrical contact phenomena is embodied in the four appendices attached to this summary. Appendices I and II were carried our principally under a subcontract from the Westinghouse Research and Development Laboratory. It was supported by the Office of Naval Research under an Advanced Research Projects Agency Contract N-00014-79-C-0110. It is useful from a chronological point of view to include this work in the present report. Appendices III and IV were carried out under the present contract. These publications and manuscripts submitted for publication represent the major results obtained so far on this project. I would, however, like to summarize here briefly the principal findings and the general direction of research efforts.

REPORT

The basic principle which underlies our efforts is that the electrical and mechanical properties of the brush-slip ring interface are determined by the topography and operating temperature of the contacting surfaces, the chemical composition of the surface and near surface regions, and the relative mechanical motion of the contacts. In our first studies we investigated the compositional changes that occur on the surfaces of four different stationary copper specimens when they were heated in ultra high vacuum. It is well known that local contact points in the interface between a brush and slip ring may get very hot during rotation when current flows. As a result, elemental surface segregation of impurities or deposits in the copper sample may occur at the surface, thereby changing its composition. The interfacial composition then would be expected to be quite different from what one has in the bulk. Such an interface could be expected to have very unusual properties. Appendix I shows that indeed drastic changes in chemical composition will occur on copper surfaces when they are heated, even when the bulk copper is 99.9999%. Appendix II focuses on the segregation of sulfur to the surface and some of the kinetics are worked out. It was shown that in most cases one can describe the mechanism of sulfur surface segregation in terms of a simple outgassing model that involves bulk diffusion of sulfur to the surface and its precipitation there. Clearly locally high temperatures will change the interfacial compositions under equilibrium conditions.

While the previous work was being carried out, the ultra high vacuum system was being modified so that one could make several important measurements _in situ_ while a slip ring was being rotated against two brushes on separate tracks and while current was being run across the interface. This new arrangement is discussed in Appendix III. It is now possible to measure _in situ_: (1) the elemental surface composition of a rotating slip ring on any particular track by Auger electron spectroscopy, (2) the electrical contact resistances of the positive and negative interfaces, (3) the average frictional force applied at both brush-slip interfaces, (4) the temperature of the brush approximately 1 mm from the interface, and (5) the residual gas spectrum in the vacuum system with a 90^{0} magnetic sector spectrometer. We can also vary the contact current and have used currents up to 50 amperes to date. After each experiment is run, we remove the brush and slip ring from the vacuum system and examine the topography of their surfaces with a scanning electron microscope. We also have the capability of x-ray mapping and x-ray line profile analysis on the SEM, features that will be extremely useful (when we start varying the composition of the brushes and slip ring) in investigating the transfer of material from one surface to the other. It should also be pointed out that the Auger data is fed directly into a Hewlett-Packard 9825A computer for storage on tape, analysis, and plotting.

The first results from this new system are presented in Appendices III and IV. This work was all done using an ultra high vacuum environment for the OFHC slip ring turning against two 99.999% Cu wire brushes. Of course, this is not a practical environment; but it is the first step in a series of experiments in which the environment will be changed.

We are presently preparing experiments in which wet CO_2 will be introduced into the vacuum system at atmospheric pressure and with the ion pump sealed off. We expect to run the slip ring for about 15 minutes in this environment and then pump it out prior to making the Auger electron spectroscopy (AES) measurements. We can of course measure the contact resistance and friction in the wet CO_2 environment as the slip ring turns. The AES measurements, however, require high vacuum. Thus from them we will get the initial and final composition of the surface of the slip ring but not changes that take place during operation. We are also planning experiments using lower wet-CO_2 partial pressures, such as 10^{-5} torr, where it would take only about 0.1 sec for an adsorbed monolayer to form. That may be sufficient time to observe the same effects as would be observed at atmospheric pressure.

These initial measurements with various gaseous ambients and at various pressures would all be carried out with copper brushes and slip rings. It is expected that several runs will be made before the end of the present contract year. In subsequent experiments the bulk compositions of the brush and slip ring materials will be varied. The specific details of the future research program were more fully described in the original 3 year proposal submitted in 1979.

Appendix I

Elemental Surface Composition of Slip Ring Copper as a Function of Temperature

by

R.W. Vook, B. Singh, E.-A. Knabbe, D.K. Bhavsar, and J.H. Ho

Presented to the 1979 Holm Conference, Chicago, Illinois

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Elemental Surface Composition of Slip Ring Copper as a Function of Temperature

RICHARD W. VOOK, BHOJ SINGH, ERNST-AXEL KNABBE, DILIP K. BHAVSAR, MEMBER, IEEE, AND JACKSON H. HO

***Abstract*—The surfaces of four copper samples—oxygen-free high-conductivity (OFHC) (99.98 percent) Cu, 99.99 percent Cu, 99.9999 percent Cu, and a dilute Ag/Cu alloy (0.086 weight percent (wt %) Ag in OFHC Cu)—were examined in an ultrahigh vacuum system by Auger electron spectroscopy as a function of temperature between 25 and 600°C. It was found that the elemental surface composition of each sample varied drastically with temperature. For example, carbon was a surface constituent in amounts ranging from 0 to about 100 at %, depending upon surface treatment. An interesting finding was the rapid surface segregation of sulfur upon heating in all samples, leading to surface concentrations ranging up to 32 at % in some cases. These results suggest that the higher temperatures occurring at the current-carrying surface asperities will alter the composition of the interface. Hence, the electrical resistance of the interface and its friction and wear properties may be grossly affected.**

INTRODUCTION

IN ELECTRICAL brush/slip ring contacts only a small fraction of the geometric contact area is active in carrying current. Much theoretical work has shown that these local points of electrical contact will have high current densities that tend to raise their temperatures considerably [1]. Thus various temperature-dependent processes can be expected to occur. One of these would be atomic diffusion resulting in changes in the surface compositions of the copper or copper-alloy slip ring and the brush material, even in cases where the bulk solute concentration is extremely dilute. Previous work in this area has shown that elemental surface composition changes are frequently observed when a specimen is heated [2]–[5]. Since the current-carrying capacity of electrical contacts could be limited by a thin film at the interface, perhaps only a few atom layers thick, chemical compositional changes at that interface could have profound effects. It was on this basis that a study of the change in elemental composition of various polycrystalline copper surfaces as a function of temperature was initiated.

EXPERIMENTAL AND ANALYTICAL TECHNIQUES

Materials Used and Surface Preparation

Four copper samples were used in this work: oxygen-free high-conductivity (OFHC) (99.98 percent) Cu, 99.99 percent Cu (Materials Research Corporation "VP" grade), 99.9999 percent Cu (Gallard–Schlesinger Co.), and a dilute Ag/Cu alloy (0.086 weight percent (wt %) Ag in OFHC Cu). The surfaces of each were polished flat with grit 600-A emery paper, ultrasonically cleaned in acetone, and then rinsed with ethanol. This procedure constitutes conventional cleaning (CC). The samples were then inserted in a stainless steel bell jar system equipped with a 3-kV Auger electron spectrometer (AES), a 90° magnetic sector partial pressure analyzer, and a 600-eV sputter ion gun. Each specimen was clamped to a stainless steel substrate holder by means of copper clamps made up of the same material as the specimen itself. The substrate holder contained a Ta wire heating element insulated by means of alumina tubes. The temperature of the holder was measured with a chromel–alumel thermocouple. Specimen temperatures ranged from 25°C to 600°C. All Auger spectra were taken with the same Auger parameters like electron beam energy, electron current density, modulation voltage, etc.

Two sets of experiments were carried out. In the first, the CC sample was heated without baking the system and without sputter-etching its surface. The pressure in the system during these experiments ranged from 3×10^{-9} Torr to 1×10^{-8} Torr. With the important exception of the vacuum environment, these experiments correspond more closely to what may occur during an actual operating contact.

In the second set of experiments the system was baked and then cooled to room temperature where argon ion sputtering was carried out to clean the surface of each sample. The base pressure prior to introducing argon was 6×10^{-10} Torr. It was found that 600-eV argon ions did not remove carbon from the surface of the sample. However, by adding another 1500 V to the specimen itself, almost all the carbon was removed after 16 h of sputtering at an argon pressure of 5×10^{-5} Torr. The remaining carbon covered approximately 2–10 atomic percent (at %) of the surface. The argon was then removed and the system pumped down to a pressure of 3×10^{-9} Torr in 1 h, where the principal constituent was argon. During heating of the samples to 600°C, the background pressure rose to approximately 5×10^{-9} Torr. Sputter-etched surfaces prepared in this manner will be designated by SE. Only the CC Ag/Cu surface was run once while two or more runs were made on each of the other specimen surfaces.

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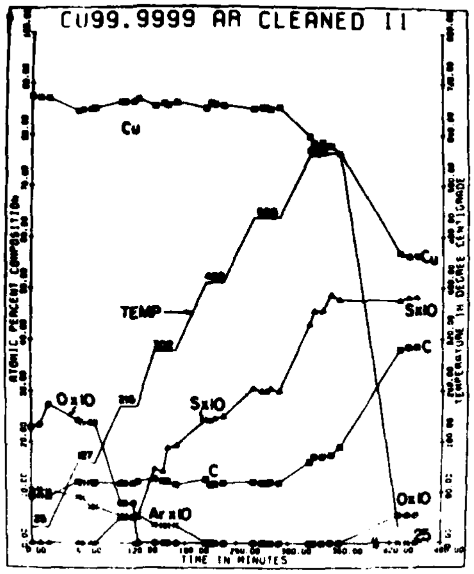

Fig. 1. Typical computer printout of surface composition of an SE sample of 99.9999 percent Cu. Run II.

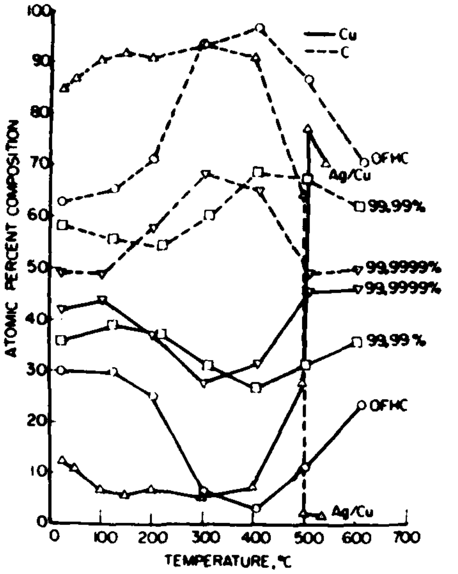

Fig. 2. Carbon and copper concentrations on surfaces of four different kinds of conventionally cleaned Cu samples as a function of temperature.

Auger Electron Spectrum Analysis

The surface chemical analysis was based on methods reported earlier [6]. For element x its atomic concentration C_x is given by

$$C_x = \frac{I_x/S_x}{\sum_i I_i/S_i}, \qquad (1)$$

where I_x is the peak-to-peak height of a particular Auger transition for element x and S_x is the relative sensitivity factor of this element x with respect to a pure elemental standard measured under identical experimental conditions. The summation is over one Auger transition of each of the n elements present on the surface. Values of S_i and typical Auger spectra have been tabulated [7]. Equation (1) is a first approximation to a concentration since it neglects changes in line shape, surface topography differences in Auger electron excitation due to the backscattering of the primary electrons, electron beam effects, and variations in the sampling depth of different elements (or different lines from the same element) due to the dependence of the escape depth on Auger electron energy. Recent reviews of these problems were given by Powell [8] and Chang [6].

The data analyses were done by computer techniques: a typical run is shown in Fig. 1 where the Auger peak-to-peak heights and specimen temperature are plotted versus the time during which the data were taken.

RESULTS

The data obtained from graphs such as Fig. 1 were used to plot Figs. 2–7, which show the average atomic concentrations at a given temperature for various elements and various copper substrate materials and the two surface preparations (CC and SE). Because the experimental objectives were to obtain the cumulative effects of elemental surface segregation during the heating process, no depth profiles were obtained. The present experiments, therefore, are more closely related to what would be expected to occur in practical cases involving operating electrical contacts. In interpreting the experimental AES results, it should be remembered that the actual elemental aggregations that occur on a surface may in some cases penetrate a distance of many atom layers into the bulk. Figs. 2 and 3 give the C and Cu concentrations for the two surface preparations as a function of temperature. It is clear from Fig. 2 that the principal surface constituent on CC copper surfaces is carbon. This effect is commonly observed in essentially all research in the surface science area. The Ag-bearing Cu shows a surprising drop in

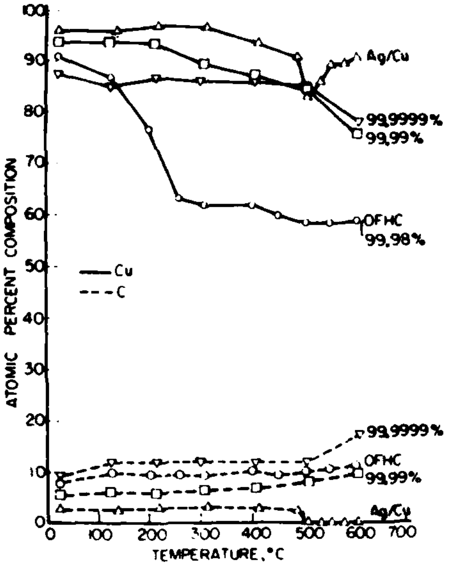

Fig. 3. Carbon and copper concentrations on surfaces of sputter-etched Cu samples as a function of temperature.

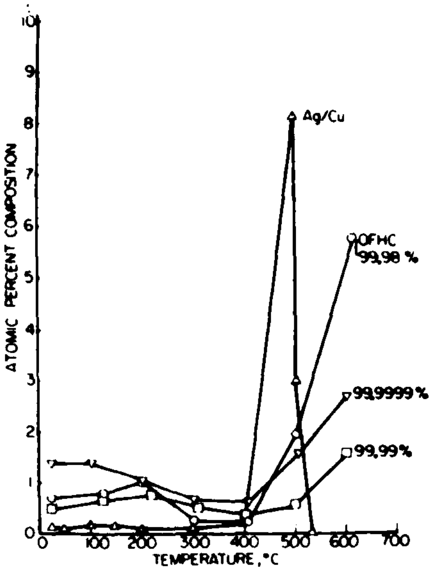

Fig. 4. Surface concentrations of sulfur on four conventionally cleaned Cu samples as a function of temperature.

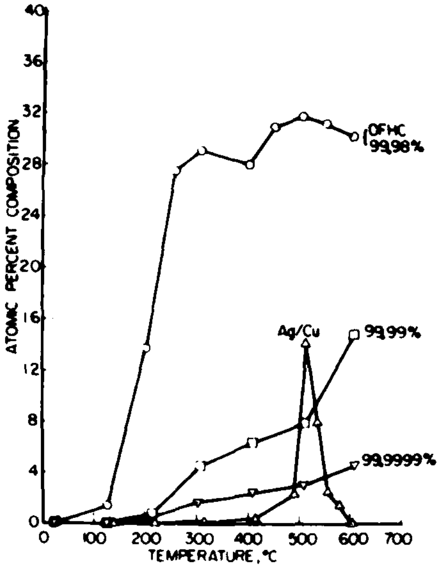

Fig. 5. Surface concentrations of sulfur on four sputter-etched Cu samples as a function of temperature.

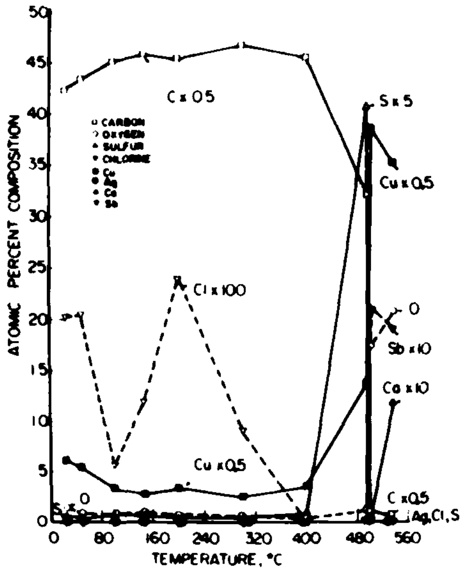

Fig. 6. Surface composition of a conventionally cleaned Ag/Cu alloy (0.086 wt% Ag) up to 535°C.

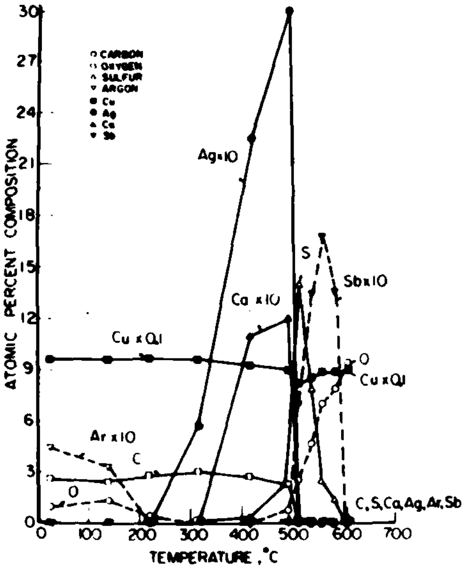

Fig. 7. Surface composition of a sputter-etched Ag/Cu alloy (0.086 wt% Ag) as a function of temperature.

carbon concentration above 500°C for both surface preparations: to two percent and to zero for the CC and SE surfaces, respectively. The initial high C concentrations on the CC surfaces are due to the adsorption of trace hydrocarbons, CO, etc., in the atmosphere prior to insertion of the samples into the ultrahigh vacuum (UHV) system. It is a result that cannot be avoided as long as atmospheric exposure is allowed to occur.

The concentrations of sulfur on copper surfaces are shown in Figs. 4 and 5. On CC surfaces (Fig. 4) there is a tendency for the sulfur concentration to drop on heating the three Cu samples to 300–400°C. Above 400°C there is rapid surface segregation in all four samples. In the case of the Ag/Cu alloy, however, there is a rapid drop to zero concentration at 535°C. The SE surfaces had zero S concentrations initially. Appreciable surface segregation occurred above 100°C for OFHC Cu and above 200°C for the other three samples. Again, above 500°C the sulfur concentration on the Ag/Cu alloy dropped sharply to zero at 600°C. In the case of SE OFHC Cu, the surface concentration of S rose to 36 at% upon cooling from 600°C to 25°C. This sulfur concentration was the highest value observed in these experiments.

Figs. 6 and 7 show the very interesting surface concentration changes that occur when an extremely small amount of Ag is added to OFHC Cu and the sample is heated. It should first be mentioned that the elements shown in these figures are the only ones that were detected. Also, the presence of implanted argon was observed for all SE samples in this work. Argon always desorbed completely as the temperature rose above 200°C. Figs. 6 and 7 also show that on heating, the surface segregation of Ag does not occur for the CC surface (Fig. 6), but does occur for the SE surface (Fig. 7). However, on cooling the CC sample of Fig. 6, a small amount of Ag segregation was observed (2 at % maximum at 205°C), which decreased to about 0.5 at % at 25°C.

Surface segregation of S, Ca, Sb, and O also occurred in the Ag/Cu specimens. Above 500°C carbon disappeared entirely in the SE case and almost so for the CC surface (to about 2 at %). Also above about 500°C, sulfur is eliminated at approximately the same time as the oxygen concentration increases. At 600°C in the SE case only Cu and O are detected on the surface. Presumably the diffusion of dissolved oxygen from the interior of the alloy to the surface is enhanced by the presence of a small amount of Ag in the bulk, since oxygen permeates Ag rapidly at high temperatures.

Small amounts of oxygen were present on the surfaces of all of the specimens at the beginning of each heating cycle. Some of it was no doubt due to CO adsorption from the residual gas in the UHV system. Only in the Ag/Cu alloys did the oxygen concentration rise appreciably upon heating. Generally, it decreased as the CO was desorbed or the oxygen atoms diffused into the bulk of the Cu specimen.

DISCUSSION

The data presented here represent a wide range of significant phenomena. A few observations can, however, be made. The evidence obtained showed that the same general features present in the surface segregation and disappearance data were reproducible from run to run on the same kind of specimen. The more quantitative reproducibility, however, always occurred for the sputter-cleaned surfaces rather than those that received the conventional cleaning.

For electrical contact phenomena it is most significant that the major constituent by far on the CC surfaces at room temperature was always carbon within about 100 Å. Also, the relative amount of carbon depended a great deal on the temperature of the surface, rising to almost 100 at % slightly above 400°C for OFHC Cu. These higher temperature results may be more applicable to current-limiting phenomena in electrical contacts because of the expected large rise in temperature of the current-carrying asperities. Also, the sharp drop to almost zero carbon on the surface of the dilute Ag/Cu alloy at 500°C was most unexpected. This result emphasizes the importance of trace elements in slip ring and brush materials.

The next important feature of the data is the rapid surface segregation of S that occurs when the samples are heated. Harris [2], using AES techniques, was the first to report that S segregates on the surface of Cu and other metals. Jenkins and Chung [9] reported the same phenomenon for (111) oriented monocrystalline Cu. Others have noted similar effects on other substrates [3]–[5]. However, no systematic study of elemental sulfur segregation on polycrystalline copper surfaces as a function of temperature, surface preparation, and bulk purity has been reported. In the present investigation it was found that even in materials such as 99.99 percent and 99.9999 percent Cu, where S was not detected by emission spectroscopy techniques, significant S segregation was observed. It is believed that the sulfur atoms diffuse from the bulk of the sample to the grain boundaries and then to the surface. Calculations show that a bulk concentration of S as low as 0.1 ppm in a 1-cm cube can lead to monolayer coverage on the surface. The nominal sulfur concentration in OFHC Cu is 12 ppm (American Society for Testing and Materials specification B170, grade 2). This problem is treated more extensively elsewhere [10].

While all these results were obtained under high vacuum conditions, they illustrate some of the tendencies for surface compositional changes that might be expected under normal or controlled atmospheric conditions. They also show very clearly that the local operating temperature of electrical contacts may be of utmost importance in determining the nature of the film resistance which controls the current-carrying capacity of the interface and its friction and wear characteristics.

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Appendix II

AES Study of Sulfur Surface Segregation on Polycrystalline Copper

by

B. Singh, R.W. Vook, and E.-A. Knabbe

Presented at the 26th National Symposium of the American
Vacuum Society, New York City, November 5-9, 1979.

AES study of sulfur surface segregation on polycrystalline copper

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(Received 20 August 1979; accepted 16 November 1979)

The surfaces of four polycrystalline copper samples were examined under UHV conditions by AES: OFHC Cu, 99.99% Cu, 99.9999% Cu, and 0.086 wt.% Ag in OFHC Cu. Surface chemical compositions in the range of 25°–600°C were determined. Sulfur concentrations on the surfaces at various temperatures were also measured as a function of time. In almost all cases the sulfur concentration increased as $(\text{time})^{1/2}$. This process was interpreted in terms of a model based on diffusion controlled "outgassing" in which the sulfur segregated to the surface instead of vaporizing. The activation energies for this process for the four samples ranged from 0.18 to 0.31 eV/atom. RHEED data from an OFHC Cu sample indicated that sulfur formed an orthorhombic Cu_2S structure on the surface. Around 500°C the Ag–Cu alloy experienced a complex, rapid stage of segregation of sulfur. Above this temperature range the surface sulfur concentration dropped rapidly, reaching zero at 600°C.

PACS numbers: 82.65.Jn, 66.30.Jt, 64.75. + g

I. INTRODUCTION

Electrical contact phenomena including sliding friction applications involve the generation of high temperatures at local asperities in the particular interface. Such high temperatures almost inevitably cause changes in the surface composition of the contact material. Consequently the electrical and mechanical properties of the interface, such as current-carrying capacity, friction, and wear, will be changed accordingly. While various materials and coatings have been used on electrical contacts in practical applications, the most commonly used material is copper, usually OFHC copper. It was for this reason that a surface chemical analysis of various polycrystalline copper samples as a function of temperature in the range 25°–600°C was carried out under ultra-high vacuum conditions and using Auger electron spectroscopy techniques.[1]

Four polycrystalline copper samples of different purity were used in this study. A discussion of the actual chemical composition of the surfaces as a function of temperature will be given elsewhere.[2] In the present report the details of the segregation of sulfur on the copper surfaces is given, sulfur being usually the third most prevalent element there, after Cu and C. It was found that sulfur segregated to the surface in all specimens, even on a copper sample of 99.9999% purity. The segregation process is diffusion controlled in all cases and at all temperatures investigated, except near 500°C in a dilute Ag–Cu alloy. For this case the segregation mechanism appeared to be quite different. Previous work has also shown that sulfur segregates on the surface of copper at elevated temperatures.[3-5] Harris[3] showed that S segregated to the surfaces of Cu and many other metals upon heating. Jenkins and Chung[4] observed the formation of an ordered sulfur superstructure on a (111) Cu monocrystal. Apparently no one has reported a detailed study of the segregation of sulfur on the surfaces of various polycrystalline copper samples of varying purity, as was carried out in the present investigation.

II. SAMPLE PREPARATION AND EXPERIMENTAL PROCEDURE

Four different copper samples were used for this study: OFHC (99.98%) Cu, 99.99% Cu (Materials Research Corporation "VP" grade), 99.9999% Cu (Gallard–Schlesinger Co.), and a dilute Ag/Cu alloy (0.086 w/o Ag in OFHC Cu). A rectangular piece, about 1 cm × 1 cm × 1.5 mm thick, was cut from each sample, polished flat using emery papers ending with grit 600 A, washed in acetone in an ultrasonic bath for approximately 30 min and finally rinsed with ethanol. Each sample was attached to a stainless steel substrate holder by means of clamps made up of the same material as the specimen itself. The substrate holder was heated by a Ta wire heating element that was electrically insulated by means of alumina tubes. The heating element itself was wound in such a way as to eliminate most of the associated magnetic field. A chromel–alumel thermocouple was clamped to the substrate for temperature measurement. The specimen and holder, mounted on a specimen manipulator, were then inserted into a stainless steel ultrahigh vacuum chamber, equipped with a 3 keV cylindrical mirror analyzer for Auger electron spectroscopy (AES), a 90° magnetic sector partial pressure analyzer, and a 600 eV inert gas sputter ion gun.

The system was baked at 200°C and then cooled to room temperature where argon ion sputtering was carried out to clean the surface of each sample. The base pressure prior to introducing argon was 5–6×10^{-10} Torr (7×10^{-8} Pa). The argon ion beam was focused and positioned on the sample by precalibrating the specimen translation knobs with iso (ion beam) current contours. The 600 eV argon ions did not remove carbon from the surface. By adding −1500 V to the specimen, almost all the carbon was removed after 16 h of sputtering at an argon pressure of 5×10^{-5} Torr (7×10^{-3} Pa). The remaining carbon covered approximately 2–10 a/o of the surface. The argon was then pumped out and the pressure reduced to 3×10^{-9} Torr (4×10^{-7} Pa) in one hour, where the principal residual constituent was argon. During heating of the sample to 600°C, the background pressure rose to approximately 5×10^{-9} Torr (7×10^{-7} Pa).

III. RESULTS

The Auger spectra reported here were obtained using a primary beam energy of 3 KeV and a modulation amplitude of 5 V (peak to peak). Typical Auger traces covering a 50–1250 eV range were recorded. In each experimental run, the sample was heated to a desired temperature from 25° to 600°C in steps and then held at that temperature while successive Auger spectra were taken during the subsequent 50–60 min. For correlating Auger traces of various runs on four samples, peak to peak heights of all signals were normalized[6] to fractional atomic surface concentrations by the relation $C_x = (I_x/S_x)/(\sum_{i=1}^{N} I_i/S_i)$, where I_x is the peak to peak Auger electron intensity for a particular Auger transition and S_x is the relative elemental sensitivity factor measured on a pure element standard under identical experimental conditions. The summation is over one Auger transition of each of the N elements present in the surface. Values of S_i and typical Auger spectra have been tabulated.[6] The equation for C_x is only a first approximation to the surface concentration since it neglects changes in line shape, surface topography differences in Auger electron excitation due to back scattering of the primary electron, electron beam effects, and variations in the depth of sampling for different elements or different lines from the same element that are caused by the dependence of escape depth on Auger electron energy.[1] However, for the purposes of the present report, the procedure used here to determine C_x is adequate.

At room temperature, sputtered clean surfaces show about 90 a/o surface concentration of Cu and a small concentration of C and Ar. See Fig. 1(a). Argon was implanted during sputtering and was completely desorbed as the temperature rose about 200°C. Carbon, however, stayed on all four surfaces. Its concentration increased slightly with temperature. However, only for the Ag/Cu alloy, above 500°C the carbon concentration decreased to almost zero ($\lesssim 2$ a/o). The slowly increasing concentrations of carbon probably arise from electron beam dissociation of CO and/or other hydrocarbons in the residual gas rather than precipitation from the bulk. Initially, sulfur concentrations were below the detection limit on the surfaces at room temperature. Appreciable surface segregation occurred above 100°C for OFHC Cu and above 200°C for the other three samples. Around 500°C, the sulfur concentration on the Ag/Cu alloy increased rapidly, then dropped sharply to zero at 600°C. Also above 500°C, the oxygen concentration started increasing at approximately the same time as the sulfur was eliminated. At 600°C only oxygen and Cu were detected on the surface. In Fig. 1(b), the AES trace at 600°C of an OFHC Cu sample shows a sulfur surface concentration of approximately 30 a/o. Upon cooling from 600° to 25°C, the surface concentration of S rose to 36 a/o. At no temperature was an equilibrium concentration of S attained on the surface. Thus additional S would be expected to precipitate on cooling from 600°C. The sulfur concentration of 36 a/o was the highest value observed in these experiments.

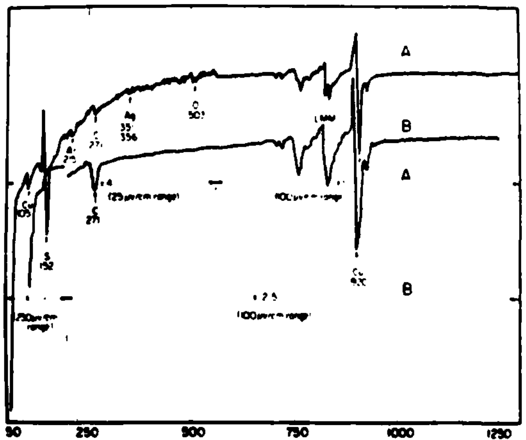

FIG. 1 (a) AES trace of sputter cleaned Ag/Cu sample. (b) AES trace of OFHC Cu sample heated to 600° C and kept there for 10 min.

A small amount of surface segregation of Ag, Ca, Sb, and O also occurred in the Ag/Cu specimens. While the alloy contained 0.086 wt.% of Ag in OFHC Cu, a 3 a/o maximum Ag segregation was detected on the surface at 400°-500°C. All surface impurities other than oxygen disappeared at 600°C. The surface composition changes for all four specimen types are given elsewhere.[2]

IV. ACTIVATION ENERGIES FOR SEGREGATION

The chemical analyses supplied by the manufacturers of the 99.99% Cu and 99.9999% Cu samples indicated that they did not detect any sulfur in the bulk. Thus an analysis that did not depend on knowing this quantity for each sample was required. The method used involved the assumption that segregation of sulfur occurred via a diffusion process essentially identical to that involved in the outgassing of materials in a vacuum. Surface segregation may be thought to differ from outgassing only to the extent that the diffusing species is not volatile but accumulates on the surface. One would expect this procedure to be valid in the present experiments where the surface concentration of the segregant was small ($\lesssim 36$ a/o in our maximum case). The diffusion controlled outgassing rate from a slab having a uniform initial concentration C_i has been obtained as a solution to Fick's law. It is given by[7]

$$Q_D = \frac{4ADC_i}{d}\sum_{n=0}^{\infty}\exp\left\{-[\pi(2n+1)]^2\frac{Dt}{d^2}\right\} \qquad (1)$$

where Q_D is in such units as CC (STP)/s, A is the surface area, d is the thickness of the slab, t is the time and n is an integer. Since diffusion coefficients D in solids are very small, the condition $Dt/d^2 < 0.05$ is satisfied for reasonable times. In this case the gas evolution rate may be approximated by[8]

$$Q_D = A\,C_i\,(D/\pi t)^{1/2}. \qquad (2)$$

If I_x is the peak to peak height of the Auger electron signal,

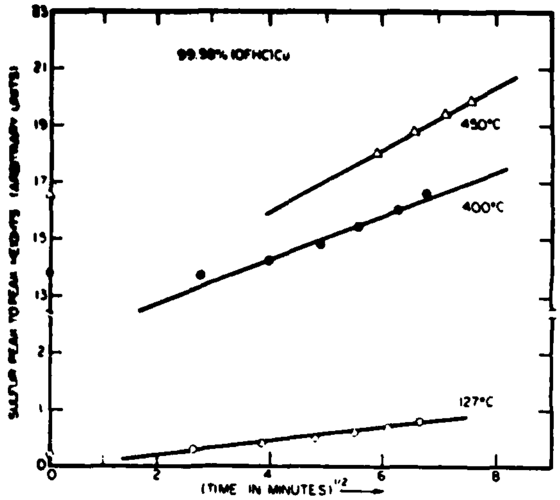

FIG. 2. AES peak to peak heights of S segregated on the surface of OFHC Cu as a function of (time)$^{1/2}$.

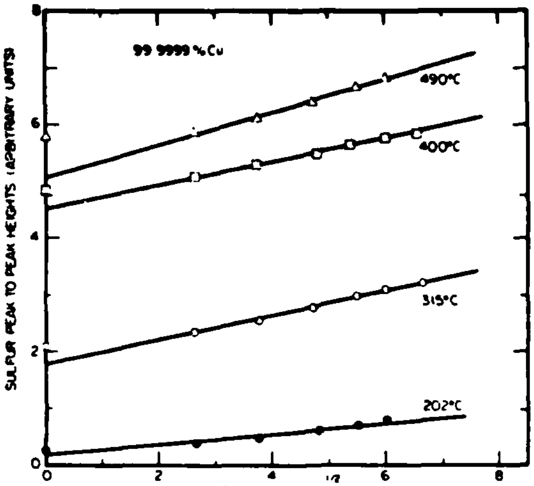

FIG. 4. AES peak to peak heights of S segregated on the surface of 99.9999% Cu (Gallard-Schlesinger Chemical Co.) as a function of (time)$^{1/2}$.

then $I_s \sim \int Q_D\, dt$. Thus

$$I_s = 2SA\, C_i\, (D/\pi)^{1/2}\, t^{1/2} + I_0 = Kt^{1/2} + I_0 \qquad (3)$$

where S is a constant of proportionality which includes the relative sensitivity. Since we do not know C_i, we can rewrite this equation as indicated in (3) where K is another constant that is proportional to $D^{1/2}$.

Plots of the sulfur peak to peak heights versus the square root of time for a slow segregation process are shown in Figs. 2, 3, 4, and 5 for four copper samples at various temperatures. The slopes K of the least squares fit lines (excluding the points at $t = 0$) are proportional to the square root of the diffusion coefficient D_T. The squares of these slopes, i.e., K^2, are plotted on a logarithmic scale vs the reciprocal of the absolute temperature T in Fig. 6. Only the data from the three lowest temperature lines in Fig. 5 are included in Fig. 6. Again the lines are the least squares fits to the points. Since

$$D_T = D_0 \exp(-E_D/kT) \qquad (4)$$

the slopes of the lines in Fig. 6 yield activation energies for sulfur segregation on copper surfaces. The values obtained are 0.31, 0.24, 0.18, and 0.29 eV/atom for OHFC Cu, 99.99% Cu (VP), 99.9999% Cu, and the Ag/Cu alloy, respectively.

For the Ag/Cu alloy, sulfur segregated very rapidly around 500°C, where relation (3) did not hold (see Fig. 5). However, this rapid segregation could be interpreted as a fast adsorption process where the rate of adsorption (or segregation in our case) is given by[9]

$$dN_s/dt = AP_s(1-\theta)N_t \exp(-E_A/kT). \qquad (5)$$

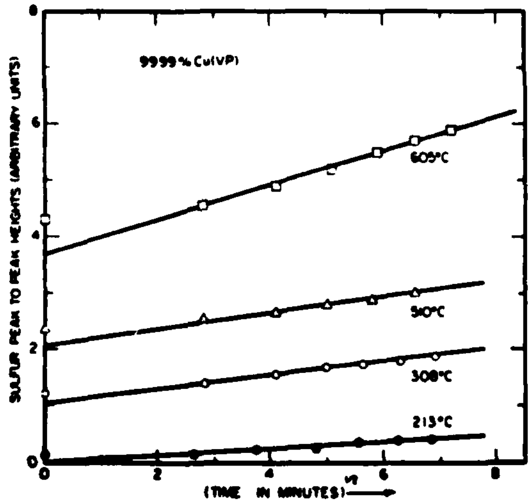

FIG. 3. AES peak to peak heights of S segregated on the surface of 99.99% Cu (VP Grade, Materials Research Corporation) as a function of (time)$^{1/2}$.

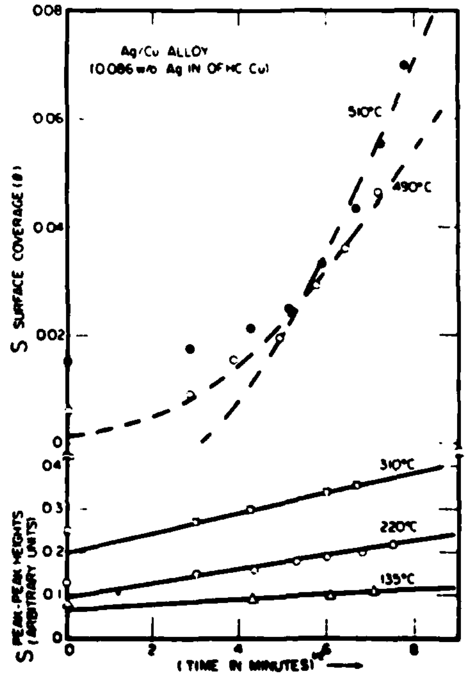

FIG. 5. AES peak to peak heights of S segregated at temperatures 135, 220, and 310°C on the surface of the Ag/Cu alloy as a function of (time)$^{1/2}$. The dashed curves at 490° and 510°C were obtained theoretically from Eq. (6) using E_A and A_o values calculated from the straight lines shown in Fig. 7.

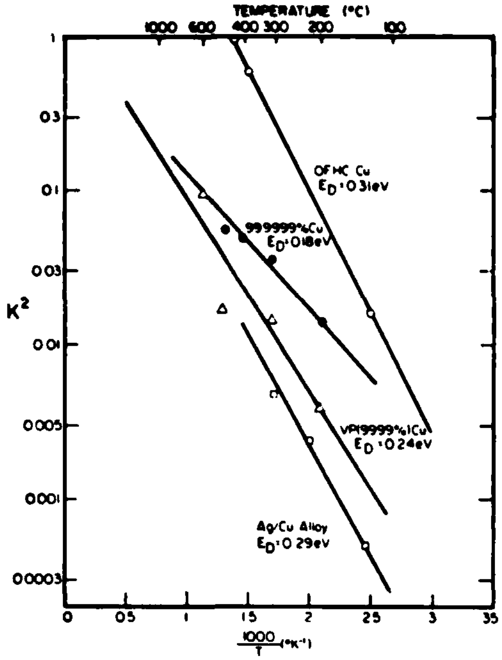

FIG. 6 The squares of the lines (K^2) in Figs 2–5 as a function of reciprocal temperature. See text.

N_x and N_t are the number of surface sites covered by the adsorbed species x and the total number of surface sites respectively; $\theta = N_x/N_t$ is the surface coverage; P_x is the residual gas pressure and A is a pre-exponential constant. In our case, P_x is related to an internal "pressure" that causes the surface segregation. Expressing this equation entirely in terms of θ, letting $AP_x = A_0$ and integrating, one gets

$$\ln (1 - \theta) = -A_0 t \exp(-E_A/kT) + \theta_0. \qquad (6)$$

Normalized peak to peak Auger signals of sulfur (i.e., C_x referred to earlier) essentially gave the ratio of surface sulfur atoms to the total number of atoms of all elements on the surface, in other words, surface coverage θ.

Sulfur data for the rapid segregation observed at 490° and 510°C on the Ag/Cu alloy are plotted in Fig. 7 according to Eq. 6. At 490°C a good fit is obtained for all the data points (except the first) to a least squares straight line. At 510°C, two linear regions are apparent, suggesting that at least two separate processes are occurring. At 535°C the surface sulfur concentration had already begun to decrease. At 600°C no sulfur remained on the surface.[2]

Reflection high energy electron diffraction (RHEED) patterns were also obtained from one of the OFHC Cu samples after it had been cooled to 25°C from 600°C. This sample then had a 36 a/o surface coverage of sulfur, as determined by AES. The RHEED work was done in a transmission electron microscope after the sample had been exposed to air for a few hours. The patterns obtained were characteristic of Cu, Cu_2O, and orthorhombic Cu_2S,[10] the Cu_2O having formed on air exposure.

V. DISCUSSION

A single mode of sulfur segregation on the surfaces of the copper samples investigated in this work predominated in the temperature range from 25° to 600°C. This mode was diffusion controlled and can be described by Eq. (3). It appears to be identical in its mathematical description to the well known phenomenon of outgassing, with the difference that the diffusant accumulated on the surface in our case rather than being vaporized. The activation energies for the four samples were 0.31, 0.24, 0.18, and 0.29 eV/atom for OFHC (99.98%) Cu, 99.99% (VP) Cu, 99.9999% Cu, and 0.086 w/o Ag in OFHC Cu, respectively. The lowest value of activation energy was characteristic of the purest sample, while the more impure samples gave higher values. It is estimated that the error in these values is of the order of ± 0.05 eV/atom.

A very rapid segregation of sulfur near 500°C on the Ag/Cu alloy was also observed. Eq. (6), which applied well at 490°C, can be solved for θ. Its time derivative $d\theta/dt$ decreases exponentially with time. It has been suggested that this result may be caused by an activation energy E_A which increases with coverage.[9] However, the linear plot at 490°C shown in Fig. 7 indicates that after a short initial period of adjustment, the slope $[-A_o \exp(-E_A/kT)]$ does not vary with time. It therefore does not vary with coverage, which increases with time. Thus E_A appears to be a constant with respect to coverage at 490°C. By solving simultaneously the equations of the 490°C line and the 510°C line at longer times (shown in Fig. 7) one can obtain an activation energy $E_A = 1.2$ eV/atom with $A_0 = 66710\ s^{-1}$. Above this 500°C temperature range, the sulfur concentration dropped and reached zero at 600°C. Simultaneously the oxygen surface concentration rose from zero to a substantial value suggesting that the mechanism of sulfur oxidation to SO_2, as observed by Holloway and Hudson[11] on nickel, may also be effective in the case of copper. The difference in the two cases would be that for Ni,

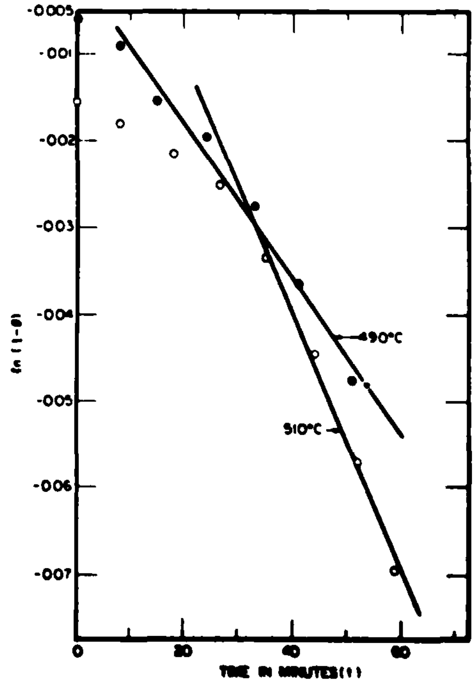

FIG. 7. Surface segregation of S on a dilute Ag/Cu alloy near 500°C. The figure shows the relationship between ln(1−θ) and time at 490° and 510° C.

oxygen had to be introduced into the residual gas spectrum for the process to occur. In the case of Cu, oxygen segregated to the surface from the bulk. Other surface composition changes on the Ag–Cu alloy in the 475–600 °C temperature range were observed also,[2] indicating a very complex situation which is presently under further investigation.

ACKNOWLEDGMENTS

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Appendix III

In Situ AES Characterization of Rotating Electrical Contacts

by

B. Singh and R.W. Vook

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Illinois, September 29 - October 1, 1980

In Situ AES CHARACTERIZATION OF ROTATING ELECTRICAL CONTACTS

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ABSTRACT

The electrical contact resistance and surface chemical composition of a rotating copper slip ring in contact with two wire brushes were investigated in situ under ultra high vacuum conditions as a function of the number of revolutions of the slip ring. The initial surface of the copper slip ring was examined by Auger electron spectroscopy (AES) techniques and found to be almost completely covered by surface impurities largely consisting of carbon. As the slip ring was rotated in contact with the brushes, the impurity concentration declined sharply to less than 10 a/o after several hundred revolutions. A corresponding decrease in electrical contact resistance and a sharp increase in friction and wear were also observed. Regardless of polarity, the brush with the higher contact pressure had the lower contact resistance. The in situ experiments were terminated when the motor turning the slip ring could no longer overcome the adhesive forces between the brushes and slip ring. Subsequent scanning electron microscopy observations of the brush and slip ring surfaces gave supplementary information on the mechanisms of friction, wear and surface cleaning during rotation. For a given experiment the change in contact resistance during rotation divided by the change in impurity concentration on the surface of the slip ring is approximately the same for the positive and negative interfaces. This parallism of contact resistance and surface impurity concentration suggests that the former is caused to a significant extent by the latter. Since the surface impurities are mostly carbon atoms, it is concluded that carbon impurities are largely responsible for the observed contact resistance.

INTRODUCTION

Most previous studies of electrical contact phenomena have been concerned with experiments carried out under normal or near normal atmospheric pressure conditions because those are the conditions of principal commercial importance.[1] Recent workers in this area, however, have used controlled environments such as for example humidified carbon dioxide,[2,3] humidified air and helium.[4] In a few cases ultra high vacuum techniques have been used to study adhesion and contact resistance under clean surface or carefully contaminated surface conditions.[5-9]

In the present experiments in situ measurements were made on a copper slip ring rotating in contact with two copper wire brushes[3] running on different tracks under ultra high vacuum conditions. The electrical contact resistance of each brush-slip ring interface was measured along with the chemical composition of the surface of the slip ring as a function of the number of revolutions. Subsequent examination of the contact surfaces by scanning electron microscopy was also carried out. The results allow correlation of contact resistance with interfacial chemical composition.

EXPERIMENTAL DETAILS

A stainless steel ultra high vacuum (UHV) system was used to investigate electrical contact phenomena associated with rotation of a copper sli ring in contact with two copper wire brushes running on different tracks. Residual pressures in the low 10^{-9} torr range were obtained in the unbaked system after several days of pumping by an arrangement consisting of a titanium sublimation pump and a Vac-ion pump.

Figure 1 shows the arrangement of the slip ring which rotates in contact with two Cu wire brushes. The brushes are pressed against the sli ring by means of a stainless steel spring that is electrically insulated with ceramic beads. The brushes can be removed from the contacting slip ring surface by manipulating two linear-rotary vacuum feedthrus. The slip ring is axially attached to a magnetically coupled rotary feedthru, which is turned by an A. C. motor coupled to it by a rubber belt. The slip ring is composed of OFHC (99.98%) Cu and the brushes each consist of 362, 0.005" diameter, 99.999% Cu wires.

The temperature of the interface is monitored in an approximate way by a copper-constantan thermocouple (TC) included in brush (1). The thermocouple junction is located about 1 mm from the interface to avoid contact and possible surface contamination. It is electrically insulated by means of glass wool sleeving. The brushes are tightly screwed to two 5 5/8" long rectangularly shaped (1" x ½") stainless steel electrodes, each having a smooth hinge in the middle. This hinge lets the brush move away from the slip ring but prevents it from collapsing towards the slip ring

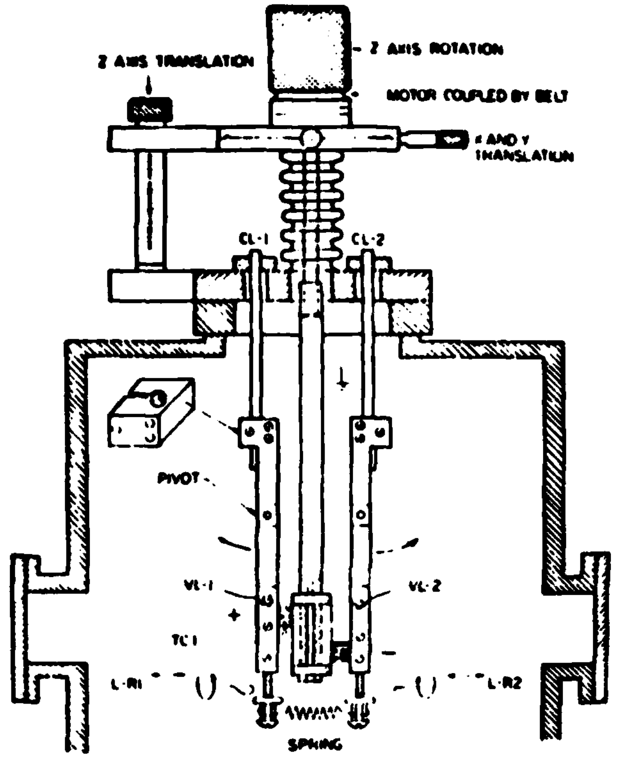

FIGURE 1. Schematic of *UHV vacuum chamber*, rotational feedthru and slip ring-brush assembly.

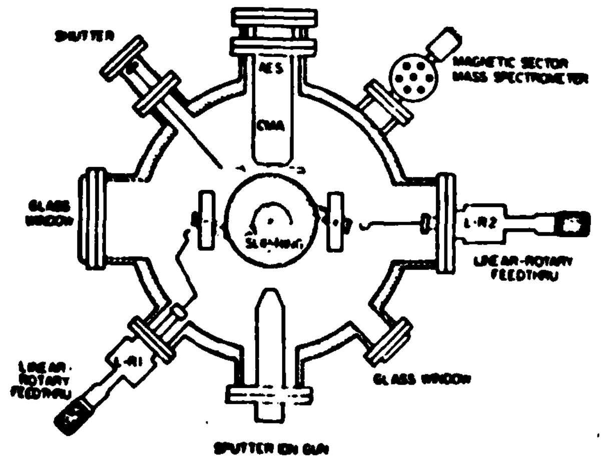

FIGURE 2. Top view of the slip ring-brush assembly, CMA, sputter-ion gun, mass spectrometer and other feedthrus in the UHV chamber.

These electrodes are then attached to two 3/8" diameter OFHC Cu-ceramic vacuum feedthrus (CL). This arrangement provides a low series resistance to the contact resistances. The voltage leads (VL) are clamped near to each brush to avoid the relatively large and erratic hinge resistance. The brushes are arranged at 180° to each other and axially displaced (1.6 cm) to make separate tracks on the slip ring. The whole brush-slip ring assembly is mounted on a specimen manipulator capable of x, y, z displacements. The UHV system contains an Auger cylindrical mirror analyzer (CMA), a 3 Kev sputter ion gun and a 90° magnetic sector partial pressure analyzer. See Figure 2.

The surfaces of the OFHC copper slip ring and the wire brushes were mechanically polished with emery papers down to grit 600-A and then rinsed in acetone and ethanol. Each brush was mounted with a thin (0.03" thick) stainless steel sheet behind it to stiffen it. The end of the stainless steel sheet was located approximately 1 mm away from the slip ring surface.

The slip ring was electrically grounded by means of thick copper wires sliding on the stainless steel axis, the neutral contact. The contact resistances between (1) the positive brush and slip ring and (2) the negative brush and slip ring were recorded on a dual pen recorder. Each measured resistance therefore includes a small lead resistance as well as the contact resistance at the brush. Since no appreciable current flowed through the neutral contact, its voltage contribution can be neglected. Experiments were performed with four brush direct currents: 50mA, 5A, 30A and 32A. The same current went through both brushes. With the brushes retracted, the slip ring rotated at a speed of about 150 rpm, measured by an optical tachometer. During the experiment, the rotational speed of the slip ring was measured at regular time intervals along with the input power to the motor. The temperature of brush (1) during slip ring contact was automatically recorded on a Honeywell temperature chart recorder at intervals of 30 secs.

The CMA of the Auger spectrometer could examine the positive and negative brush traces on the slip ring surface by raising or lowering the slip ring assembly with the bellows-sealed specimen manipulator after the brushes were retracted with the two linear-rotary motion feedthrus. The Auger electron spectrometer was fully controlled by a Hewlett Packard 9825A desktop computer using a multi-programmer. Typical Auger traces covering a 50 to 1300 eV range were directly digitized with an energy increment of 0.65 eV. Computerized values of peak to peak heights of the AES signals of various elements were obtained with a precision of 1 in 2000. These Auger spectra were taken using a primary beam energy of 3 KeV, a modulation amplitude of 5 volts (peak to peak) and a 50 μA beam current. The Auger spectra were taken while the slip ring was rotating and the brushes were retracted, thus giving an average surface composition of the track. Residual gas analyses were also performed before and after the contact resistance measurements were carried out.

After the experiment was over, the slip ring and brushes were removed from the vacuum chamber.

SEM pictures were then taken of both the positive and negative surface tracks and also of the brush contact surfaces.

RESULTS

A. Contact Resistance and Friction Measurements

The contact resistance measurements are shown in Figs. 3 and 4 for both positive and negative interfaces and for forward and reverse brush polarity. Clearly the contact resistance decreases with increasing number of revolutions at ambient temperature. In addition the curves are more or less parallel to each other indicating that the initial conditions determine whether a curve is "high" or "low". These initial conditions include such variables as surface impurities, brush wire orientation and stiffness, lead resistance, contact force, etc. Also the frictional force increased substantially, resulting eventually in appreciable vibration caused by alternate welding and fracture at the interface. Finally the motor stopped rotating due to excessive friction. In addition, the rotational speed of the motor driving the slip ring decreased by about 25% (from 150 to 115 rpm) while the current to the motor increased by about 20% (from 96 mA to 115 mA) during the experiments.

In Figs. 3 and 4 the circled points indicate the contact resistance R before the slip ring started rotating and after it stopped. It is clear that R did not depend significantly on the angular velocity of the slip ring since the circled values for a non-rotating slip ring are essentially the same as those obtained when it was rotating. The total number of slip ring revolutions at the time each resistance was measured was calculated from a graph of the speed of rotation versus time of the experiment. Figs. 3 and 4 also show that the number 1 brush-slip ring interface (Fig. 1) always had the lower resistance. This result is due to the higher contact force exerted on brush 1. It arises from the manner in which the forces on these brushes are applied. Ex situ experiments showed that the forces needed to break electrical contact on brushes 1 and 2 were 260 and 180 gms, respectively.

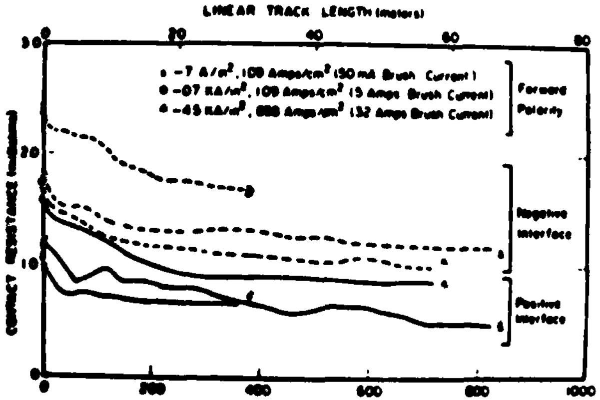

FIGURE 3. Contact resistance versus slip ring revolutions for various current densities. Forward polarity.

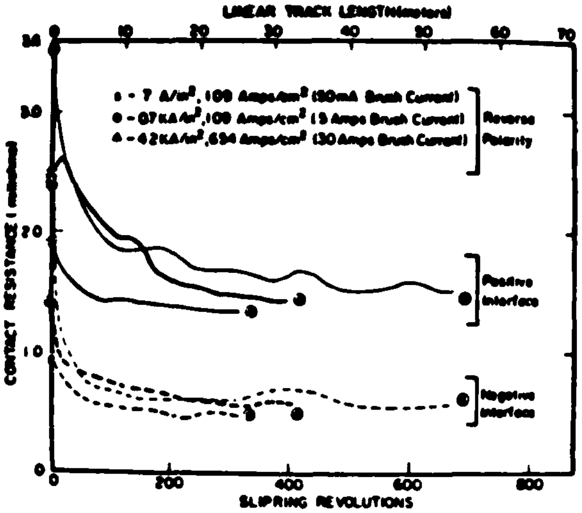

FIGURE 4. Contact resistance versus slip ring revolutions for various current densities. Reverse polarity.

B. AES Results

In each experiment, Auger electron spectra were taken in situ from both the positive and negative brush tracks on the slip ring before and after (but not during) the contact resistance measurements. The brushes were retracted during the AES measurements and the slip ring was continuously rotated. This procedure gave the average impurity concentrations of the whole track with only negligible loss in resolution. Peak to peak heights of all Auger signals were normalized[10,11] to fractional atomic surface concentrations without making any other corrections.[12,13] The observed decrease in the impurity concentration during rotation is given in Table I. The Cu and C concentrations are listed along with the sum of all the impurity concentrations. These impurities consisted of C, S, O, Cl, N. No clear correlation of final impurity concentration with current or with the positive and negative brushes was observed, suggesting that any possible electromigration effects were not significant in the present study. On the other hand, the change in contact resistance ΔR divided by the change in concentration ΔI of the surface impurities on the slip ring was approximately the same for the (+) and (-) interfaces in each of the experiments.

An additional set of experiments was performed to determine the surface composition of the slip ring as a function of the number of revolutions n, rather than just before and just after the resistance measurements were made. These results showed that the change in impurity concentration with n more or less parallels the change in contact resistance.

At the end of each experiment and before the system was opened to dry nitrogen, AES measurements were made on the rotating slip ring (brushes retracted) to determine the magnitude and nature of impurity buildup on the surface due to the residual gases present in the system. It was found that the concentrations of all the impurities increased only very slightly with time. The rate of increase of the total impurity concentration on the rotating slip ring varied from 0.05 to 0.26 a/o per minute. This effect therefore has a negligible influence on the results presented in this report. These results along with the temperature increases observed in the number 1 brush are listed in Table I.

C. Scanning Electron Microscopy

SEM pictures of the brush tracks on the slip ring, taken after the slip ring was removed from the vacuum system, showed that the surface material was smeared out in the track areas, suggesting that the initial surface impurities were buried during rotation. Also small pieces of brush wires W can be seen to be adhering to various places on the slip ring as shown in Fig. 5. The ridges shown in this micrograph lie parallel to the direction of travel. Each ridge arises from random localized welding of a wire to the slip ring during rotation resulting in tensile plastic deformation of the region near the weld area along the direction of motion and eventual fracture of the weld. Continued rotation tends to smooth these areas into ridges. SEM pictures of the brush ends also showed occasional abrupt, broken ends of wires, along with smeared out flat regions also containing ridges, as shown in Fig. 6.

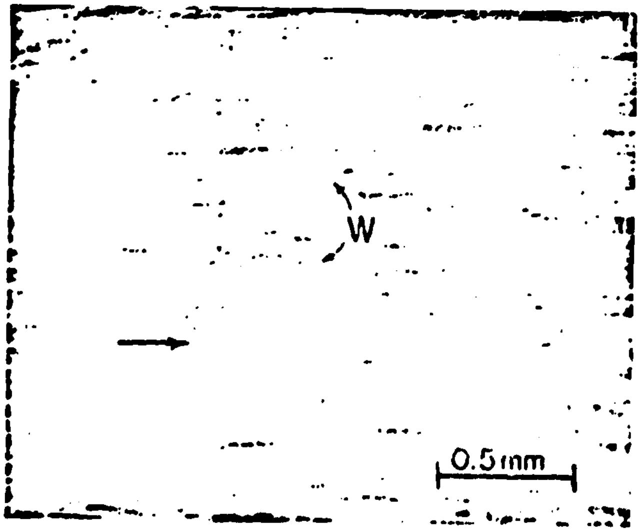

FIGURE 5. SEM photograph of OFHC Cu slip ring, negative track, showing topography and broken brush wire pieces W. Arrow gives direction of relative brush motion.

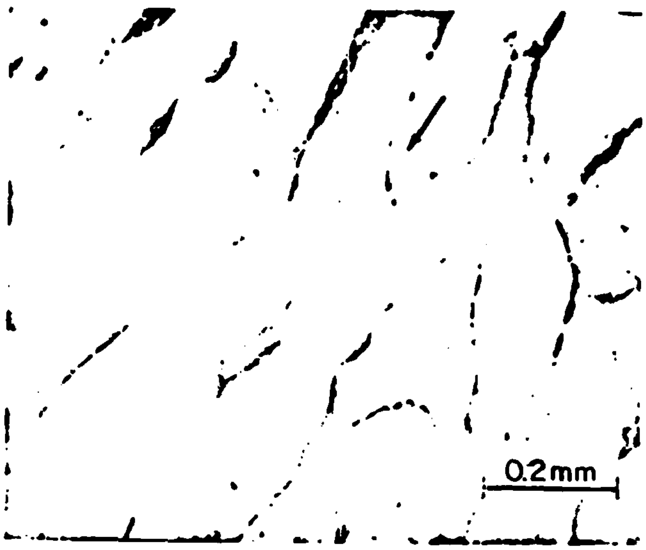

FIGURE 6. SEM photograph of the wear surfaces on the Cu wires of the brush. Arrow gives the direction of motion of the slip ring across these surfaces.

DISCUSSION

A series of experiments in which an OFHC Cu slip ring rotated in contact with a multi-wire 99.999% Cu brush was carried out under UHV conditions. The current densities used were 1.09, 109, 654, and 698 A/cm^2. Both forward and reverse polarity experiments were carried out. Use of a neutral brush permitted the measurement of contact resistance for both the positive and negative interfaces. No systematic relationship between contact resistance and current density was observed. An Auger electron spectrometer was used to determine the chemical composition of the positive and negative tracks on the slip ring.

The results show that for each experiment, the ratio of the change in resistance ΔR to the change in surface impurity concentration ΔI is approximately the same for the positive and negative brushes. Carbon was found to be the major constituent of the impurity concentration. However, for different experiments, the surface composition and contact resistances do not correlate well. This result is due to different initial conditions in different experiments, such as for example slightly different lead resistances, different chemical composition at the initial interface, and different contact pressures and geometries of the brush wires. These experimental variables presumeably gave rise to the parallelism of the contact resistance curves in Figs. 3 and 4. On this basis it is the change in contact resistance ΔR that is due to the chemical impurities at the brush slip ring interface, the absolute values being due largely to systematic experimental variables determined by the initial conditions of the experiment.

It should also be noted that the initial interfacial impurities are distributed throughout a volume of material close to the initial interface by the mechanical "mixing" arising from the plastic deformation that occurs while the slip ring rotates in contact with the brushes. Thus an initial interfacial contact resistance is converted to the volume resistance of a thin layer near the original interface during the course of the experiment. The electrical resistance of this layered distribution of impurities is significantly lower than that of the initial surface distribution of the same impurities. Nevertheless, it will depend on the total number of impurities at the original interface and of course the number of slip ring revolutions that have occurred. Thus the curves in Figs. 3 and 4 would be expected to have small vertical displacements, after hundreds of revolutions, determined by the total surface impurity concentration at the initial interface.

It was also observed that the contact friction increased substantially as the contact surfaces became cleaner. As the rotation proceeded the frictional forces increased until the adhesive forces on the clean portions of the interface exceeded the available torque of the motor turning the slip ring. At this point permanent welding occurred, and it caused the slip ring to stop rotating. The SEM work showed that pieces of the brush wires had broken off and became permanently welded to the slip ring. The wear surfaces themselves consisted of an extremely rough and ridged topography that had clearly undergone severe plastic deformation.

Finally, the temperature measurements indicated that during rotation the temperature near the interface rose some 10 to 25°C above room temperature, the higher values applying to the higher current densities. Apparently much higher temperatures were prevented by the high thermal conductivity of the brush and slip ring materials. Clearly the temperature increase was not significantly related to the current density since a more than two orders of magnitude increase in current resulted in only a modest rise in temperature. These results suggest that interfacial friction, rather than current density may be the principal contributor to the temperature increase observed in the present experiments.

TABLE I

Brush	Maximum pressure 10^{-9} torr	Brush current (amps)	Composition before rot.(a/o)			No. of rot. (n)	Composition after rot.(a/o)			Contact Resist.		ΔR/ΔI	Increase in brush temp. at end (°C)
			Cu	total impurities	C		Cu	total impurities	C	before rot. (mΩ)	after rot. (mΩ)		
1	4	0.05 +	42	58	44	825	98.3	1.7	1.0	1.21	0.47	0.013	11
2		-	46	54	37		97.9	2.1	1.4	1.78	1.17	0.012	--
1	6	-0.05 -	59	41	34	675	96.4	3.6	2.3	2.40	0.60	0.048	9
2		+	60	40	30		97.6	2.4	1.5	3.50	1.50	0.053	--
1	4	5 +	56	44	30	365	87.9	12.1	7.9	1.05	0.64	0.013	13
2		-	51	49	33		86.7	13.1	9.3	2.40	1.68	0.020	--
1	5	-5 -	60	40	34	305	94.8	5.2	3.4	0.92	0.48	0.013	13
2		+	56	44	38		94.2	5.8	3.8	1.92	1.45	0.012	--
1	4	32 +	48	52	42	710	97.1	2.9	2.0	1.60	0.85	0.015	24
2		-	53	47	35		95.2	4.8	3.7	1.73	1.01	0.017	--
1	4	-30 -	31	69	59	400	91.5	8.5	3.5	1.40	0.54	0.014	17
2		+	32	68	55		92.2	7.8	3.8	2.50	1.49	0.017	--

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The authors would like to thank Rolf Ziemer and Bing Hwai Hwang for technical assistance; E.-A. Knabbe for the computer interfacing; R.A. Burton, I.R. McNab, J. K. Johnson, J. J. Schreurs, and P. K. Lee for helpful discussions; the Westinghouse Research and Development Center for the loan of certain equipment; and the Office of Naval Research for financial support under contract no. N00014-79-C-0763.

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Appendix IV

Interfacial Characterization of Copper Slip Ring - Wire Brush Contacts

by

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INTERFACIAL CHARACTERIZATION OF COPPER SLIP RING-WIRE BRUSH CONTACTS

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Abstract

The chemical, electrical, and mechanical properties of the interface between a Cu slip ring that rotates in contact with two Cu wire brushes were investigated by Auger electron spectroscopy(AES), scanning electron microscopy(SEM), contact resistance, and frictional force measurements. The experiments were carried out in an ultra high vacuum system. The contact resistance at both the positive and negative interfaces decreased with increasing number of slip ring revolutions while the frictional force increased. In situ AES measurements showed that the composition of the slip ring surface, which was initially covered by about 50 atomic percent(a/o) of carbon, changed drastically during rotation. After many revolutions it approached that of a clean Cu surface (total impurities < 10 a/o). The decrease in contact resistance with the number of slip ring revolutions more or less paralleled the decrease in total impurities. This parallelism suggests that the contact resistance is caused predominantly by the surface impurities. No systematic relationship between contact resistance and brush current density was observed. SEM observations showed that the surface material was smeared out in the brush track areas and the initial surface impurities were buried during rotation.

1. Introduction

The current carrying capacity and the dynamic friction and wear properties of electrical contacts are important technological considerations which are presently not very well understood. The application of surface science tools {1-5} to these problems has brought with it an increased understanding of the complexity of the situation. The present communication describes the results obtained in the application of some of these techniques to an in situ, ultra high vacuum(UHV) study of the interface between a rotating slip ring in contact with a wire brush. It represents the initial stage of a broader research program in which the effects of controlled gaseous environments will be determined.

2. Experimental Arrangement and Method of Measurement

A stainless steel UHV system (residual gas pressure $3\text{x}10^{-9}$ torr, $4\text{x}10^{-7}$ Pa) was used to study electrical contact phenomena associated with rotation of a copper slip ring (OFHC-99.98%Cu) in contact with two copper wire brushes(each consisting of 362, 0.005" diameter, 99.999%Cu wires) running on separate tracks {6}. The brushes were pressed against the slip ring surface by means of an insulated steel spring. They can be removed from the surface by manipulating two linear-rotary vacuum feedthrus. The slip ring is attached to a magnetically coupled rotary vacuum feedthru which is turned by an A.C. motor coupled to it by a rubber belt. The UHV system contains an Auger cylindrical mirror analyzer(CMA), a 3 KeV sputter ion gun and a 90° magnetic sector partial pressure analyzer.

The surfaces of the slip ring and the wire brushes were polished smooth with emery paper (grit 600-A) and then rinsed in acetone and ethanol before being put in the vacuum system. The slip ring was electrically grounded and the

contact resistances between (1) the positive brush and slip ring and (2) the negative brush and slip ring were recorded on a dual pen recorder for brush currents of 50mA, 5A and 30A. With brushes retracted, the slip ring rotated at a speed of about 150 rpm, measured by an optical tachometer. The normal force on each brush was measured. The average frictional force was determined from the decrease in rotational speed of the slip ring by comparing it to a calibration involving known torques applied to the slip ring in a separate experiment in air. The Auger electron spectrometer used to determine the elemental surface composition of the slip ring surfaces was controlled by a Hewlett Packard 9825A desk top computer using a multi-programmer. Typical Auger traces covering a 50 to 1300 eV range were directly digitized. Computerized values of peak to peak heights and concentrations of the various elements were obtained. A schematic diagram of the slip ring-brush assembly and further details of the experimental techniques are given elsewhere [7]. After each experiment was over, the slip ring and brushes were removed from the vacuum chamber, and SEM pictures were taken of both the positive and negative surface tracks on the slip ring and also of the brush contact surfaces.

3. Contact Resistance and Friction

The contact resistance decreased with increasing number of revolutions. Also the rotational speed of the motor driving the slip ring decreased while the current to the motor increased, both as a result of increasing frictional force. Alternate welding and fracture at the interface resulted in an appreciable vibration culminating in permanent welding which caused the motor to stop. The ratio of the frictional force to the measured average normal contact forces on both brushes gave the average coefficient of friction μ. The contact resistances for the positive and negative interfaces at a 5 Amps.D.C. brush current and the average μ are shown in Fig.1. Similar results were obtained for 50mA and 30 A brush currents for both forward and reverse polarity. In Fig.1 the circled points indicate the contact resistance R before the slip ring started rotating and after it stopped. It is clear that R did not depend significantly on the angular velocity of the slip ring. The lower contact resistance at the negative brush was due to the higher contact force there. It arises from the manner in which the forces on these brushes were applied[7]. In the experiment of Fig.1 the normal brush forces needed to break electrical contact with the slip ring were 260(-) and 180(+) gms respectively. No systematic relationship between contact resistance and current density was observed.

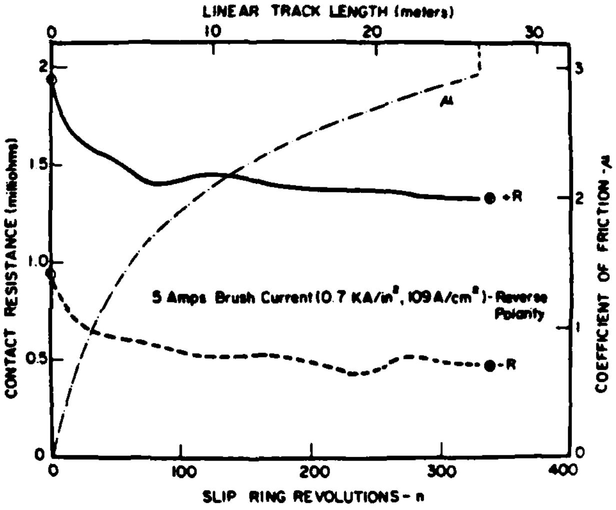

Fig.1: Contact resistance and average coefficient of friction versus slip ring revolutions.

4. AES Measurements

Elemental surface compositions of both the positive and negative brush tracks on the slip ring, and interface contact plus lead resistances for both brushes are shown in Fig.2 as a function of the number of revolutions n for a 50 mA brush current. The circled points represent the values before the slip ring started rotating. In this set of experiments a different lead arrangement from that used for Fig.1 was employed for the contact resistance measurements. It included the lead resistance and thus gave the higher resistance values. Also the normal brush contact forces were comparatively small (169 and 118 gms), leading to an increased number of revolutions prior to permanent welding. During AES measurements the brushes were retracted and the slip ring was continuously rotated. The measurements thus gave the average surface concentrations of the whole track, with only negligible loss in resolution. Peak to peak heights of all Auger signals were normalized to fractional atomic surface concentrations without making any other corrections. Nitrogen and sulfur were also detected in addition to the impurities shown in Fig.2, but their concentrations decreased to almost zero from initial values that were less than 5 a/o.

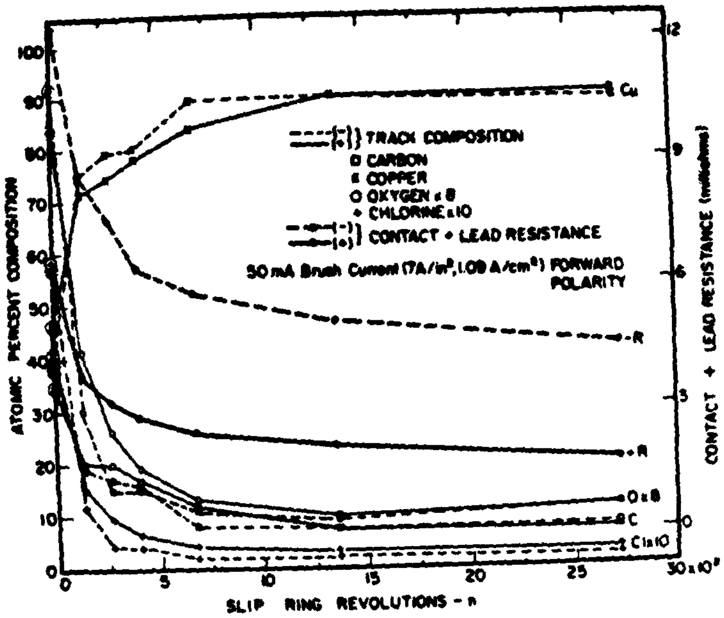

Fig.2: Contact plus lead resistance and composition of both slip ring track surfaces as a function of slip ring revolutions.

Fig. 2 shows that the change in impurity concentration with n more or less parallels the change in contact resistance. Similar parallels were also observed in experiments in which currents were stepped from 5 to 30 Amps. These results suggest that the interface impurity concentration is directly related to the contact resistance. Finally no significant electromigration effects up to current densities of 654 amps/in^2 were detected.

Fig.3: SEM photograph of OFHC Cu slip ring, negative track, showing broken wire pieces W and ridges. Arrow gives direction of relative brush motion.

5. Scanning Electron Microscopy

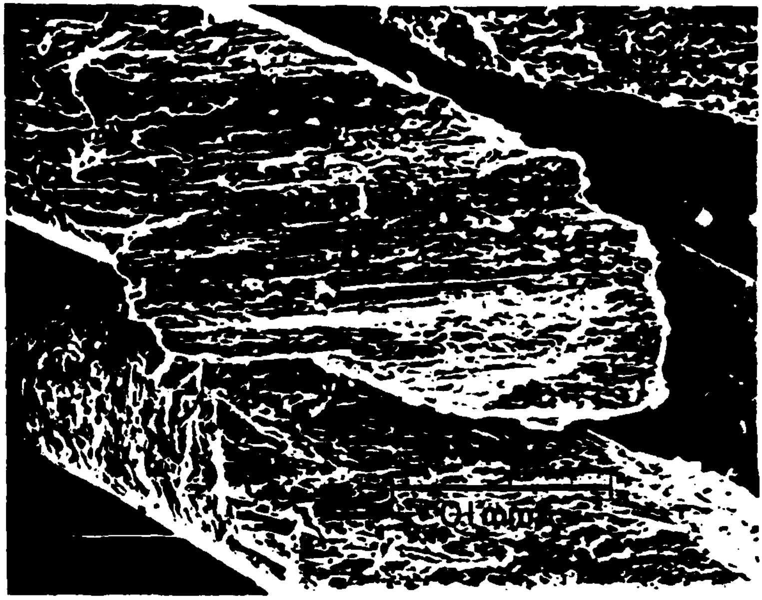

Fig.4: SEM photograph of the wear surfaces on the Cu wires of the brush. Arrow gives the direction of motion of the slip ring.

SEM pictures of the brush tracks on the slip ring showed that surface material was smeared out along the track areas. Thus the initial surface impurities appear to have been buried during rotation. Small pieces of brush wires W in Fig.3 were seen adhering to various places on the slip ring. The ridges shown in this micrograph lie parallel to the direction of travel. SEM pictures of the brush ends also showed similar ridges parallel to the direction of relative motion. See Fig.4. Note also the buildup of material on the leading edges of the wire brush ends. The ridges arise from random localized welding of a wire to the slip ring during rotation resulting in tensile plastic deformation of the region near the weld area along the direction of motion followed by eventual fracture of the weld. Continued rotation tends to smooth these areas into ridges.

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